STEERMAN ENVIRONMENTAL

via electronic transfer

February 6, 2012

Michelle Kerr
Remedial Project Manager
U.S EPA – Region 5
77 W. Jackson Blvd.
Mail Code: S-6J
Chicago, IL 60604-3590

Re: Additional Groundwater Studies Supplemental Work Plan
United States of America v. AK Steel Corporation et. al.
Case No. 1:10-cv-00996-KMO
Chemical Recovery Systems Superfund Site, Elyria, Ohio

Dear Ms. Kerr:

Enclosed is a proposed work plan for collection of supplemental data that is necessary to finalize the Additional Groundwater Studies report, which is a requirement of the Consent Decree and Statement of Work, and to finalize the Conceptual Site Model of geologic and hydrogeologic conditions. The need to perform the proposed supplement investigative work was discussed during the meeting between the CRS Site RD/RA Group and USEPA on 14th of November 2011.

If you have any questions regarding the rationale and scope of the proposed work plan and assessment activities please feel free to contact the undersigned at (770) 992-2836, or by electronic message to psteerman@charter.net.

Best Regards,

Patrick S. Steerman

Patrick S. Steerman
CRS Site Project Coordinator

ec: Larry Antonelli, Ohio EPA (electronic & printed copy)
Richard Karl, Director, Superfund Division EPA Region 5 (electronic copy)
Doug McWilliams, CRS Site RD/RA Group Chair/Common Counsel (electronic & printed copy)
Tom Nash, Esq., U.S. EPA (electronic copy)
Jim Peeples, Brown & Caldwell (electronic copy)
CRS Site RD/RA Group Performing Parties (electronic copy)

Steerman Environmental
Management & Consulting, LLC
422 Creek View Lane
Roswell, Georgia 30075
(404) 421-3275 - Voice
psteerman@charter.net

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

March 14, 2012

SR-6J

REPLY TO THE ATTENTION OF:

Mr. Patrick Steerman
Steerman Environmental Management & Consulting, LLC
422 Creek View Lane
Roswell, GA 30075

Re: Chemical Recovery Systems Inc. Site
Comments on Additional Groundwater Studies Supplemental Work Plan and Technical Impracticability Determination Scope of Work

Dear Mr. Steerman:

On February 6, 2012, the U.S. Environmental Protection Agency (EPA) and Ohio Environmental Protection Agency (OEPA) received the additional groundwater studies supplemental work plan (AGWS-S) and Technical Impracticability Determination Scope of Work from Steerman Environmental Management & Consulting, representing the Chemical Recovery Systems Remedial Design/Remedial Action Group Performing Parties (Group). The additional work proposed for the site was discussed during the November 14, 2011 meeting between the Group and EPA. The need for additional investigation activities discussed in this meeting prompted the current supplement submitted by the Group to the March 2011 additional groundwater studies work plan (AGWS). This AGWS-S proposes activities to further evaluate the ground water, underlying bedrock, and non-aqueous phase liquids (NAPL) at the site. The Group intends to submit the results of these additional studies to the Agencies sometime in July 2012. The work will be completed to refine the hydrogeologic conceptual site model and to finalize the AGWS report.

Following are questions and comments from the Agencies on the plan. Mainly, we request clarifications.

1. Technical Impracticability Determination Scope of Work

For the three categories of data, analysis, and criteria used in evaluating the application of a technical impracticability (TI) waiver, item three (and task 10) seems to encompass an evaluation of the site's restoration potential, as described in EPA's TI guidance. Either here or in the proposed TI work plan, consider adding to the scope an outline of how the Group will evaluate whether other remedial technologies could feasibly attain cleanup levels in a reasonable timeframe. Also for task 10, consider adding an evaluation of costs for remedy options.

2. Standard Operating Procedures (SOPs), Calibration Procedures, and Quality Assurance/Quality Control (QA/QC) During Field Activities.

Recycled/Recyclable • Printed with Vegetable Oil Based Inks on 100% Recycled Paper (50% Postconsumer)

a. Field sampling SOPs detailing the ground water and surface water investigation activities were not included in the Supplemental Work Plan. The associated SOPs were not referenced and/or included in the AGWS-S to further support the field sampling procedures. SOPs should be referenced and/or included for all field readings and sample collections proposed in the work plan to further support the field sampling procedures.

b. A discussion is needed concerning the calibration procedures and standards to be used during field sampling. The AGWS-S lacks any discussion concerning the calibration standards that will be used during equipment calibration and during recovery and/or measurement activities.

c. Clarification is needed for those QA/QC procedures that will be employed during field sampling. The submittal did not discuss the collection of QA/QC samples during the various field sampling activities. Additional information should be provided which includes a discussion of the QA/QC procedures to be used.

d. Additional information is needed detailing the container preservation, shipping, and packaging procedures for the ground water samples, surface water samples, NAPL samples, and rock core samples. The submittal did not provide a discussion concerning container preservation, shipping, and packaging activities. The AGWS-S needs to briefly discuss or cite procedures for container preservation, shipping, and packaging.

e. A discussion on decontamination procedures is needed in the AGWS-S. The submittal lacks any discussion concerning field decontamination procedures.

The Agencies acknowledge that the SOPs and standards are contained within Attachment A (Field Sampling Plan) of the March 2011 AGWS Work Plan, and the original work plan was noted in the *References* of the AGWS-S. However, there does not appear to be any back reference of these procedures and protocols within the text of the AGWS-S. This could be stated within the *Introduction* of the 2012 Work Plan which does mention that the work described in the plan itself will supplement the information collected in the summer and fall of 2011.

3. Section 2.2 – Additional Clarification Needed Concerning the Temporal Level Measurement Activities.

a. Section 2.2 indicates that a *"...barometric transducer will be installed in the unsaturated zone of one of the wells..."* (pg. 2-1). The criteria that will be used to select this well were not discussed in the text. Inclusion of this well within the final report would be sufficient if its location is currently not known.

b. As with the comment above, Section 2.2 states that a stilling well will be installed *"...in the river at the location of the existing gauging rod..."* (pg. 2-1). The location of this stilling well should be illustrated in future report figures.

c. Section 2.2 indicates that "...data will be downloaded periodically during the collection period to minimize the potential for data loss ... " (pg. 2-1). It is unclear how frequently these data will be downloaded. Please state the frequency.

d. The last paragraph of this section states that a goal of the work is to determine if the river is, on average, a gaining stream. It should be recognized that a stream might be on average "gaining", yet have sections that locally are losing or might be on average "losing", yet have sections that locally are gaining.

4. Section 2.3 -- Observe Seep Zones

What is the anticipated accuracy of the "approximate measurements" proposed and at what frequency/how often will the measurements be made?

5. Section 2.4 -- Complete a Bathymetric Survey

How will the bathymetric survey facilitate identifying intervals of higher density of site bedrock fractures? What is the proposed resolution (i.e. the spacing), both horizontally and vertically of the bathymetric survey elevation measurements proposed?

6. Section 2.5 – The procedure for rock permeability testing should be included within the final report.

a. The testing procedure proposed involves adding water to the aquifer. We generally do not recommend that water be added to the aquifer. There exist alternate approaches for testing the permeability of open rock sections of bedrock wells that do not require the addition of water. However, if water is to be added, the source of the "potable" water being used should be specified, the water should be tested (for volatile and semi-volatile organic compounds), and those results provided to the Agencies. Before wells to which water has been added are sampled again, the wells should be redeveloped to remove/reduce the effect of the added water.

b. While the work plan refers to ASTM D4630-96 as the testing method to be followed, a SOP specifically applicable to this project should provided. This should include recording the total volume of water added to each well interval tested.

c. The work plan does not specify the depths of bedrock well segments to be tested or the lengths of the segments. Will this information be provided prior to initiating the testing?

7. Section 2.6 -- Surface Water Sampling

a. Will any screening be done to attempt to identify ground water discharge points? Samples should be collected from near the bottom of the water column (e.g. six inches above the river bottom).

b. The work plan proposes collecting the samples in collection bottles and then transferring the samples into the appropriate laboratory bottles/vials. While it is stated that care will be taken to minimize exposure to air and the disturbance of the water, the process of transferring will result in some volatilization. Samples should be collected in such a way that volatilization will not occur. This may be done, for example, by using the laboratory bottles/vials as the collection bottle (although preservation (if required) must be done after sample collection) or by collecting the samples using a pump with a short length of tubing.

c. It is stated that " The sampling procedures and chain-of-custody and shipping protocols will be completed according to the methods detailed in the Site Field Sampling Plan (FSP) (provided with the AGWS Work Plan)...". There was no surface water sampling procedures provided in the June 2011 Additional Ground Water Studies Work Plan. Will a FSP Addendum be provided?

8. Section 3.1 – Information on NAPL Recovery and Measurement Activities.

a. Additional information is needed concerning which wells will be used for NAPL recovery activities. Section 3.1 indicates that *"...NAPL bail-down tests will be conducted to quantify recovery rates and onsite NAPL transmissivities."* (pg. 3-1). However, the wells selected for this activity were not discussed in the submittal. If this information is not known yet, it can be documented in the final report.

b. In the second paragraph of this section, reference is made to the Field Sampling Plan (FSP). There was no dense NAPL sampling procedures provided in Appendix A: FSP of the June 2011 Additional Ground Water Studies Work Plan. Will a FSP Addendum be provided with SOPs for collecting NAPL directly and by using absorbent socks?

9. Section 3.2 – Bedrock Coring

a. Clarification is needed regarding petrophysical analysis. What properties will be measured (would the testing be limited to pore fluid saturation of NAPLs)? Will a SOP be provided for this testing?

b. Reference is made in the second paragraph to "the required depth". What is the required depth of the rock coring? If coring goes deeper that than the first impacted interval, how will pull-down be prevented?

c. The third paragraph mentions that cores will be preserved. What exactly does this mean? Perhaps the applicable SOP may explain this.

d. For chemical analysis of the rock core samples, will the rock be crushed prior to analysis (e.g. in the lab)?

Thank you for your attention to these questions and comments. If you choose to, we can set up a call to talk about the work plan and comments soon. If you have any questions or concerns, please contact me at (312) 886-8961.

Sincerely,

Michelle Kerr
Remedial Project Manager

cc via email: L. Antonelli, OEPA
L. Mencin, Sherwin Williams
J. Peeples, B&C
L. Vanderpool, EPA

STEERMAN ENVIRONMENTAL

via first class mail

April 5, 2012

Michelle Kerr
Remedial Project Manager
U.S EPA – Region 5
77 W. Jackson Blvd.
Mail Code: S-6J
Chicago, IL 60604-3590

Re: Response to USEPA Comments Regarding Additional Groundwater Studies Supplemental Work Plan and Technical Impracticability Determination Scope of Work
United States of America v. AK Steel Corporation et. al.
Case No. 1:10-cv-00996-KMO
Chemical Recovery Systems Site, Elyria, Ohio

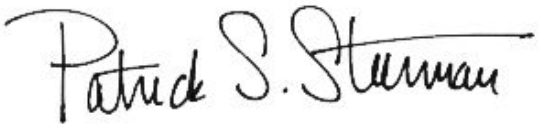
Dear Ms. Kerr:

On February 6, 2012, the Chemical Recovery Systems, Inc. (CRS) Site Remedial Design/Remedial Action (RD/RA) Group Settling Performing Defendants (Group) submitted an Additional Groundwater Studies Supplemental (AGWS-S) Work Plan and, under separate cover, a letter discussing a Technical Impracticability (TI) Determination Scope of Work for the CRS site (the Site). The additional work proposed for the Site was discussed during the November 14, 2011 meeting between the Group and the United States Environmental Protection Agency (USEPA). The need for additional investigation activities discussed in this meeting prompted the February 6, 2012 supplement to the approved March 2011 Additional Groundwater Studies Work Plan (AGWS). In a letter dated March 14, 2012, the USEPA provided questions and comments on the AGWS-S Work Plan and the TI Determination Scope of Work. The questions and comments were reviewed by the Group and are addressed in the enclosed letter prepared by Brown and Caldwell. In this letter, the questions and comments are listed and are followed by responses by the Group shown in italics. As referenced in the letter, a revised AGWS-S work plan, which includes a copy of the revised Field Sampling Plan and Quality Assurance Project Plan, is enclosed for your review.

If you have any questions regarding the rationale and scope of the proposed TI investigation and assessment activities please feel free to contact the undersigned at (770) 992-2836, or by electronic message to psteerman@charter.net.

Steerman Environmental
Management & Consulting, LLC
422 Creek View Lane
Roswell, Georgia 30075
(404) 421-3275 - Voice
psteerman@charter.net

Best Regards,



Patrick S. Steerman
CRS Site Project Coordinator

ec: Larry Antonelli, Ohio EPA
Nigel Goulding, EHS Support
Larry Mencin, CRS PRP Group, Technical Committee Chair,
Jim Peeples, Brown & Caldwell
Doug McWilliams, CRS PRP Group Counsel

6055 Rockside Woods Boulevard, Suite 350
Independence, Ohio 44131
Tel: 216-606-1300
Fax: 216-606-1350
www.brownandcaldwell.com

Brown AND Caldwell

April 5, 2012

Patrick S. Steerman
Steerman Environmental Management & Consulting, LLC
422 Creek View Lane
Roswell, Georgia 30075 139452

Subject: Response to USEPA Comments regarding the Additional Groundwater Studies Supplemental Work Plan and Technical Impracticability Determination Scope of Work
United States of America v. AK Steel Corporation et. al.
Case No. 1:10-cv-00996-KMO
Chemical Recovery Systems Site, Elyria, Ohio

Dear Mr. Steerman:

On February 6, 2012, the Chemical Recovery Systems, Inc. (CRS) Site Remedial Design/Remedial Action (RD/RA) Group Settling Performing Defendants (Group) submitted an Additional Groundwater Studies Supplemental (AGWS-S) Work Plan and, under separate cover, a letter discussing a Technical Impracticability (TI) Determination Scope of Work for the CRS site (the Site). The additional work proposed for the Site was discussed during the November 14, 2011 meeting between the Group and the United States Environmental Protection Agency (USEPA). The need for additional investigation activities discussed in this meeting prompted the February 6, 2012 supplement to the approved March 2011 Additional Groundwater Studies Work Plan (AGWS). The AGWS-S proposes activities to further evaluate the ground water, underlying bedrock, and non-aqueous phase liquids (NAPL) at the Site; to refine the hydrogeologic conceptual site model; and to finalize the AGWS report. As discussed in the AGWS-S, proposed field activities include the collection of Site field data proposed in the TI Determination Scope of Work. In a letter dated March 14, 2012, the USEPA provided questions and comments on the AGWS-S Work Plan and the TI Determination Scope of Work. The questions and comments are provided below and are followed by responses by the Group shown in italics.

1. Technical Impracticability Determination Scope of Work

For the three categories of data, analysis, and criteria used in evaluating the application of a technical impracticability (TI) waiver, item three (and task 10) seems to encompass an evaluation of the site's restoration potential, as described in EPA's TI guidance. Either here or in the proposed TI work plan, consider adding to the scope an outline of how the Group will evaluate whether other remedial technologies could feasibly attain cleanup levels in a reasonable timeframe. Also for task 10, consider adding an evaluation of costs for remedy options.

Group Response:

In accordance with the TI framework, the Group proposes to develop a focused feasibility study that assesses any other remedial technologies demonstrated capable of achieving the current cleanup levels within a reasonable timeframe. This focused feasibility study will be supported by:

1. *A literature review of remedial activities and demonstrated successes at other Manufactured Gas Plant sites where LNAPL and DNAPL are present within bedrock; and*
2. *Fate and transport modeling to assess the benefits of source remediation on plume extent and longevity (key considerations under the criteria outlined in the NCP).*

As part of the focused feasibility study, the Group will evaluate the costs of the technically feasible remedial options in the context of the net environmental benefits (if any) of each remedial alternative.

2. Standard Operating Procedures (SOPs), Calibration Procedures, and Quality Assurance/ Quality Control (QA/QC) During Field Activities.

a. Field sampling SOPs detailing the ground water and surface water investigation activities were not included in the Supplemental Work Plan. The associated SOPs were not referenced and/or included in the AGWS-S to further support the field sampling procedures.

   SOPs should be referenced and/or included for all field readings and sample collections proposed in the work plan to further support the field sampling procedures.

   Group Response:

   References have been added to the AGWS-S to indicate the location of relevant Standard Operating Procedures (SOPs) and work descriptions that are contained in the approved March 2011 Field Sampling Plan (FSP). A description of the surface water sampling methods has been added to the FSP and is referenced in the AGWS-S. Additional references have been added to the AGWS-S to indicate locations within the FSP where specific and detailed procedures for obtaining field readings and completing field sampling can be found. The revised FSP and AGWP-S are transmitted with this letter.

b. A discussion is needed concerning the calibration procedures and standards to be used during field sampling. The AGWS-S lacks any discussion concerning the calibration standards that will be used during equipment calibration and during recovery and/or measurement activities.

   Group Response:

   Descriptions of calibration procedures and standards for work to be completed in the AGWS-S are provided in the approved FSP and Quality Assurance Project Plan (QAPP). References to the relevant sections of the FSP and QAPP have been added to the revised AGWS-S.

c. Clarification is needed for those QA/QC procedures that will be employed during field sampling. The submittal did not discuss the collection of QA/QC samples during the various field sampling activities. Additional information should be provided which includes a discussion of the QA/QC procedures to be used.

Group Response:

Sampling Quality Assurance/Quality Control (QA/QC) procedures are detailed in the September 2010 approved QAPP and in the FSP. The procedures indicate the number and type of QA/QC samples that will be collected for the field activities performed for the AGWS-S. References to relevant sections of the QAPP and FSP have been added to the revised AGWS-S Work Plan.

d. Additional information is needed detailing the container preservation, shipping, and packaging procedures for the ground water samples, surface water samples, NAPL samples, and rock core samples. The submittal did not provide a discussion concerning container preservation, shipping, and packaging activities. The AGWS-S needs to briefly discuss or cite procedures for container preservation, shipping, and packaging.

Group Response:

Procedures for groundwater, surface water, and NAPL sample container preservation, packaging and shipping for all sample matrices are contained in the updated FSP, attached to the revised AGWS-S Work Plan. References to the FSP have been inserted at the appropriate locations within the AGWS-S Work Plan. With respect to rock core collection, handling and preservation will be conducted in accordance with API Recommended Practice 40 (RP 40) which is the industry standard for collection of NAPL and core samples for petrophysical testing. These methods have been developed for the oil industry for non-aqueous phase fluids and are recognized as the industry standards by which samples should be collected, handled, preserved and shipped to the laboratory. API RP 40 and the PTS laboratories recommendations for shipment have been added to the FSP. Once received at the laboratory the samples will be kept frozen at cryogenic temperatures to ensure that the pore fluids are retained within the core prior to core photography and/or analysis.

e. A discussion on decontamination procedures is needed in the AGWS-S. The submittal lacks any discussion concerning field decontamination procedures.

Group Response:

Decontamination procedures are discussed in the FSP and references to the FSP have been added to the AGWS-S regarding decontamination procedures.

The Agencies acknowledge that the SOPs and standards are contained within Attachment A (Field Sampling Plan) of the March 2011 AGWS Work Plan, and the original work plan was noted in the *References* of the AGWS-S. However, there does not appear to be any back reference of these procedures and protocols within the text of the AGWS-S. This could be stated within the *Introduction* of the 2012 Work Plan which does mention that the work described in the plan itself will supplement the information collected in the summer and fall of 2011.

Group Response:

The suggested references to the FSP and QAPP have been added to the introduction of the AGWS-S, and references to the FSP and QAPP also

have been added in several locations within relevant task descriptions. An updated version of the FSP is now included as an appendix to the AGWS-S.

3. Section 2.2 - Additional Clarification Needed Concerning the Temporal Level Measurement Activities.

a. Section 2.2 indicates that a *"…barometric transducer will be installed in the unsaturated zone of one of the wells…*" (pg. 2-1). The criteria that will be used to select this well were not discussed in the text. Inclusion of this well within the final report would be sufficient if its location is currently not known.

Group Response:

The reason for placing the barometric probe inside a well casing is to minimize the temperature fluctuations that the probe is exposed to and improve barometric pressure data quality. To achieve this, the barometric transducer/data logger (probe) can be added to the unsaturated portion of any monitoring well at the site. The text of the AGWS-S has been updated to indicate this. The final selection of a well for placement of the barometric probe will be made at the time of the field investigation.

b. As with the comment above, Section 2.2 states that a stilling well will be installed *"…in the river at the location of the existing gauging rod…"* (pg. 2-1). The location of this stilling well should be illustrated in future report figures.

Group Response:

The location of the stilling well and the river gauge will be illustrated in future report figures.

c. Section 2.2 indicates that "…*data will be downloaded periodically during the collection period to minimize the potential for data loss*…" (pg. 2-1). It is unclear how frequently these data will be downloaded. Please state the frequency.

Group Response:

The maximum time between data downloads for the transducers will be three weeks. A statement indicating this has been added to the AGWS-S.

d. The last paragraph of this section states that a goal of the work is to determine if the river is, on average, a gaining stream. It should be recognized that a stream might be on average "gaining", yet have sections that locally are losing or might be on average "losing", yet have sections that locally are gaining.

Group Response:

It is recognized that the river will likely be a gaining stream in some locations and a losing stream in other locations. The intent of the work described in the AGWS-S Work Plan will be to evaluate the segment of the river along the boundary of the Site. It is very likely that conditions along this small stretch of river will be consistent and can be characterized as being, on average, gaining or losing. The intent of the work described in

the AGWS-S will be to collect and evaluate data necessary to make this determination for just the stretch of the river along the Site boundary.

4. Section 2.3 - Observe Seep Zones

What is the anticipated accuracy of the "approximate measurements" proposed and at what frequency/how often will the measurements be made?

Group Response:

It is recognized that there are limitations with the quantification of flows using the methodologies proposed in the AGWS-S Work Plan. However, the purpose of the assessment of the rock phase is to provide a weight of evidence approach to the assessment of hydraulic conductivities and groundwater flux. The seep face assessment will be supported by hydraulic conductivity testing conducted on-site which will be used to develop Darcian estimates of groundwater flux. In all cases the seeps will be marked, mapped and the dimensions measured and information recorded in the field notes. In addition, photographs of the observed seeps (if present) will be provided.

The accuracy of the measurements made will depend on the ability to account for all the flow from a given seep. It should be understood that, to date, the seeps have been observed as wet spots with minimal to no observable water flow or dripping. If no water flow is observed at a given seep, we will quantify the maximum rate of flow as the rate of evaporation from the wet rock surface, given the environmental conditions at the time of the observation. If observable flow is present it will be directed, as much as is reasonably possible, to a central location where it will be quantified based on the rate of filling a known container volume. Under the appropriate circumstances, these measurements can be quite accurate. If accurate flow rate measurements cannot be obtained at a given location, this result will be documented.

The intent of this work is to obtain seep flow measurements from all active seeps on a single day, and that only one set of seep flow measurements will be obtained. These estimates of seep flows will be compared and contrasted to the Darcian mass fluxes in order to estimate (for the purposes of the hydrogeologic conceptual site model) the potential bank and basal discharge of groundwater to the river. It is anticipated that collectively, seeps from the Site only contribute a very small component of the groundwater flow to the river.

5. Section 2.4 - Complete a Bathymetric Survey

How will the bathymetric survey facilitate identifying intervals of higher density of site bedrock fractures? What is the proposed resolution (i.e. the spacing), both horizontally and vertically of the bathymetric survey elevation measurements proposed?

Group Response:

The river bed topography will be characterized using a grid of survey points placed approximately 10-feet on center through the study area (from the east river bank to the west bank and at least to the north and south boundaries of the Site). For each survey point, an observation will be made regarding the presence of a hard bottom, gravel/rubble, or a soft bottom, indicating the nature of the river base material at each location. This determination will be made with a survey rod that has a "shoe" (to prevent it from sinking into the alluvial soil) to

define the presence and upper elevation of alluvial materials and a survey rod with a point (advanced to refusal) to define the upper surface of the bedrock. This will allow for measurement of the rock and alluvial surfaces and the thickness of alluvial materials, where present, through the study area.

The bathymetric survey will not provide information regarding intervals of higher fracture density in the bedrock. The AGWS-S Work Plan indicates that the base of the river (accurately depicted with the bathymetric survey) will be included in cross sections that will show the Site topographic features (such as the base of the river) in comparison to high fracture density portions of the bedrock identified within the Site. The fracture density of the bedrock will be determined from the boring logs and geophysical logging conducted onsite as part of the completed AGWS and the proposed AGWS-S. The elevation and orientation of these identified fractures will be projected from the site towards the river to assess if they are potentially intercepted. The fractures evident on the bedrock outcrop in the southwest area of the Site will be surveyed and also noted in the cross sections.

6. Section 2.5 - The procedure for rock permeability testing should be included within the final report.

   a. The testing procedure proposed involves adding water to the aquifer. We generally do not recommend that water be added to the aquifer. There exist alternate approaches for testing the permeability of open rock sections of bedrock wells that do not require the addition of water. However, if water is to be added, the source of the "potable" water being used should be specified, the water should be tested (for volatile and semi-volatile organic compounds), and those results provided to the Agencies. Before wells to which water has been added are sampled again, the wells should be redeveloped to remove/reduce the effect of the added water.

Group Response:

It is agreed that water reasonably free of VOCs and SVOCs should be used for the bedrock testing. To accomplish this, only locally available potable water (i.e., City of Elyria drinking water) will be added to the aquifer as part of permeability testing. The source of this water will be documented in the final AGWS report. To the extent necessary, test data regarding VOCs/SVOCs can be obtained from the potable water supplier.

While there are methods for testing bedrock permeability that involve the withdrawal of water from the boring, rather than the addition of water, it is believed that injection methods are better suited for this specific application. The purpose of permeability testing is to measure bedrock permeability in areas of the borehole that are isolated by a packer system. Because of the packer placement and the size of the bore hole, a pump cannot be installed within the packer system for water withdrawal. We recognize that methods are available for water extraction under vacuum from the surface, but these approaches would limit the scope and accuracy of what can be accomplished. The proposed injection procedure is believed to provide greater flexibility, and generally better accu-

racy for this testing, particularly in bedrock zones with lower permeability.

Because the injection procedure will be conducted over a relatively short time period, the amount of potable water injected into the formation is expected to be small. However, the total volume of potable water injected into each well tested will be recorded, and if a tested well is sampled for chemical analysis in the future, a similar volume of water will be purged from the well prior to initiating the standard well sampling procedure, Section 2.5 of the AGWS-S Work Plan was revised to include this statement.

b. While the work plan refers to ASTM D4630-96 as the testing method to be followed, a SOP specifically applicable to this project should provided. This should include recording the total volume of water added to each well interval tested.

Group Response:

A field procedure, based on the ASTM method has been added to the FSP. The procedure includes recording of the volume of water added at each interval and the total water added at any given well.

c. The work plan does not specify the depths of bedrock well segments to be tested or the lengths of the segments. Will this information be provided prior to initiating the testing?

Group Response:

The intent of this work will be to test all open rock zones in each of the wells listed in the AGWS-S for testing. However, it may not be feasible to test some intervals based on field observations of the configuration of the open rock segment, casing, etc. The actual dimensions of the bedrock segments tested will be included in the final report.

7. Section 2.6 - Surface Water Sampling

a. Will any screening be done to attempt to identify ground water discharge points? Samples should be collected from near the bottom of the water column (e.g. six inches above the river bottom).

Group Response:

The use of temperature and conductivity profiles vertically and laterally along the edge of the river was considered as a possible methodology for identifying groundwater discharge zones. However, the conductivity and temperatures of groundwater are considered too similar to surface water and the flux of groundwater too low to make this a viable methodology for the CRS Site. On this basis, the collection of multiple grab samples, focusing on areas where groundwater equipotentials indicate potential preferential discharge areas and where groundwater concentrations are highest, is considered the most viable methodology, and this is what has been proposed.

As indicated in this comment, the water samples will be collected near the river bed (approximately six-inches above the river bed). The re-

quired depth at any given location will be determined based on the bathymetric survey and will be recorded with each sample. A more detailed description of the surface water sampling has been added to the FSP.

b. The work plan proposes collecting the samples in collection bottles and then transferring the samples into the appropriate laboratory bottles/vials. While it is stated that care will be taken to minimize exposure to air and the disturbance of the water, the process of transferring will result in some volatilization. Samples should be collected in such a way that volatilization will not occur. This may be done, for example, by using the laboratory bottles/vials as the collection bottle (although preservation (if required) must be done after sample collection) or by collecting the samples using a pump with a short length of tubing.

Group Response:

Based on this comment, surface water sampling will be completed with the actual containers that will be used to ship the samples to the laboratory. For VOC samples, the container will be preserved after the sample is collected.

c. It is stated that "*The sampling procedures and chain-of-custody and shipping protocols will be completed according to the methods detailed in the Site Field Sampling Plan (FSP) (provided with the AGWS Work Plan)…*". There was no surface water sampling procedures provided in the June 2011 Additional Ground Water Studies Work Plan. Will a FSP Addendum be provided?

Group Response:

A section has been added to the FSP (attached) that addresses surface water sampling.

8. Section 3.1 - Information on NAPL Recovery and Measurement Activities.

a. Additional information is needed concerning which wells will be used for NAPL recovery activities. Section 3.1 indicates that *"…NAPL bail-down tests will be conducted to quantify recovery rates and onsite NAPL transmissivities."* (pg. 3-1). However, the wells selected for this activity were not discussed in the submittal. If this information is not known yet, it can be documented in the final report.

Group Response:

Wells MW-6, MW-7A, MW-13A, and MW-14A will be tested. These wells were selected because they contained NAPL following installation and it is assumed that NAPL has re-accumulated since the wells were last sampled. If one or more of these wells no longer contains a measurable quantity of NAPL, testing will not be performed on the well. The wells selected for NAPL recovery will be documented in the final report.

b. In the second paragraph of this section, reference is made to the Field Sampling Plan (FSP). There was no dense NAPL sampling procedures provided in Appendix A: FSP of the June 2011 Additional Ground Water Studies Work Plan. Will a

FSP Addendum be provided with SOPs for collecting NAPL directly and by using absorbent socks?

Group Response:

NAPL sampling procedures have been addressed in previous correspondence for this project (at the time that NAPL sampling was completed in MW-6). The procedures followed previously have been expanded and are now included in the FSP (attached to this AGWS-S Work Plan).

9. Section 3.2 - Bedrock Coring

a. Clarification is needed regarding petrophysical analysis. What properties will be measured (would the testing be limited to pore fluid saturation of NAPLs)? Will a SOP be provided for this testing?

Group Response:

A range of petrophysical parameters will be included in the proposed testing program. These parameters will be defined somewhat by field observations and the fluid saturations observed in the core. The initial petrophysical testing will focus on defining pore fluid saturations, the physical properties of the rock matrix (grain density, porosity, conductivity and intrinsic permeability) and if sufficient fluids are extractable from the core to determine the chemical properties and physical properties of the NAPL. All of the initial testing of the cores will be conducted in accordance with standard methodologies as outlined by ASTM or the API. These initial tests will comprise the following:

- *Grain Density (API RP40)*
- *Total Porosity (API RP40)*
- *Air-Filled Porosity (API RP40)*
- *Effective Porosity (Mod. ASTM D425)*
- *Pore Fluid Saturations (API RP40)*
- *Hydraulic Conductivity (API RP40/EPA 9100)*
- *Intrinsic Permeability (API RP40)*
- *Specific Gravity of Fluids (ASTM D854) or Fluid Density (ASTM D1481)*
- *Viscosity of Fluids (ASTM D445)*

To define the residual saturation (the lowest NAPL saturation achievable through mechanical means) of the rock cores a series of test methods will be employed based on the findings of the initial petrophysical tests. These tests will be conducted similarly in accordance with ASTM and API methodologies used extensively in the petroleum industry. The methods will comprise:

- *Residual Saturation by Direct Centrifuge Method (API RP 40) – this methodology is a centrifuge method (refer to attached API RP40 document) which applies 1000 time gravity for one hour to assess product mobility. The method provides initial and residual pore fluid saturations, total porosity, dry bulk density and grain density measurements. This methodology is an air displacing oil (nonaqueous flu-*

id) methodology and applies high stresses and forces that cannot be replicated under field conditions. On this basis the method is considered highly conservative for assessing the amounts of fluids that could be potentially removed from the core. This method is utilized extensively as a screening methodology based on the ease of testing and low cost.

- *Residual Saturation by LNAPL/Water Imbibition Capillary Pressure Package (API RP40 and ASTM6836) – this also is a centrifuge method but involves the water displacing oil rather than air. The methodology uses a lower range of capillary pressures (typically up to 5 psi) that better reflect the stresses that can be induced by remedial processes. The method is more involved and expensive and is typically used to assess NAPL mobility at and below the water table (where NAPL is displaced by water). The analytical method provides measures of initial and residual fluid saturations, NAPL production versus capillary pressure, total porosity, dry bulk density, specific permeability to LNAPL and hydraulic conductivity.*

This list of petrophysical parameters and a copy of API RP40 has been added to the FSP to detail the standard methodologies to be employed in these analyses. Additional detail on the petrophysical testing has also been added to the AGWS-S in response to this comment.

b. Reference is made in the second paragraph to "the required depth". What is the required depth of the rock coring? If coring goes deeper that than the first impacted interval, how will pull-down be prevented?

Group Response:

The boring and rock coring methods described in the AGWS Work Plan will be followed during implementation of the AGWS-S work, with the exception that wells will not be installed. References have been added to the AGWS-S to indicate this. The intent of this boring program will be to collect whole rock cores from the first interval where NAPL is encountered. This will limit the chance that NAPL will be dragged to a greater depth. The borings will also be grouted upon completion, so there will be little opportunity for NAPL to travel to greater depths in the boring. If insufficient whole core material is obtained from the first interval where NAPL is encountered, additional coring will occur only to the depth necessary to collect the quantity of whole rock core material needed for laboratory testing.

c. The third paragraph mentions that cores will be preserved. What exactly does this mean? Perhaps the applicable SOP may explain this.

Group Response:

The core sampling, preservation and sampling will be conducted in accordance with API RP 40 which has been discussed in detail in the response above and has been added to the FSP.

d. For chemical analysis of the rock core samples, will the rock be crushed prior to analysis (e.g. in the lab)?

Group Response:

Chemical analysis of the rock cores will be conducted to characterize the chemical composition of both mobile and residual NAPL within the rock matrix. These data are critical for determining the dissolution of NAPL into groundwater, the likely fate and transport of NAPL in groundwater and the longevity of the plume.

Subsamples of the cryogenically frozen core will be sent to a chemical testing laboratory, preserved and crushed in the laboratory for chemical extraction and tested in accordance with standard analytical methodologies for VOCs, SVOC's, Volatile Petroleum Hydrocarbons (VPH) and Extractable Petroleum Hydrocarbons (EPH). To assess the potential solubility and mobility of VOC and SVOC compounds contained within the NAPL, the effective solubility of these compounds will be calculated using Raoults Law. The VPH and EPH methodologies and associated carbon chain length concentrations will be used to estimate the average molecular weight of the NAPL and then the mole fraction of VOCs and SVOC's within the NAPL solution.

Both the mass fractions and effective solubility are critical input parameters in the proposed screening level groundwater model that will be included as part of the data assessment and focused feasibility study.

Please contact me at 216-606-1309 if you have any questions regarding the responses provided above or the changes/additions made to the attached AGWS-S.

Sincerely,

Brown and Caldwell

Michael L. Watkins

Michael Watkins, P.G.
Project Manager

cc via email: J. Peeples, BC

Attachment:

Additional Groundwater Studies Supplemental Work Plan (Revision 1 - April 2012)

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

April 23, 2012

REPLY TO THE ATTENTION OF: SR-6J

Mr. Patrick Steerman
Steerman Environmental Management & Consulting, LLC
422 Creek View Lane
Roswell, GA 30075

Re: Chemical Recovery Systems Inc. Site
Second Comments on Additional Groundwater Studies Supplemental Work Plan

Dear Mr. Steerman:

On February 6, 2012, the U.S. Environmental Protection Agency (EPA) and Ohio Environmental Protection Agency (OEPA) received the additional groundwater studies supplemental work plan (AGWS-S) from Steerman Environmental Management & Consulting, representing the Chemical Recovery Systems Remedial Design/Remedial Action Group Performing Parties (Group). This work plan supplements the March 2011 additional groundwater studies work plan (AGWS). The AGWS-S proposes activities to further evaluate the ground water, underlying bedrock, and non-aqueous phase liquids (NAPL) at the site. On March 14, 2012, the Agencies commented on the AGWS-S and the Group responded on April 5, 2012.

Overall those responses are acceptable and satisfactory. Following are several additional comments from the Agencies. Final approval of the plan is pending on the Group revising the AGWS-S and Field Sampling Plan and re-submitting them.

1. Response to Comment #4 on Section 2.3 – Observe Seep Zones
   The details provided regarding the approach planned for quantifying the discharge from the seeps is appreciated. Unfortunately this explanation is not incorporated into the revised work plan or the revised Field Sampling Plan; please include these details. EPA's original comment was referring to the accuracy and frequency of the measurements of the elevation of any active seeps. The Group's Response does not answer this question.

2. Response to Comment # 6.a. on Section 2.5 –Permeability Testing of Bedrock
   The source of and test data on the potable water proposed to be added to the aquifer during the testing procedure needs to be documented in the final AGWS report.

3. Response to Comment # 6.b. on Section 2.5 –Permeability Testing of Bedrock
   Please state the page number of the Field Sampling Plan where this addition was made.

Recycled/Recyclable • Printed with Vegetable Oil Based Inks on 100% Recycled Paper (50% Postconsumer)

4. Response to Comment # 6.c. on Section 2.5 –Permeability Testing of Bedrock
   This comment relates to the depths and lengths of borehole segments to be tested. The explanation provided in this response has not been incorporated into the work plan; please incorporate it. The Field Sampling Plan states that generally 5 or 10 foot test intervals will be used, but no explanation is provided regarding how the lengths will actually be determined. Please provide some explanation.

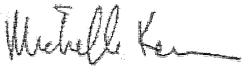
5. Response to Comment 7.a. on Section 2.6 - Surface Water Sampling
   The comment relates to how the surface water sample locations are being determined. The explanation is acceptable; however the revised work plan does not provide this rationale. Please include this in the work plan. The response states that sampling was focusing on areas where groundwater equipotentials indicate preferential discharge areas and where groundwater concentrations are highest. Please provide supporting flow maps and incorporate the rationale into the work plan text.

6. Response to Comment 8.b. on Section 3.1- NAPL Recovery
   While the work plan mentions absorbent socks, the Field Sampling Plan does not. If absorbent socks might be used, the procedures involved should be included; if absorbent socks will not be used, the work plan should not suggest that they might be used.

7. Response to Comment 9.b. on Section 3.2 – Bedrock Coring
   The explanation provided in the response (intent is to collect cores from the first interval where NAPL is encountered) is appreciated. This clarification was not incorporated in the revised work plan and should be added.

Thank you for your attention to these comments. I appreciate the level and quality of work that the Group is producing for this project. If you have any questions or concerns, please contact me at (312) 886-8961.

Sincerely,



Michelle Kerr
Remedial Project Manager

cc via email: L. Antonelli, OEPA
L. Mencin, Sherwin Williams
J. Peeples, B&C
L. Vanderpool, EPA

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

May 14, 2012

REPLY TO THE ATTENTION OF: SR-6J

Mr. Patrick Steerman
Steerman Environmental Management & Consulting, LLC
422 Creek View Lane
Roswell, GA 30075

Re: Chemical Recovery Systems Inc. Site
Approval of Additional Groundwater Studies Supplemental Work Plan

Dear Mr. Steerman:

On February 6, 2012, the U.S. Environmental Protection Agency (EPA) and Ohio Environmental Protection Agency (OEPA) received the additional groundwater studies supplemental work plan (AGWS-S) from Steerman Environmental Management & Consulting, representing the Chemical Recovery Systems Remedial Design/Remedial Action Group Performing Parties (Group). The AGWS-S proposes activities to further evaluate the ground water, underlying bedrock, and non-aqueous phase liquids at the site. On March 14, 2012, the Agencies commented on the AGWS-S and the Group responded on April 5, 2012. We provided a second set of comments to the Group on April 23, 2012, and the Group responded on May 7, 2012.

This constitutes EPA's final approval of the AGWS-S. Please reference and incorporate the flow map that supports the discussion in Section 2.6 into the AGWS-S. EPA and OEPA would appreciate a final paper and electronic copy of the complete revised package.

Thank you for the clarifications and revisions. We look forward to receiving a schedule of field activities. If you have any questions or concerns, please contact me at (312) 886-8961.

Sincerely,

Michelle Kerr
Remedial Project Manager

cc via email: L. Antonelli, OEPA
L. Mencin, Sherwin Williams
J. Peeples, B&C
L. Vanderpool, EPA

Recycled/Recyclable • Printed with Vegetable Oil Based Inks on 100% Recycled Paper (50% Postconsumer)

STEERMAN ENVIRONMENTAL

via first class mail

May 17, 2012

Michelle Kerr
Remedial Project Manager
U.S EPA – Region 5
77 W. Jackson Blvd.
Mail Code: S-6J
Chicago, IL 60604-3590

Re: Final Additional Groundwater Studies Supplemental Work Plan and Technical Impracticability Determination Scope of Work
United States of America v. AK Steel Corporation et. al.
Case No. 1:10-cv-00996-KMO
Chemical Recovery Systems Site, Elyria, Ohio

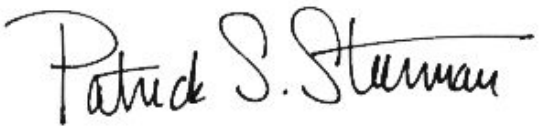
Dear Ms. Kerr:

On February 6, 2012, the Chemical Recovery Systems, Inc. (CRS) Site Remedial Design/Remedial Action (RD/RA) Group Settling Performing Defendants (Group) submitted an Additional Groundwater Studies Supplemental (AGWS-S) Work Plan and, under separate cover, a letter discussing a Technical Impracticability (TI) Determination Scope of Work for the CRS site (the Site). The additional work proposed for the Site was discussed during the November 14, 2011 meeting between the Group and the United States Environmental Protection Agency (USEPA). The need for additional investigation activities discussed in this meeting prompted the February 6, 2012 supplement to the approved March 2011 Additional Groundwater Studies Work Plan (AGWS). In a letter dated March 14, 2012, the USEPA provided questions and comments on the AGWS-S Work Plan and the TI Determination Scope of Work. The questions and comments were reviewed by the Group and addressed in an April 5, 2012 letter prepared by Brown and Caldwell and transmitted by Steerman Environmental Management and Consulting. U.S. EPA provided additional comments on April 23, 2012. These comments were reviewed by the Group and addressed in a May 7, 2012 draft response to comments. U.S. EPA approved the draft response on May 14, 2012. Enclosed are a letter responding to comments provided by U.S. EPA on April 23, 2012 and the final AGWS-S Work Plan with all attachments.

Steerman Environmental
Management & Consulting, LLC
422 Creek View Lane
Roswell, Georgia 30075
(404) 421-3275 - Voice
psteerman@charter.net

If you have any questions regarding the rationale and scope of the proposed TI investigation and assessment activities please feel free to contact the undersigned at (770) 992-2836, or by electronic message to psteerman@charter.net.

Best Regards,



Patrick S. Steerman
CRS Site Project Coordinator

ec: Larry Antonelli, Ohio EPA
Nigel Goulding, EHS Support
Larry Mencin, CRS PRP Group, Technical Committee Chair,
Jim Peeples, Brown & Caldwell
Doug McWilliams, CRS PRP Group Counsel

6055 Rockside Woods Boulevard, Suite 350
Independence, Ohio 44131
Tel: 216-606-1300
Fax: 216-606-1350
www.brownandcaldwell.com

May 17, 2012

Brown AND Caldwell

Patrick S. Steerman
Steerman Environmental Management & Consulting, LLC
422 Creek View Lane
Roswell, Georgia 30075 139452

Subject: Response to USEPA Additional Comments regarding the Revised Additional Groundwater Studies Supplemental Work Plan and Technical Impracticability Determination Scope of Work
United States of America v. AK Steel Corporation et. al.
Case No. 1:10-cv-00996-KMO
Chemical Recovery Systems Site, Elyria, Ohio

Dear Mr. Steerman:

On April 5, 2012, Brown and Caldwell submitted to the Chemical Recovery Systems, Inc. (CRS) Site Remedial Design/Remedial Action (RD/RA) Group Settling Performing Defendants (Group) the Revised Additional Groundwater Studies Supplemental (AGWS-S) Work Plan. The additional work proposed for the Site was discussed during the November 14, 2011 meeting between the Group and the United States Environmental Protection Agency (USEPA). The need for additional investigation activities discussed in this meeting prompted the initial February 6, 2012 supplement to the approved March 2011 Additional Groundwater Studies Work Plan (AGWS). The AGWS-S Work Plan proposes activities to further evaluate the groundwater, underlying bedrock, and non-aqueous phase liquids (NAPL) at the Site; to refine the hydrogeologic conceptual site model; and to finalize the AGWS report. As discussed in the AGWS-S Work Plan, proposed field activities include the collection of Site field data proposed in the Technical Impractability (TI) Determination Scope of Work. In a letter dated March 14, 2012, the USEPA provided questions and comments on the AGWS-S Work Plan and the TI Determination Scope of Work. Group responses to those questions and comments were in Brown and Caldwell's letter dated April 5, 2012. In a letter dated April 23, 2012 the USEPA provided additional comments on the Revised AGWS-S Work Plan. The additional comments are provided below and are followed by responses by the Group shown in italics.

1. Response to Comment #4 on Section 2.3 – Observe Seep Zones

The details provided regarding the approach planned for quantifying the discharge from the seeps is appreciated. Unfortunately this explanation is not incorporated into the revised work plan or the revised Field Sampling Plan; please include these details. EPA's original comment was referring to the accuracy and frequency of the measurements of the elevation of any active seeps. The Group's response does not answer this question.

Group Response:

Section 2.3 in the Revised AGWS-S Work Plan has been expanded to include the discussion presented in the Group's original response letter. As indicated in that response, the intent of this work is to obtain seep flow measurements from all active seeps on a single day, and that only one set of seep flow measurements will be obtained. However, the presence/absence of flow from the identified seeps will be noted during subsequent rounds of water level acquisitions from the Site. The accuracy of the surveyed seep locations will be in accordance with State of Ohio survey standards, which are ±0.1 feet in the horizontal plane and ±0.01 feet in the vertical plane.

2. **Response to Comment #6.a. on Section 2.5 – Permeability Testing of Bedrock**

The source of and test data on the potable water proposed to be added to the aquifer during the testing procedure needs to be documented in the final AGWS report.

Group Response:

As indicated in the second paragraph of Section 2.5 in the Revised AGWS-S Work Plan, any water added to the aquifer during the permeability testing will be derived from a locally available public potable water source, and that the source of the potable water will be included in the final AGWS report. Because this water will be drinking water quality, no additional analysis of the water prior to the permeability testing will be done. However, available water testing results from the local water purveyor (i.e., City of Elyria) obtained during the timeframe of the testing will be requested and included in the final AGWS report. This provision has been added to Section 2.5 in the Revised AGWS-S Work Plan.

3. **Response to Comment #6.b. on Section 2.5 – Permeability Testing of Bedrock**

Please state the page number of the Field Sampling Plan where this addition was made.

Group Response:

A summary of the packer testing procedure to be used at the Site is provided in the Revised Field Sampling Plan (FSP) on Page A-6 in Section 3.1.1.

4. **Response to Comment #6.c. on Section 2.5 – Permeability Testing of Bedrock**

This comment relates to the depths and lengths of borehole segments to be tested. The explanation provided in this response has not been incorporated into the work plan; please incorporate it. The Field Sampling Plan states that, in general, 5- or 10-foot test intervals will be used, but no explanation is provided regarding how the lengths will actually be determined. Please provide some explanation.

Group Response:

The explanation included in the Group's original response has been added to Section 2.5 in the Revised AGWS-S Work Plan. Generally, five-foot long test intervals will be targeted. However, other intervals may be necessary to provide complete coverage of the total interval of bedrock to be tested within a given well. For example, if the total length to be tested is 12 feet, then the testing might consist of two six-foot long test intervals, or one five-foot interval and one seven-foot interval. The actual interval lengths in these situations will be deter-

mined based on review of the boring log, well construction details, etc. This discussion has been added to the summary of the packer testing procedure presented in Section 3.1.1 of the Revised FSP.

5. Response to Comment #7.a. on Section 2.6 – Surface Water Sampling

The comment relates to how the surface water sample locations are being determined. The explanation is acceptable; however the revised work plan does not provide this rationale. Please include this in the work plan. The response states that sampling was focusing on areas where groundwater equipotentials indicate preferential discharge areas and where groundwater concentrations are the highest. Please provide supporting flow maps and incorporate the rationale into the work plan text.

Group Response:

The northern-most proposed surface water sampling location will be upstream of the Site and will be used to provide background data for water quality in the river. The second sampling location will be adjacent to the Site in the area where the bedrock groundwater potentiometric contours, based on review of existing data, appear to converge toward the river. The third location will correspond approximately to the bedrock outcrop at the Site where the potentiometric surface appears to show an alternate flow direction toward the river, and the southern-most (fourth) proposed surface water sampling location will be just downstream of the Site. Additional bedrock potentiometric maps will be provided in the final AGWS report once the supplemental data are evaluated and the maps are finalized.

6. Response to Comment #8.b. on Section 3.1 – NAPL Recovery

While the work plan mentions absorbent socks, the Field Sampling Plan does not. If absorbent socks might be used, the procedures involved should be included; if absorbent socks will not be used, the work plan should not suggest that they might be used.

Group Response:

Bailers and absorbent socks were originally considered as a means to remove NAPL from the wells along with a peristaltic pump and dedicated/disposable tubing. However, the peristaltic pump was selected as the preferred method based on our experience at this and other sites. The reference to absorbent socks has been removed from Section 3.1 of the Revised AGWS-S Work Plan.

7. Response to Comment #9.b. on Section 3.2 – Bedrock Coring

The explanation provided in the response (intent is to collect cores from the first interval where NAPL is encountered) is appreciated. This clarification was not incorporated into the revised work plan and should be added.

Group Response:

The intent of the investigation to collect the cores from the first, or uppermost, interval where NAPL is encountered has been included in Section 3.2 of the Revised AGWS-S Work Plan.

The final AGWS-S Work Plan (Revision 2) with all attachments is enclosed.

Please contact me at 216-606-1309 if you have any questions regarding the responses provided above or the changes/additions made to the attached Revised AGWS-S Work Plan and FSP page.

Sincerely,

Brown and Caldwell

Michael L. Watkins

Michael Watkins, P.G.
Project Manager

Cc via email: J. Peeples, BC

Enclosures:

Additional Groundwater Studies Supplemental Work Plan

Prepared for

CRS Site RD/RA Performing Parties

Chemical Recovery Systems, Inc. (CRS) Site

Elyria, OH

May 2012

Additional Groundwater Studies Supplemental Work Plan

Prepared for
CRS Site RD/RA Performing Parties
Chemical Recovery Systems, Inc. (CRS) Site
Elyria, OH
May 2012

139452

Brown AND Caldwell
4700 Lakehurst Court, Suite 100
Columbus, Ohio 43016

Table of Contents

1. Introduction 1-1
   1.1 Background 1-1
   1.2 Additional Groundwater Studies (AGWS) Conducted in 2011 1-2
   1.3 Objectives 1-3
   1.4 Schedule 1-3
2. Additional Groundwater Studies Supplemental Work 2-1
   2.1 Site Preparation 2-1
   2.2 Obtain Temporal Water Level Data 2-1
   2.3 Observe Seep Zones in Bedrock on the West River Bank 2-2
   2.4 Complete a Bathymetric Survey of the River Bed 2-3
   2.5 Permeability Testing of Bedrock 2-3
   2.6 Surface Water Sampling 2-4
3. Supplemental LNAPL and DNAPL Assessments 3-1
   3.1 Obtain NAPL Recovery Rates from Wells 3-1
   3.2 Bedrock Coring 3-2
4. Additional Groundwater Studies Report 4-1

References REF-1

Figures ..... Follows Text

   Figure 1-1 Site Location

   Figure 1-2 Site Layout

   Figure 1-3 Additional Groundwater Studies Borings/Wells - 2011

   Figure 1-4 Project Schedule

   Figure 2-1 Proposed Boring Locations

Tables ..... Follows Text

   Table 1-1 Groundwater Sampling Results

   Table 1-2 NAPL Sampling Results

Attachment A Field Sampling Plan (Revised May 2012) ..... A

Attachment B Quality Assurance Project Plan (Revised April 2012) ..... B

Section 1
Introduction

This Additional Groundwater Studies (AGWS) Supplemental Work Plan is being submitted on behalf of the Chemical Recovery Systems, Inc. (CRS) Site Remedial Design/Remedial Action (RD/RA) Group Settling Performing Defendants (the Performing Parties) to describe additional work needed to meet a requirement of the Consent Decree; United States of America v. AK Steel Corporation et al., Case No. 1:10-cv-00996 (CD), executed between the U.S. Environmental Protection Agency (USEPA) and the Performing Parties, and entered by the Court on July 16, 2010, along with the Record of Decision (ROD) and the Statement of Work (SOW) for the CRS Site (Site). This AGWS Supplemental Work Plan provides details regarding additional work that is required to finalize the hydrogeologic conceptual site model (CSM) for the CRS Site and to complete the AGWS as specified in the SOW. The work described in this work plan supplements work completed during the summer of 2011 in accordance with the AGWS Work Plan approved by USEPA on June 16, 2011.

In addition to the work required to refine and finalize the CSM, a number of supplemental investigation tasks are included to refine our understanding of contaminant distribution and behavior in the subsurface. These tasks are associated with a parallel program of investigation activities that has been discussed with USEPA to assess the applicability of the Site for a Technical Impracticability (TI) determination for non-aqueous phase liquid (NAPL) in groundwater and bedrock.

Field procedures associated with implementing this AGWS Supplemental Work Plan are described in the Field Sampling Plan (FSP), included as Appendix A to the Remedial Design Work Plan (RDWP) prepared for the Site by Brown and Caldwell, Inc. (BC), dated September 2010 and approved by USEPA in March 2011. Associated analytical and quality assurance/quality control (QA/QC) procedures are described in the Quality Assurance Project Plan (QAPP), included as Appendix B to the approved RDWP. The FSP and QAPP have been revised to address additional field activities described in this AGWS Supplemental Work Plan that were not originally included in those documents. These revised documents are included as Attachments A and B, respectively, to this AGWS Supplemental Work Plan.

1.1 Background

This section contains a summary of Site features and history. A more comprehensive summary is provided in Section 1 of the RD Work Plan that also includes a detailed description of previous investigations completed at the Site.

The Site is located at 142 Locust Street (formerly Maple Street) in Elyria, Lorain County, Ohio (Figure 1-1) in a predominantly industrial area. The Site's approximate latitude is 41 degrees, 22' 14.45" N and its approximate longitude is 82 degrees, 06' 14.8" W. The western boundary of the Site borders the East Branch of the Black River. To the north and east (opposite Locust Street), the Site is bordered by the BASF Corporation (formerly Engelhard Corporation, and prior to that the Harshaw Chemical Company). The M&M Aluminum Siding property to the south was recently acquired by BASF. The Site layout, including on-site features, is presented as Figure 1-2.

The Site has been in industrial use since the late 1800s, and housed a manufactured gas plant (MGP) from 1878 to approximately 1900. Review of Sanborn maps from the area indicate that the MGP was no longer in operation in 1904 and the MGP was not operating in all subsequent Sanborn maps reviewed. A single, circular gas holder was present in the southern-most location noted on Figure 1-2,

and the second gas holder (the northern-most) was noted in Sanborn maps beginning in 1893. The Sanborn maps do not show additional industrial activity at the Site until 1970, when the presence of drums is indicated on the map, after the Obitts Chemical Company began processing spent solvents at this location, which occurred from 1960 to 1974. On January 1, 1974, CRS assumed operation of the Site through a stock purchase agreement with the Obitts Chemical Company. From 1974 to 1981, CRS continued solvent reclamation operations and sales at the Site. The solvents were stored at the Site in 55-gallon drums, above-ground storage tanks, and tanker trucks, and it is alleged that several spills and releases occurred while the Site operated as a solvent processing facility. In 1980, USEPA filed a lawsuit against CRS, requiring the facility owners to abate environmental issues identified at the Site. In response to this lawsuit, CRS ceased the receipt, processing, and storage of spent solvents and removed tanks, drums, and other solvent containers from the Site. A soil removal remedy was conducted by CRS under an Order from USEPA requiring the abatement of impacts related to site operations. CRS ceased operations and filed for bankruptcy circa 1982.

1.2 Additional Groundwater Studies (AGWS) Conducted in 2011

On September 20, 2010, prior to submittal of the initial AGWS Work Plan, BC provided a letter to USEPA proposing groundwater sampling of existing monitoring wells at the Site during September 2010. This work was proposed to update the Site groundwater database prior to completion of the RD, providing a more current assessment of the groundwater conditions. At that time, groundwater had not been sampled at the Site since 2003.

USEPA provided approval of this work and the wells were redeveloped and sampled during September 2010. The groundwater samples were submitted to the laboratory for volatile organic compound (VOC) analysis. During the sampling program, NAPL was detected in well MW-6. The NAPL was composed of both light (LNAPL) and dense (DNAPL) phases. In general, very low concentrations of VOCs were detected in other wells, consistent with past results.

USEPA was notified that the presence of NAPL had been confirmed in well MW-6 and the data were submitted to USEPA for review and discussion. Based on conditions encountered, BC conducted additional work in this area of the Site that included additional well sampling, soil analyses, and installation of soil gas probes. This work was completed during November 2010. The results of this investigation were provided to USEPA for review and discussion and served as the basis for developing the initial AGWS Work Plan that was submitted to USEPA on March 31, 2011 and approved on June 16, 2011.

The approved AGWS Work Plan was implemented during the summer and fall of 2011. Between July 11, 2011 and August 1, 2011, borings and wells were installed in accordance with the work plan with the exception that the deep borings were not installed at proposed locations 7S, X and A (i.e., wells MW-7A, MW-13A, and MW-14A, respectively) due to the observed presence of NAPL in the shallow borings/wells installed at these locations. There was a concern that the shallow zone could not be adequately sealed off in this area (near the former gas holders) and that NAPL might be carried to deeper bedrock zones during the drilling operation. Instead, two borings were advanced between the MW-7A and MW-13A area and the river at locations approved by USEPA. Monitoring wells MW-15A (shallow) and MW-15B (deep) were installed at this alternate location. Figure 1-3 shows the locations of all borings/wells that were installed during the 2011 AGWS work.

All of the Site wells (7 existing wells and 13 new wells) were sampled following the installation and development of the new wells. The groundwater analytical results are provided in Table 1-1. NAPL was observed in wells MW-7A, MW-13A, and MW-14A (see Figure 1-3). Table 1-2 provides analytical results for the NAPL samples collected during the 2011 AGWS work, as well as for samples that were previously

collected from well MW-6. Summary data for the AGWS work was provided to USEPA for review and discussion.

Following completion of the work outlined in the AGWS Work Plan, a meeting between the Performing Parties and USEPA was held on November 14, 2011 to discuss the results of the investigation. The meeting focused on the nature and extent of NAPL found at the Site and the implications that the AGWS results could have on the approved remedy in light of the presence of NAPL in bedrock (resulting from the former MGP that operated at the Site in the late 1800s and early 1900s). Subsequent to the November 14th meeting, the Performing Parties developed additional work that is necessary to further refine the hydrogeologic CSM and to assess whether it is technically practical to remediate the NAPL that was identified in bedrock. The additional work that was developed serves as the basis for this work plan.

The supplemental AGWS work described in this document is designed to complete the refinement of the hydrogeologic CSM in light of observations of NAPL in the sandstone bedrock and our current understanding of groundwater conditions. The supplemental tasks are presented in Section 2. Additional work will be completed to assess the technical practicability of remediating the NAPL/groundwater in bedrock at the Site. Two tasks for this assessment are provided in Section 3 of this Work Plan, and these tasks will be completed along with the AGWS supplemental work described in Section 2.

1.3 Objectives

The objective of the initial AGWS work was to provide additional information regarding the nature and extent of groundwater impact and to meet the conditions detailed in Section II.C. of the SOW that requires the completion of additional groundwater studies. The objective of this AGWS Supplemental Work Plan is to provide information needed to fill data gaps identified while completing the AGWS and to facilitate the development of a comprehensive hydrogeologic CSM for the Site.

1.4 Schedule

The scope of work described in this AGWS Supplemental Work Plan will be performed in accordance with the schedule provided on Figure 1-4. The major milestones of the work are listed below.

- Submit TI Scoping Document — February 6, 2012
- Submit Second Responses to USEPA Comments On the Draft AGWS-S Work Plan — May 7, 2012
- USEPA Approval of AGWS-S Work Plan — May 14, 2012
- Submit Final Copies of AGWS-S Work Plan — May 17, 2012
- Fieldwork Mobilization — May 28, 2012
- Completion of AGWS Supplemental Work — May – July, 2012
- Submit AGWS Report — October 8, 2012

Section 2

Additional Groundwater Studies Supplemental Work

Several tasks will be completed to refine the hydrogeologic CSM and to finalize the AGWS report. These tasks are described below.

2.1 Site Preparation

Prior to initiating the investigative activities outlined in this AGWS Supplemental Work Plan, the following Site preparations will be conducted.

- Subcontract with a State of Ohio licensed driller, surveyor, and analytical laboratory.
- Notify U.S. EPA of the schedule for mobilizing to the Site and for performing the investigative work.
- Notify the adjacent BASF facility of the schedule for performing the work and provide documents and notifications required by the access agreement.
- Identify and mark equipment laydown areas, decontamination areas, and the investigation derived waste (IDW) staging area.

2.2 Obtain Temporal Water Level Data

Monitoring of bedrock groundwater and surface water elevations is necessary to assess groundwater and surface water interactions, the influence of surface water elevation and change on groundwater levels in bedrock, and the rate of response over time. To collect these data, continuous monitoring of groundwater levels and the river stage will be conducted using pressure transducers equipped with data loggers placed at selected locations throughout the Site. Pressure transducers/data loggers will be placed in wells MW-6B, MW-6C, MW-10A, MW-10B, MW-11A, MW-11B, MW-15A, MW-15B, in a stilling well to be installed in the river at the location of the existing gauging rod, and at the storm sewer manhole located near the northeast corner of the Site. An on-site rain gauge will also be installed to provide site-specific rainfall data, and a barometric transducer will be installed in the unsaturated zone of one of the wells to allow barometric corrections of the groundwater level data. The well selected to contain the barometric transducer will be determined in the field at the time the pressure transducers are installed, and is anticipated to be a well that will be not be opened, or opened minimally, during the monitoring period. Placing the barometric transducer within the unsaturated zone of a locked monitoring well provides security while improving barometric data quality by shielding the transducer from large temperature fluctuations. The locations of the monitoring wells, stilling well and barometric transducer well will be illustrated on a Site map included in the final AGWS report.

The water levels will be monitored for a period of at least 30 days with transducer data collection occurring at one-minute intervals. It is anticipated that periods of recharge to groundwater from rainfall at the Site and rising water levels in the river will occur during this time. Data will be downloaded no later than three weeks into the monitoring period to minimize the potential for data loss if transducers fail during the test period. Data will also be obtained from the USGS gauging station located approximately one mile downstream of the Site for the same time period.

The length of the monitoring period is intended to include a number of recharge events that will be used to assess responses to recharge and groundwater/surface water interactions. As part of the assessment process, it will be useful to obtain data from periods when the river reacts to rainfall events that only occur upstream of the Site. Such events will provide opportunities to observe interaction between the bedrock aquifer and the river independent of influence from on-site recharge. Similarly, it may be possible to evaluate on-site recharge events that do not result in significant changes to the water level in the river, and events where the storm sewer may become surcharged with and without river level rises and on-site recharge. Each of these events will be useful in developing a Site hydrogeologic CSM that incorporates interactions between the river and the bedrock groundwater with potential influences from the storm sewer. If, after 30 days, insufficient recharge events occur of the types described above, the monitoring period may be extended to 60 days.

Data from the USGS gauging station downstream of the Site will be uploaded and compared to the river level data obtained from the transducer/data logger. BC will attempt to develop a correlation between the river level data at the Site and the USGS gauging station data to allow extrapolation of historical Site river levels and to evaluate previous relative levels between Site groundwater and the river. It is recognized that the river is likely a gaining stream along certain stretches and a losing stream along other stretches. However, the goal of this work will be to determine if the river is, on average, a gaining stream, as expected, along the stretch of river adjacent to the Site boundary.

2.3 Observe Seep Zones in Bedrock on the West River Bank

Seep zones have been observed on several occasions on bedrock surfaces on the west bank of the Black River across from the CRS Property. The elevation of these seeps is an indication of the elevation of the water table in the bedrock. The current conceptual model was constructed using these observations and shows that groundwater elevations in bedrock are higher than the average water level of the river (making the Black River a gaining stream at this location).

The location and elevation of seep zones on the west river bank will be surveyed and correlated with groundwater elevations on-site, surface water elevations in the river, and the elevation of the base of the river. An elevation mark (paint and/or metal pin) will be placed at each active seep, allowing approximate measurements of the water table elevation in the bedrock outcrops. The seep locations will be surveyed, mapped and the dimensions measured and information recorded in the field notes. In addition, photographs of the observed seeps (if present) will be obtained. The accuracy of the surveyed locations will be ± 0.1 feet in the horizontal and ± 0.01 feet in the vertical.

If possible, the flow from the seep zones observed on the east bank of the river (adjacent to the Site) will be quantified to provide additional evidence of hydraulic conductivity and the rate of groundwater flow from bedrock. If seepage is not measurable at a location, qualitative observations will be made for comparison to estimates of groundwater flux obtained from other Site data. The accuracy of the measurements made will depend on the ability to account for all the flow from a given seep. It should be understood that, to date, the seeps have been observed as wet spots with minimal to no observable water flow or dripping. If no water flow is observed at a given seep, the maximum rate of flow will be estimated as the rate of evaporation from the wet rock surface, given the environmental conditions at the time of the observation. If observable flow is present it will be directed, as much as is reasonably possible, to a central location where it will be quantified based on the rate of filling a known container volume. Under the appropriate circumstances, these measurements can be quite accurate. If accurate flow rate measurements cannot be obtained at a given location, this result will be documented.

The intent of this work is to obtain seep flow measurements from all active seeps on the east river bank on a single day, and that only one set of seep flow measurements will be obtained. These estimates of seep flows will be compared and contrasted to the Darcian mass fluxes calculated from the on-site

bedrock permeability testing in order to estimate (for the purposes of the hydrogeologic conceptual site model) the potential bank and basal discharge of groundwater to the river. It is anticipated that collectively, seeps from the Site only contribute a very small component of the groundwater flow to the river.

2.4 Complete a Bathymetric Survey of the River Bed

During the previous AGWS work, the river depth was measured in a transect near the north end of the Site to determine if the base of the river was composed of bedrock, rock rubble, or finer grained material. The supplemental work will complete a survey of the base of the river between the north and south boundaries of the Site. The information from this survey will be used with the stream and groundwater elevation measurements and seep data to complete a detailed hydrogeologic cross section that will address the following:

1. Groundwater elevation in the shallow and deeper bedrock wells and temporal variability in groundwater elevations;
2. River surface water levels and temporal variability in these levels;
3. Seep locations and elevations relative to groundwater and surface water;
4. The base of the River relative to groundwater and seep elevations; and
5. Intervals of higher densities of Site bedrock fracturing as they relate to the features described above.

The river bed topography will be characterized using a grid of survey points placed approximately 10-feet on center through the study area (from the east river bank to the west bank and at least to the north and south boundaries of the Site). For each survey point, an observation will be made regarding the presence of a hard bottom, gravel/rubble, or a soft bottom, indicating the nature of the river base material at each location. This determination will be made with a survey rod that has a “shoe” (to prevent it from sinking into the alluvial soil) to define the presence and upper elevation of alluvial materials and a survey road with a point (advanced to refusal) to define the upper surface of the bedrock. This will allow for measurement of the rock and alluvial surfaces, and the thickness of alluvial materials, where present, through the study area. The survey will be completed from a small boat. Interpolation will be completed between points to provide a topographic map the base of the river. This work will result in Site maps and cross sections that include the Site ground surface, the bedrock zones identified by the on-site borings, and the river base.

2.5 Permeability Testing of Bedrock

Hydraulic conductivity/permeability tests of the Site bedrock will be completed in order to determine the conductivity of the bedrock to both NAPL and water in pore spaces and in fracture zones. The intent of this work will be to test all open rock zones in each of the wells intended for testing. However, it may not be feasible to test some intervals based on field observations of the configuration of the open rock segment, casing, etc. The actual dimensions of the bedrock segments tested will be included in the final AGWS report. Permeability, or packer, tests will be performed on the open rock sections in bedrock wells, with pneumatic packers used to isolate a specific segment of the borehole for testing. The specific segments of each boring will be chosen based on boring logs, with intervals targeted to cover a wide range of conditions, from unfractured zones to zones with relatively dense fracturing. The tested interval will generally be approximately five feet in length, although other test intervals may be necessary in some instances to provide complete coverage of the length to be tested for a given location. The procedure for conducting the packer testing is described below.

The packer assembly will consist of a pipe with an open end positioned between an inflatable rubber packer and the bottom of the borehole, or will be a perforated pipe positioned between two packers (upper and lower). A non-perforated pipe extends from the upper packer to the surface. The packer test interval is generally five feet, although other packer spacing may be used. The rubber packers are inflated with compressed nitrogen to seal off the test zone. Potable water from the surface is pumped at a constant, known pressure into the test section. The volume of water pumped is measured through time and recorded. The rate of water entry into the bedrock (under the specified pressure) is a function of hydraulic conductivity. The potable water used for this testing will be from a locally available public water source. The source of the potable water will be included in the final AGWS report. Analytical data associated with the potable water will be requested from the local potable water purveyor (Elyria Water Company) and included in the final AGWS report.

It is recognized that alternative down-hole hydraulic conductivity testing procedures are available that do not rely on injecting potable water into the test intervals. However, based on the relatively small diameter of the boreholes and the relatively low hydraulic conductivities expected, the procedure proposed herein is considered to be the most appropriate. Because the injection procedure will be conducted over a relatively short time period, the amount of potable water injected into the formation is expected to be small. However, the total volume of potable water injected into each well tested will be recorded, and if a tested well is sampled for chemical analysis in the future, a similar volume of water will be purged from the well prior to initiating the standard well sampling procedure,

Standard methods for completion and analysis of the tests, described in ASTM D4630–96 and included in Attachment A of the FSP, will be followed. The maximum test pressures will be determined prior to testing by multiplying the depth to the top of the test interval in feet by a factor of 0.7. This value, in pounds per square inch (psi), will be the maximum pressure applied during the test at that interval. For example, if the top of the test interval is 10 feet below ground surface, a pressure of 7 psi will be used. Higher pressures have the potential to "lift" the tested rock, thus providing results that are not representative.

The duration of a single test will generally be five (5) minutes. If during the five minute test period less than 0.1 gallons of water have flowed into the formation, the test will be continued for an additional five (5) minutes to provide better resolution of low conductivity intervals. All materials and equipment required to complete the packer testing will be provided by a contracted driller, and all tests will be conducted under the observation of a qualified hydrogeologist, who will be responsible for directing the test, recording the data, and ensuring that the tests are conducted appropriately.

Only wells that do not contain NAPL will be used for this testing. The nine wells likely to be tested include MW-6RA, MW-6RB, MW-6RC, MW-10A, MW-10B, MW-11A, MW-11B, MW-15A, and MW-15B, although not all portions of the bedrock in each well will be tested. This work will provide a representation of various conditions in the bedrock, including sandstone with no fractures (primary porosity), moderately fractured sections of sandstone, and more heavily fractured zones. Data from this testing will be used in conjunction with other methods to estimate the bedrock permeability, to better characterize the Site hydrogeology, and for use in future modeling efforts that may be conducted to support the TI evaluation.

2.6 Surface Water Sampling

To further refine the understanding of groundwater/surface water interactions, surface water samples will be collected from the river to assess the potential mass flux of constituents (or lack of) relative to the groundwater fluxes quantified above and will supplement data collected as part of the Remedial Investigation (RI).

Surface water samples will be collected from the river at the four (4) locations shown on Figure 2-1. The river will be sampled at an upstream location, two (2) locations adjacent to the Site, and one (1) location

downstream of the Site. The northern-most proposed surface water sampling location will be upstream of the Site and will be used to provide background data for water quality in the river. The second sampling location will be adjacent to the Site in the area where the bedrock groundwater potentiometric contours, based on review of existing data, appear to converge toward the river. The third location will correspond approximately to the bedrock outcrop at the Site where the potentiometric surface appears to show an alternate flow direction toward the river, and the southern-most (fourth) proposed surface water sampling location will be just downstream of the Site.

The four (4) locations will be sampled three (3) times during the field investigation, with a separation of at least a week between each sampling event. The samples will be collected at three (3) river stages, including low pool stage, at a river level approximately six inches above low pool, and at approximately one foot above low pool elevation. Low pool elevation will be estimated based on the transducer data described in Section 2.2 above. The primary purpose of the sampling will be to confirm the results obtained during the RI that VOCs or semi-volatile organic compounds (SVOCs) in the groundwater at the Site are not impacting the river.

Surface water samples will be collected at a distance of eight (8) feet from the east bank of the river and from a depth of six inches above the river bed. Samples will be collected directly into the appropriate laboratory bottles by attaching each unpreserved sample bottle to an extension pole, then lowering the pole/bottle below the water surface with the open end of the bottle facing down to limit the entrance of water until it reaches the desired sampling depth. This procedure will minimize disturbance of the water that enters the bottle and ensure the collection of representative samples. The bottle will be lowered to approximately six inches above the river bed. The pole/bottle will then be slowly rotated to allow water to enter. When the bottle is fully rotated and is full of river water, it will be raised to the surface and retracted back to the bank. The VOC sample bottles requiring pH adjustment will be immediately preserved upon retrieval of the sample bottle.

The sampling procedures and chain-of-custody and shipping protocols will be completed according to the methods detailed in Section 5.3.3 of the FSP, and the samples will be handled and analyzed in the laboratory in accordance with the methods detailed in the QAPP.. The extension pole will be decontaminated between sample locations using the procedures described in Section 5.6 of the FSP.

Section 3

Supplemental LNAPL and DNAPL Assessments

As discussed in Section 1, additional investigation activities will be conducted to refine our understanding of contaminant distribution in the subsurface and provide further data to support the potential applicability of a TI waiver for NAPL and groundwater at this site. This scope of work includes two (2) main components designed to address the following three (3) objectives:

1. To obtain further information on the mobility and recoverability of LNAPL and DNAPL in the subsurface;
2. To refine our understanding of LNAPL and DNAPL distribution in the subsurface to better understand the controls on LNAPL and DNAPL mobility; and
3. To estimate the total mass of NAPL present in the formation.

Further investigations (beyond those described in the work plan) may be required to characterize the nature and distribution of LNAPL and DNAPL in the subsurface, NAPL interactions with groundwater and amenability of the NAPL to cleanup. These tasks will be further developed following completion and approval of the CSM by USEPA.

3.1 Obtain NAPL Recovery Rates from Wells

To date, borings and wells placed in areas where NAPL was found have shown limited NAPL accumulation and low recovery rates. This is consistent with the evolving NAPL conceptual site model. The highest NAPL saturations and greatest accumulations in wells are observed in wells closest to the former source area (the former gas holders). The focus of this task is to provide additional lines of evidence and quantitative data to support these observations.

NAPL bail-down tests will be conducted to quantify recovery rates and on-site NAPL transmissivities. The four wells to be tested include MW-6, MW-7A, MW-13A, and MW-14A. These wells were selected because they contained NAPL following installation, and NAPL may have re-accumulated since they were last sampled. If a target well no longer contains a measurable quantity of NAPL, testing will not be performed on that well. The tests will include the manual removal of NAPL from each well and then measurement (over time) of NAPL re-accumulation. Measurement of NAPL will occur immediately following NAPL removal on the first day, then weekly for a period of approximately one month. During removal, samples of the NAPL will be collected for laboratory analysis of density, viscosity (at average groundwater temperatures), VOCs and SVOCs, and average molecular weight. As with soil and groundwater samples, the NAPL samples will be packed and stored on ice to minimize the potential loss of volatile constituents, and handled in accordance with the procedures described in Section 5.3.4 of the FSP. If insufficient NAPL volumes are present for density and viscosity tests, then samples will only be analyzed for chemical properties.

Information from these analyses will be used to further assess the mobility and solubility of the NAPL constituents, and to quantify the potential mass flux of chlorinated and non-chlorinated species from the NAPL to groundwater.

3.2 Bedrock Coring

The ability to remove NAPL that is held within the bedrock pores is an important consideration in the evaluation of the practicability of remediating the NAPL at this Site. To better evaluate the potential to remove the NAPL and to facilitate better quantification of NAPL mass, bedrock cores will be collected from within the area where NAPL is likely to be found based on previous borings. Rock cores will be obtained from three borings placed at the locations shown on Figure 2-1 (borings I, J and K). The cores from these borings will be inspected in the field to identify zones where NAPL is present. Core segments from these zones will be sent to laboratories for chemical and petrophysical analysis.

Prior to rock coring, a boring will be advanced with air rotary methods through the overburden soils and into the top two to three feet of bedrock or until competent sandstone is encountered. A five-inch diameter steel casing will be set through the overburden into the upper portion of the bedrock. The steel casing will be grouted in place with a mixture of cement and bentonite to seal off the overburden from the rock. After the grout has set for at least 24 hours, the boring will be advanced inside the steel casing using a 94 millimeter diameter core barrel advanced by wireline. Rock cores will be collected continuously to the required depth, in five (5) foot intervals, and brought to the surface for inspection by the geologist. The geologist will log the core for features such as fractures and will identify zones where NAPL impact may be present. The intent of the coring is to collect a core containing the first encountered, or uppermost, zone of NAPL. Additional coring deeper into the bedrock may occur if insufficient NAPL impact is found within the first five feet, or to determine the depth at which the NAPL impact ends. The corehole will be grouted to grade immediately upon completion of coring in order to minimize the potential for NAPL to cross-contaminate a deeper unimpacted zone. The bedrock coring procedures will be consistent with those described in the AGWS Work Plan and Section 3.1.1 of the FSP, except that monitoring wells will not be installed during this supplemental work.

The rock cores will be photographed and portions of the cores will be selected for laboratory analysis. Sections of the cores will be immediately preserved for chemical and petrophysical testing. Petrophysical testing will follow standard procedures described in various published ASTM and American Petroleum Institute (API) methods used extensively in the petroleum industry. Specific tests and test methods are included in Attachment A of the FSP. In general, laboratory testing will consist of the following:

1. Chemical analysis of the mass of VOCs, fractionated volatile petroleum hydrocarbons (VPH), and fractionated extractable petroleum hydrocarbons (EPH); and
2. If sufficient LNAPL or DNAPL is present in the core sample, pore fluid saturation of DNAPL and LNAPL will be quantified using petrophysical (centrifuge based) extraction and quantification methodologies.

If during the process of petrophysical testing, sufficient NAPL is extracted from the core, chemical analyses will be conducted on the LNAPL and DNAPL portions of the samples to assess the differences in the chemical properties of LNAPL and DNAPL sources within the bedrock, and these will be compared to similar properties observed in NAPL samples obtained from wells. Based on the principles of NAPL mobility, the less mobile NAPL fractions may be present in bedrock, with the samples of NAPL collected from wells likely containing the less viscous and more mobile NAPL fractions. The sections of rock core sent to the laboratory for chemical analysis will be crushed in the laboratory to the appropriate size for analysis.

Following collection of the rock cores, the steel casings will either be pulled from the ground or will be cut off below the surface. The cored hole in the rock will be filled with tremied-bentonite to a point above the soil/bedrock interface, and the overburden hole (or steel casing if the casing is left in place) will be grouted to the ground surface.

Section 4

Additional Groundwater Studies Report

The AGWS report will include the methods and results of the work completed under the approved AGWS Work Plan (2011 work) and the work described in this AGWS Supplemental Work Plan. A detailed Site CSM will be developed that will focus on the hydrogeologic framework (groundwater flow, and groundwater/river interactions), the distribution of contaminants (both NAPL and dissolved phases) within this hydrogeologic framework and the associated potential lateral and vertical mass flux components. The final CSM will describe the Site in sufficient detail to allow the formation of a supplemental investigation program (if required) to complete an evaluation of the technical practicability of achieving the specific applicable or relevant and appropriate requirements (ARARs) provided in the ROD within a reasonable time frame.

As part of the CSM development, the following information will be provided in the Additional Ground Water Studies Report following completion of the supplemental field investigation activities and data analysis described in this Work Plan.

- Summary of investigative means and methods
- Deviations from the scope outlined in this AGWS Work Plan and this AGWS Supplemental Work Plan
- Photographic summary
- Field notes
- Tabulated field and analytical data
- Boring logs and well construction diagrams
- Geophysical raw data and interpretation
- Survey data
- Potentiometric surface maps for bedrock
- Geologic cross sections including bedrock elevations in relation to the Black River bed to assess the potential for cross-river transport
- Aquifer potentiometric and river water level evaluations to assess the potential for cross-river transport
- Definition of NAPL and groundwater impact in the sandstone bedrock
- Data interpretation and conclusions

The AGWS Report will be submitted according to the schedule provided on Figure 1-4.

References

Brown and Caldwell, 2011. Additional Groundwater Studies Work Plan. June, 2011. Approved by USEPA on June 16, 2011.

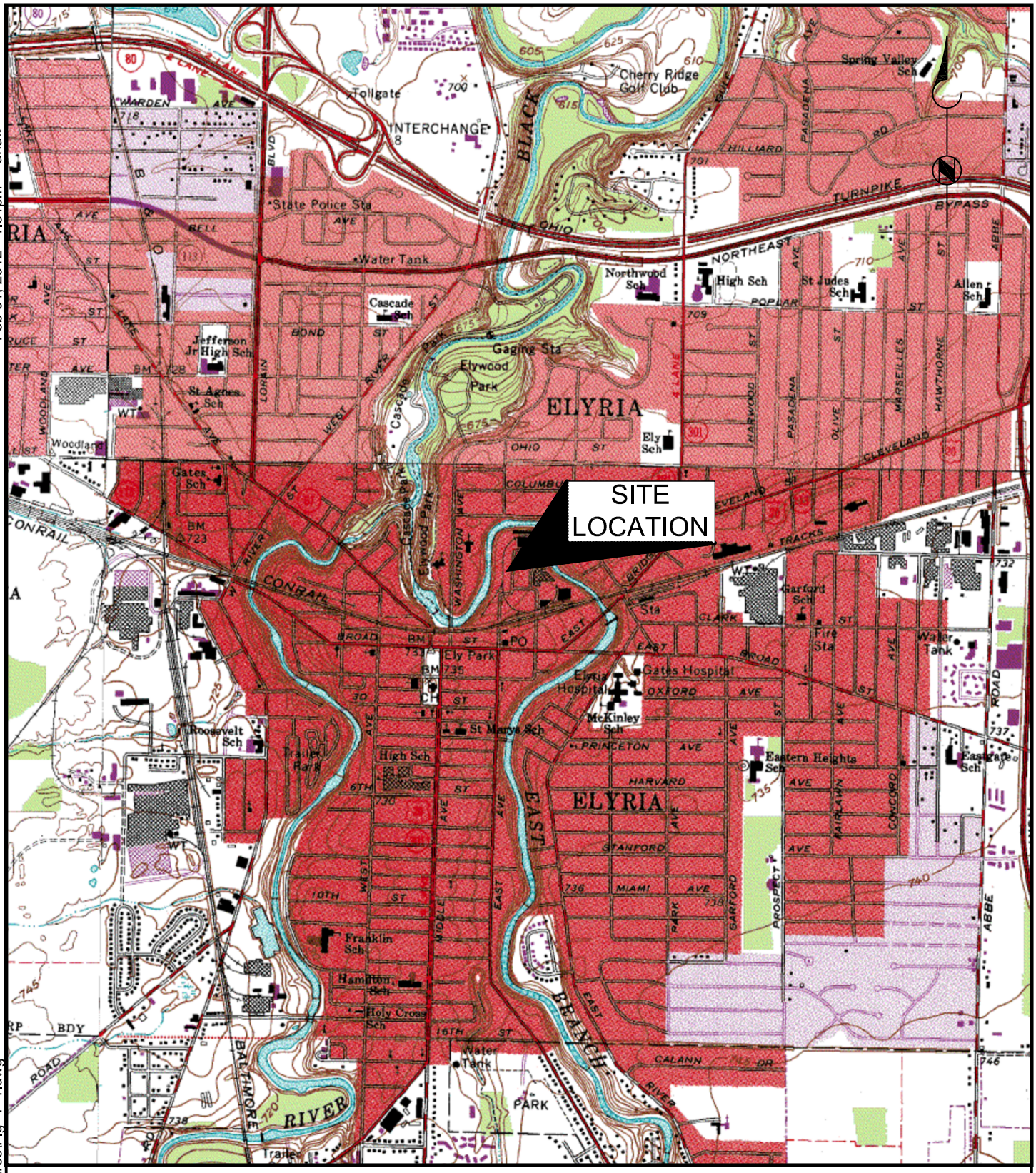
CRS, 2010. Statement of Work for the Remedial Design and Remedial Action at Chemical Recovery Systems Inc. Superfund Site, Elyria, Lorain County, Ohio. February 2010.

USDC, 2010. RD/RA Consent Decree (Consent Decree 1:10-cv-00996) between the United States of America and the Settling Defendants and Settling Owner Defendants, CRS Site, Elyria, Ohio. Entered July 18, 2010.

USEPA, 2007. Record of Decision, Chemical Recovery Systems, Elyria, Lorain County, Ohio. USEPA Region 5. October 2007.

Figures

Figure 1-1 Site Location
Figure 1-2 Site Layout
Figure 1-3 Additional Groundwater Studies Borings/Wells - 2011
Figure 1-4 Project Schedule
Figure 2-1 Proposed Boring Locations



CHEMICAL RECOVERY SYSTEMS, INC. SITE
ELYRIA OHIO

SOURCE: BASE MAP USGS 7.5 MIN. QUAD., AVON, OH, 1994

2000 1000 0 2000

SCALE IN FEET

OHIO
QUADRANGLE LOCATION
LATITUDE 41° 22' 15"
LONGITUDE 82° 06' 12"

Brown AND Caldwell

Figure 1-1
SITE LOCATION

Feb 01, 2012 - 1:51pm ahutt

O:\CRS\Drawings\CAD\Figures\Fig_1_1.dwg

EAST BRANCH BLACK RIVER

FLOW

EAST BANK HIGH WATER MARK

TYPICAL EDGE OF WATER

STAFF GUAGE
BM=687.00'

SW/SD-1 SW/SD-2 SW/SD-3 SW/SD-4 SW/SD-5

TOP OF BANK

HA-1 HA-2 HA-3 HA-4 HA-5 HA-6 HA-7

SS-1 SS-2 SS-3 SS-4 SS-5

CLAY PIPE SW SAMPLE

MW-1 MW-4 MW-5 MW-6 MW-6R (BORING 6R) MW-7A MW-7D MW-8D MW-9D MW-10A (BORING B) MW-10B (BORING F) MW-11A (BORING E) MW-11B (BORING D) MW-12 (BORING C) MW-13A (BORING X) MW-14A (BORING A) MW-15A MW-15B MW-16

GP-1 GP-2 GP-3 GP-4 GP-5 GP-6 GP-7 GP-8 GP-9 GP-10 GP-11 GP-12 GP-13 GP-14 GP-15 GP-16 GP-17 GP-18 GP-20 GP-21 GP-22 GP-23 GP-24 GP-25 GP-26 GP-27 GP-28 GP-29 GP-30 GP-31 GP-32 GP-33 GP-34 GP-35 GP-36 GP-37 GP-38 GP-39 GP-40 GP-41 GP-42 GP-43 GP-44 GP-45 GP-46 GP-47 GP-48 GP-49 GP-50 GP-51 GP-52 GP-53 GP-54 GP-55 GP-56 GP-57 GP-58 GP-59 GP-60 GP-61 GP-62 GP-63 GP-64 GP-65 GP-66 GP-67 GP-68 GP-69 GP-70 GP-71 GP-72 GP-73 GP-74 GP-75 GP-76 GP-77 GP-78 GP-79 GP-80 GP-81 GP-82 GP-83 GP-84 GP-85 GP-86 GP-87 GP-88 GP-89 GP-90 GP-91 GP-92 GP-93 GP-94 GP-95 GP-96 GP-97 GP-98 GP-99 GP-100 GP-101 GP-102 GP-103 GP-104 GP-105

ST-1 ST-2

FORMER TANKER STAGING AREA 1

FORMER TANKER STAGING AREA 2

FORMER DRUM STORAGE AREA 1

FORMER DRUM STORAGE AREA 2

FORMER DRUM STORAGE AREA 3

FORMER GAS HOLDER LOCATIONS

FORMER GAS HOLDER PIT

FORMER BRIGHTEN STILL BUILDING LOCATION

FORMER ABOVE-GROUND STORAGE TANK AREA

FORMER BUILDING LOCATION

FORMER RODNEY HUNT STILL BLDG. LOCATION

ABANDONED RAILROAD TRACK

WAREHOUSE AND OFFICE

M & M ALUMINUM SIDING

RODNEY HUNT STILL BUILDING

EXPOSED FOUNDATION FLUSH WITH GROUND

WOODED

CONCRETE

ASPHALT

GRAVEL

6' FENCE

6' CHAIN LINK FENCE

10' CHAIN LINK FENCE

GATE

6' C.L.F. GATE

LOCUST STREET (FORMERLY MAPLE STREET)

JOHN STREET

LEGEND:

- SITE BOUNDARY LINE
- FENCE LINE
- ROAD CENTERLINE
- EXISTING STRUCTURES
- EXISTING SITE FEATURES
- FORMER STRUCTURE LOCATIONS
- FORMER STORAGE AREAS
- STORM SEWER LINE
- SANITARY SEWER LINE
- COMBINED SEWER LINE
- WATER LINE
- GAS LINE
- OVERHEAD ELECTRIC LINE
- OVERHEAD TELEPHONE LINE
- CATCH BASIN
- STORM SEWER MANHOLE
- FORMER SUMP
- ABANDONED MONITORING WELL
- MONITORING WELL
- SEDIMENT/SURFACE WATER SAMPLE
- RI SOIL BORING
- SOIL BORING WITH GROUNDWATER SAMPLE
- SHELBY TUBE BORING
- HAND AUGER BORING
- SOIL SAMPLE
- 2010/2011 SOIL BORING

0 20 40
SCALE IN FEET

Brown AND Caldwell
COLUMBUS, OHIO

SUBMITTED: _____ DATE: _____ (PROJECT MANAGER)
APPROVED: _____ DATE: _____ (BROWN AND CALDWELL)

LINE IS 2 INCHES AT FULL SIZE (IF NOT 2" - SCALE ACCORDINGLY)
DESIGNED:
DRAWN: ADH
CHECKED:
CHECKED:
APPROVED: JP

EXTERNAL REFERENCE FILES

REVISIONS					
ZONE	REV.	DESCRIPTION	BY	DATE	APP.

CHEMICAL RECOVERY SYSTEMS SITE
ELYRIA, OHIO

SITE BASEMAP

FILENAME: site basemap.DWG
BC PROJECT NUMBER: XXXXXX
CLIENT PROJECT NUMBER:
FIGURE NUMBER: 1-2

O:\CRS\DRAWINGS\CAD\FIGURES\9-09-2011 FIGURES PDF\SITE BASEMAP.DWG January 30, 2012

January 30, 2012

EAST BRANCH BLACK RIVER

FLOW

EAST BANK HIGH WATER MARK

TYPICAL EDGE OF WATER

STAFF GUAGE
BM=687.00'

SW/SD-1 SW/SD-2 SW/SD-3 SW/SD-4 SW/SD-5

TOP OF BANK

HA-1 HA-2 HA-3 HA-4 HA-5 HA-6 HA-7

SS-1 SS-2 SS-3 SS-4 SS-5

CLAY PIPE SW SAMPLE

MW-15A MW-15B MW-16 MW-7A MW-7D MW-8D MW-9D MW-10A (BORING B) MW-10B (BORING F) MW-11A (BORING E) MW-11B (BORING D) MW-12 (BORING C) MW-13A (BORING X) MW-14A (BORING A) MW-1 MW-3 MW-4 MW-5 MW-6 MW-6R (BORING 6R)

GP-1 GP-2 GP-3 GP-4 GP-5 GP-6 GP-7 GP-8 GP-9 GP-10 GP-11 GP-12 GP-13 GP-14 GP-15 GP-16 GP-17 GP-18 GP-19 GP-20 GP-21 GP-22 GP-23 GP-24 GP-25 GP-26 GP-27 GP-28 GP-29 GP-30 GP-31 GP-32 GP-33 GP-34 GP-35 GP-36 GP-37 GP-38 GP-39 GP-40 GP-41 GP-42 GP-43 GP-44 GP-45 GP-46 GP-47 GP-48 GP-49 GP-50 GP-51 GP-52 GP-53 GP-54 GP-55 GP-56 GP-57 GP-58 GP-59 GP-60 GP-61 GP-62 GP-63 GP-64 GP-65 GP-66 GP-67 GP-68 GP-69 GP-70 GP-71 GP-72 GP-73 GP-74 GP-75 GP-76 GP-77 GP-78 GP-79 GP-80 GP-81 GP-82 GP-83 GP-84 GP-85 GP-86 GP-87 GP-88 GP-89 GP-90 GP-91 GP-92 GP-93 GP-94 GP-95 GP-96 GP-97 GP-98 GP-99 GP-100 GP-101 GP-102 GP-103 GP-104 GP-105

FORMER DRUM STORAGE AREA 1

FORMER TANKER STAGING AREA 1

FORMER TANKER STAGING AREA 2

FORMER GAS HOLDER LOCATIONS

FORMER DRUM STORAGE AREA 2

FORMER DRUM STORAGE AREA 3

FORMER BRIGHTEN STILL BUILDING LOCATION

FORMER ABOVE-GROUND STORAGE TANK AREA

FORMER BUILDING LOCATION

FORMER GAS HOLDER PIT

EXPOSED FOUNDATION FLUSH WITH GROUND

FORMER RODNEY HUNT STILL BUILDING LOCATION

ABANDONED RAILROAD TRACK

WOODED

CONCRETE

WAREHOUSE AND OFFICE

M & M ALUMINUM SIDING

RODNEY HUNT STILL BUILDING

ASPHALT

GRAVEL

CONCRETE

6' FENCE

6' CHAIN LINK FENCE

10' CHAIN LINK FENCE

GATE

6' C.L.F. GATE

LOCUST STREET (FORMERLY MAPLE STREET)

JOHN STREET

LEGEND:

- SITE BOUNDARY LINE
- FENCE LINE
- ROAD CENTERLINE
- EXISTING STRUCTURES
- EXISTING SITE FEATURES
- FORMER STRUCTURE LOCATIONS
- FORMER STORAGE AREAS
- STORM SEWER LINE
- SANITARY SEWER LINE
- COMBINED SEWER LINE
- WATER LINE
- GAS LINE
- OVERHEAD ELECTRIC LINE
- OVERHEAD TELEPHONE LINE
- CATCH BASIN
- STORM SEWER MANHOLE
- FORMER SUMP
- ABANDONED MONITORING WELL
- MONITORING WELL
- NEW BORING/WELL

C:\CRS\DRAWINGS\CAD\FIGURES\8-09-2011 FIGURES PDF\ADDITIONAL WELLS_2011.DWG January 30, 2012

January 30, 2012

Brown AND Caldwell
COLUMBUS, OHIO

SUBMITTED: _____ DATE: _____
PROJECT MANAGER

APPROVED: _____ DATE: _____
BROWN AND CALDWELL

LINE IS 2 INCHES AT FULL SIZE (IF NOT 2" - SCALE ACCORDINGLY)

DESIGNED:
DRAWN: ADH
CHECKED:
CHECKED:
APPROVED: JP

EXTERNAL REFERENCE FILES

REVISIONS					
ZONE	REV.	DESCRIPTION	BY	DATE	APP.

0 20 40
SCALE IN FEET

CHEMICAL RECOVERY SYSTEMS SITE
ELYRIA, OHIO

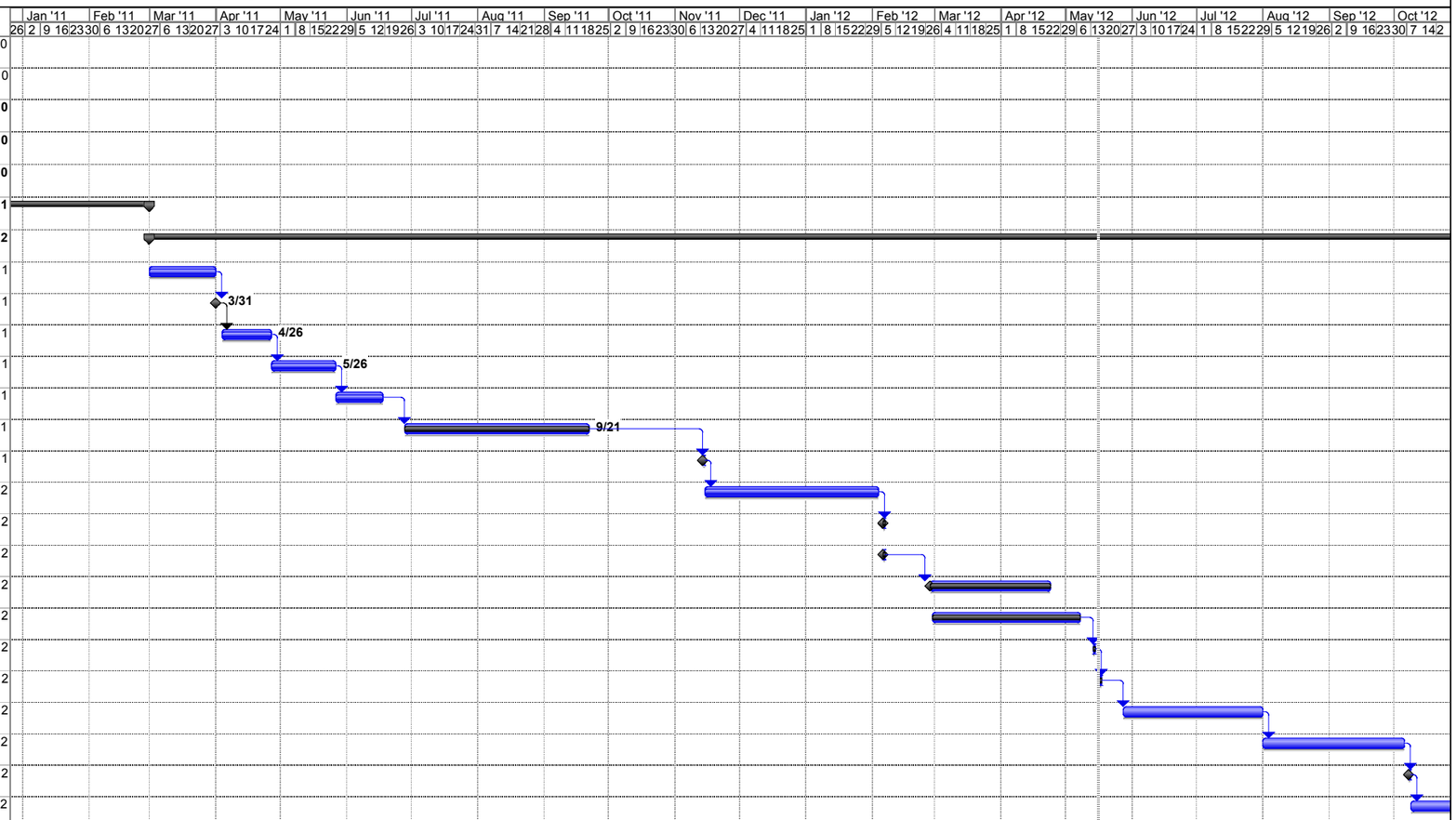
ADDITIONAL GROUNDWATER STUDIES BORINGS / WELLS - 2011

FILENAME: additional wells_2011.DWG
BC PROJECT NUMBER: XXXXXX
CLIENT PROJECT NUMBER:
FIGURE NUMBER: 1-3

Brown AND Caldwell

Figure 1-4
Project Schedule - Remedial Design/Remedial Action
Chemical Recovery Systems Superfund Site - Elyria, Ohio

ID	Task Name	% Complete	Start	Finish
1	Consent Decree (CD) Lodged	100%	Wed 4/28/10	Wed 4/28/10
2	CD Entered by Court	100%	Thu 7/22/10	Thu 7/22/10
3	**Supervising Contractor Selection**	**100%**	**Wed 6/2/10**	**Thu 7/22/10**
7	**Institutional Control Work Plan**	**100%**	**Wed 6/2/10**	**Thu 8/5/10**
12	**Implement IC Work Plan**	**100%**	**Thu 8/5/10**	**Wed 10/20/10**
18	**Remedial Design Work Plan and Associated Plans**	**100%**	**Thu 7/22/10**	**Mon 2/28/11**
28	**Additional Groundwater Studies**	**71%**	**Tue 3/1/11**	**Wed 11/7/12**
29	Prepare Draft GW Studies Work Plan	100%	Tue 3/1/11	Thu 3/31/11
30	Submit to USEPA	100%	Thu 3/31/11	Thu 3/31/11
31	USEPA Review of Draft GSWP	100%	Mon 4/4/11	Tue 4/26/11
32	Revise GSWP per USEPA Comments	100%	Wed 4/27/11	Thu 5/26/11
33	USEPA Review and Approval	100%	Fri 5/27/11	Fri 6/17/11
34	Field Work	100%	Tue 6/28/11	Wed 9/21/11
35	Meeting with USEPA	100%	Mon 11/14/11	Mon 11/14/11
36	Prepare AGWS Supplemental Work Plan	100%	Tue 11/15/11	Fri 2/3/12
37	Submit AGWS Supplemental Work Plan	100%	Mon 2/6/12	Mon 2/6/12
38	Submit TI Scoping Document	100%	Mon 2/6/12	Mon 2/6/12
39	USEPA Review and Provide Comments on AGWS Supplemental Work Plan	100%	Tue 2/28/12	Mon 4/23/12
40	Response to USEPA Comments and AGWS Supplemental Work Plan Revisions	100%	Wed 2/29/12	Mon 5/7/12
41	Approval of AGWS Supplemental Work Plan	100%	Mon 5/14/12	Mon 5/14/12
42	Submit Revised AGWS Supplemental Work Plan	100%	Thu 5/17/12	Thu 5/17/12
43	Complete AGWS Supplemental Field Work	0%	Mon 5/28/12	Tue 7/31/12
44	Prepare AGWS Report	0%	Wed 8/1/12	Fri 10/5/12
45	Submit AGWS Report	0%	Mon 10/8/12	Mon 10/8/12
46	USEPA Review of AGWS Report	0%	Tue 10/9/12	Wed 11/7/12



Project: 139452 - CRS Superfund Site
Date: Wed 5/16/12

Task | Progress | Summary | External Tasks | Deadline
Split | Milestone | Project Summary | External Milestone

EAST BRANCH BLACK RIVER

FLOW

EAST BANK HIGH WATER MARK

TYPICAL EDGE OF WATER

STAFF GUAGE
BM=687.00'

SW/SD-1 SW/SD-2 SW/SD-3 SW/SD-4 SW/SD-5

TOP OF BANK

HA-1 HA-2 HA-3 HA-4 HA-5 HA-6 HA-7

SS-1 SS-2 SS-3 SS-4 SS-5

CLAY PIPE SW SAMPLE

MW-1 MW-4 MW-5 MW-6 MW-6R (BORING 6R) MW-7A MW-7D MW-8D MW-9D MW-10A (BORING B) MW-10B (BORING F) MW-11A (BORING E) MW-11B (BORING D) MW-12 (BORING C) MW-13A (BORING X) MW-14A (BORING A) MW-15A MW-15B MW-16

BORING I BORING J BORING K

GP-1 GP-2 GP-3 GP-4 GP-5 GP-6 GP-7 GP-8 GP-9 GP-10 GP-11 GP-12 GP-13 GP-14 GP-15 GP-16 GP-17 GP-18 GP-20 GP-21 GP-22 GP-23 GP-24 GP-25 GP-26 GP-27 GP-28 GP-29 GP-30 GP-31 GP-32 GP-33 GP-34 GP-35 GP-36 GP-37 GP-38 GP-39 GP-40 GP-41 GP-42 GP-43 GP-44 GP-45 GP-46 GP-48 GP-49 GP-50 GP-52 GP-53 GP-54 GP-55 GP-56 GP-57 GP-58 GP-59 GP-60 GP-61 GP-62 GP-63 GP-64 GP-66 GP-67 GP-68 GP-69 GP-70 GP-71 GP-72 GP-73 GP-74 GP-75 GP-76 GP-77 GP-78 GP-79 GP-80 GP-81 GP-82 GP-83 GP-84 GP-85 GP-86 GP-87 GP-88 GP-89 GP-90 GP-91 GP-92 GP-93 GP-94 GP-95 GP-96 GP-97 GP-98 GP-99 GP-100 GP-101 GP-102 GP-103 GP-104 GP-105

FORMER DRUM STORAGE AREA 1

FORMER TANKER STAGING AREA 1

FORMER TANKER STAGING AREA 2

FORMER GAS HOLDER LOCATIONS

FORMER DRUM STORAGE AREA 2

FORMER GAS HOLDER PIT

FORMER BRIGHTEN STILL BUILDING LOCATION

FORMER ABOVE-GROUND STORAGE TANK AREA

FORMER DRUM STORAGE AREA 3

FORMER BUILDING LOCATION

FORMER RODNEY HUNT STILL BLDG. LOCATION

EXPOSED FOUNDATION FLUSH WITH GROUND

ABANDONED RAILROAD TRACK

WOODED

CONCRETE

WAREHOUSE AND OFFICE

M & M ALUMINUM SIDING

RODNEY HUNT STILL BUILDING

ASPHALT

GRAVEL

CONCRETE

6' FENCE

6' CHAIN LINK FENCE

10' CHAIN LINK FENCE

GATE

6' C.L.F. GATE

LOCUST STREET (FORMERLY MAPLE STREET)

JOHN STREET

LEGEND:

- SITE BOUNDARY LINE
- FENCE LINE
- ROAD CENTERLINE
- EXISTING STRUCTURES
- EXISTING SITE FEATURES
- FORMER STRUCTURE LOCATIONS
- FORMER STORAGE AREAS
- STORM SEWER LINE
- SANITARY SEWER LINE
- COMBINED SEWER LINE
- WATER LINE
- GAS LINE
- OVERHEAD ELECTRIC LINE
- OVERHEAD TELEPHONE LINE
- CATCH BASIN
- STORM SEWER MANHOLE
- FORMER SUMP
- ABANDONED MONITORING WELL
- MONITORING WELL
- SEDIMENT/SURFACE WATER SAMPLE
- RI SOIL BORING
- SOIL BORING WITH GROUNDWATER SAMPLE
- 2010/2011 SOIL BORING
- SHELBY TUBE BORING
- HAND AUGER BORING
- SOIL SAMPLE
- PROPOSED BORING FOR SUPPLEMENTAL AGWS
- PROPOSED SURFACE WATER SAMPLING LOCATION

Brown AND Caldwell
COLUMBUS, OHIO

SUBMITTED: _____ DATE: _____ PROJECT MANAGER

APPROVED: _____ DATE: _____ BROWN AND CALDWELL

LINE IS 2 INCHES AT FULL SIZE (IF NOT 2" - SCALE ACCORDINGLY)

DESIGNED:
DRAWN: ADH
CHECKED:
CHECKED:
APPROVED: JP

EXTERNAL REFERENCE FILES

REVISIONS					
ZONE	REV.	DESCRIPTION	BY	DATE	APP.

0 20 40
SCALE IN FEET

CHEMICAL RECOVERY SYSTEMS SITE
ELYRIA, OHIO

PROPOSED SUPPLEMENTAL BORING LOCATIONS

FILENAME: Figure 3,1 BKK 01-23-12.DWG
BC PROJECT NUMBER: XXXXXX
CLIENT PROJECT NUMBER:
FIGURE NUMBER: 2-1

C:\CRS\DRAWINGS\CAD\FIGURES\8-09-2011 FIGURES PDF\FIGURE 3,1 BKK 01-23-12.DWG February 1, 2012

February 1, 2012

Tables

Table 1-1 Groundwater Sampling Results

Table 1-2 NAPL Sampling Results

TABLE 1-1. Groundwater Sampling Results Summary, CRS Site - Elyria, Ohio

Parameter	Units	MW-1	MW-5	MW-5	MW-6	MW-6	MW-6RA	MW-6RB	MW-6RC	MW-7A	MW-7D	MW-8D	MW-9D	MW-10A	MW-10B	MW-11A	MW-11B	MW-12	MW-13A	MW-14A	MW-15A	MW-15B	MW-16	MW-16
		8/18/2011	9/23/2010	8/17/2011	11/19/2010	8/19/2011	7/28/2011	8/18/2011	8/19/2011	8/19/2011	8/18/2011	8/18/2011	8/17/2011	8/18/2011	8/17/2011	8/18/2011	8/17/2011	7/25/2011	8/19/2011	8/23/2011	8/19/2011	8/17/2011	9/23/2010	8/19/2011
Volatile Organic Compound																								
Acetone	ug/L	**1.1** J	< 1.9 JB	< 10	NA	< 4800 JB	**41000**	< 6500 JB	< 3300	< 33000	< 10	< 20	< 10	**1800** J	**1.3** J	< 630	< 100	< 3300	< 67	**2400** J	< 63	< 100	< 1.4 JB	< 10
Benzene	ug/L	**0.28** J	< 1	< 1	NA	< 2500	**5800**	**4500**	**160** J	**49000**	**4.4**	**12**	**0.5** J	**7100**	**17**	**620**	**13**	**840**	< 6.7	**6300**	**9.3**	< 10	< 1	< 1
2-Butanone	ug/L	< 10	< 10	< 10	NA	< 25000	**34000**	< 25000	< 3300	< 33000	< 10	< 20	< 10	< 8300	< 10	< 630	< 100	< 330	< 67	< 20000	< 63	< 100	< 10	< 10
Carbon tetrachloride	ug/L	< 1	< 1	< 1	NA	< 2500	< 560	< 2500	< 330	< 3300	< 1	< 2	< 1	< 830	< 1	< 63	< 10	< 330	< 6.7	< 2000	< 6.3	< 10	< 1	< 1
Chloroethane	ug/L	< 1	< 1	< 1	NA	< 2500	< 560	< 2500	< 330	< 3300	< 1	< 2	< 13	< 830	< 1	**99**	< 10	< 330	< 6.7	< 2000	< 6.3	< 10	< 1	< 1
Chloroform	ug/L	**0.22** J	**0.36** J	**0.35** J	NA	< 2500	< 560	< 2500	< 330	< 3300	< 1	< 2	< 1	< 830	< 1	< 63	< 10	< 330	< 6.7	< 2000	< 6.3	< 10	< 1	< 1
Cyclohexane	ug/L	< 1	< 1	**0.6** J	NA	< 2500	< 560	< 2500	< 330	< 3300	< 1	< 2	< 1	< 830	**0.18** J	< 63	< 10	**56** J	< 6.7	**270** J	< 6.3	< 10	< 1	< 1
1,2-Dichlorobenzene	ug/L	**0.13** J	< 1	< 1	NA	< 2500	< 560	< 2500	< 330	< 3300	< 1	< 2	< 1	< 830	< 1	< 63	< 10	< 330	< 6.7	< 2000	< 6.3	< 10	< 1	< 1
1,1-Dichloroethane	ug/L	**32**	**4.4**	**6.7**	NA	**6200**	**460** J	< 2500	**800**	**1300** J	**0.69** J	**3.2**	< 1	< 830	**23**	**360**	**11**	< 330	< 6.7	< 2000	< 6.3	< 10	**0.29** J	**0.6** J
1,2-Dichloroethane	ug/L	< 1	< 1	< 1	NA	< 2500	< 560	< 2500	< 330	< 3300	< 1	< 2	< 1	< 830	< 1	< 63	< 10	< 330	< 6.7	< 2000	< 6.3	< 10	< 1	< 1
cis-1,2-Dichloroethene	ug/L	**30**	**2.2**	**15**	NA	**81000**	**11000**	**49000**	**9100**	**70000**	**11**	**50**	**2**	**21000**	**16**	**960**	**140**	**8200**	**3.2** J	**50000**	**22**	< 10	**0.42** J	**1.8**
trans-1,2-Dichloroethene	ug/L	**5.5**	**0.24** J	**0.91** J	NA	< 2500	< 560	< 2500	< 330	< 3300	< 1	**0.43** J	< 1	< 830	< 1	< 63	< 10	< 330	< 6.7	< 2000	< 6.3	< 10	< 1	< 1
1,1-Dichloroethene	ug/L	< 1	< 1	**0.63** J	NA	**1700** J	< 560	**1300** J	**690**	**770** J	< 1	< 2	< 1	**170** J	**0.2** J	**16** J	**2.4** J	< 330	< 6.7	< 2000	< 6.3	< 10	< 1	< 1
trans-1,3-Dichloropropene	ug/L	< 1	< 1	< 1	NA	< 2500	< 560	< 2500	< 330	< 3300	< 1	< 2	< 1	< 830	< 1	< 63	< 10	< 330	< 6.7	< 2000	< 6.3	< 10	< 1	< 1
Ethylbenzene	ug/L	< 1	< 1	< 1	NA	**990** J	**1100**	**3100**	**570**	**1500** J	**0.18** J	**1.1** J	< 1	**1800**	**0.85** J	**86**	< 10	**460**	**2.6** J	**1100** J	**12**	**8.2** J	< 1	< 1
2-Hexanone	ug/L	< 10	< 10	< 10	NA	< 25000	**700** J	< 25000	< 3300	< 33000	< 10	< 20	< 10	< 8300	< 10	< 630	< 100	< 3300	< 67	< 20000	< 63	< 100	< 10	< 10
Isopropylbenzene	ug/L	**0.23** J	< 1	< 1	NA	< 2500	< 560	< 2500	< 330	< 3300	< 1	< 2	< 1	< 830	< 1	< 63	< 10	< 330	< 6.7	< 2000	**1.1** J	< 10	< 1	**0.54** J
4-Methyl-2-pentanone	ug/L	< 10	< 10	< 10	NA	**2100** J	**4800** J	< 25000	< 3300	**9000** J	< 10	< 20	< 10	**1400** J	**0.72** J	< 630	< 100	< 3300	< 67	< 20000	< 63	< 100	< 10	< 10
Methylene chloride	ug/L	< 1	< 1	< 1	NA	**9000**	< 560	< 2500	< 330	< 3300	< 1	< 2	< 1	< 830	< 1	< 63	< 10	**120** J	< 6.7	< 2100	< 6.3	< 10	< 1	< 1
Methylcyclohexane	ug/L	< 1	< 1	< 1	NA	< 2500	< 560	< 2500	< 330	< 3300	**0.14** J	< 2	< 1	< 830	< 1	< 63	< 10	< 330	< 6.7	< 2000	< 6.3	< 10	< 1	< 1
Naphthalene	ug/L	< 0.87 JB	NA	< 1	NA	**8600**	**4400**	< 6000 JB	**5200**	**5300**	< 1	< 2	< 1	< 1700 JB	< 1.1 JB	< 25 J	< 10	NA	**160**	**8800**	**230**	**270**	NA	**4**
Styrene	ug/L	< 1	< 1	< 1	NA	**380** J	< 560	**1400** J	**440**	**1700** J	< 1	< 2	< 1	< 830	< 1	< 63	< 10	< 330	**5.0** J	**3300**	**38**	**32**	< 1	< 1
Tetrachloroethene	ug/L	**4.3**	**4.4**	**5.3**	NA	< 2500	< 560	< 2500	< 330	< 3300	< 1	< 2	< 1	< 830	**0.72** J	< 63	**23**	< 330	**13**	< 2000	**31**	< 10	**6.6**	**26**
Toluene	ug/L	< 1	< 1	< 1	NA	**16000**	**19000**	**75000**	**9800**	**92000**	< 1	< 2	< 1	**12000**	**8.4**	**120**	**2.6** J	**680**	**24**	**40000**	**120**	**71**	< 1	< 1
1,1,1-Trichloroethane	ug/L	**9.3**	**0.41** J	**0.96** J	NA	**23000**	**410** J	**19000**	**5800**	**7300**	< 1	< 2	< 1	**910**	< 1	< 63	< 10	**220** J	**7.5**	**2600**	**3.5** J	< 10	**0.40** J	**1.2**
Trichloroethene	ug/L	**13**	**3.3**	**17**	NA	**9300**	**100** J	< 2500	< 330	**45000**	**0.51** J	**1.4** J	< 1	< 830	**0.48** J	**19** J	**13**	< 330	**13**	**16000**	**7.2**	< 10	**1.9**	**6.1**
1,1,2-Trichloro-1,2,2-trifluoroethane	ug/L	< 1	**2.6**	**4.6**	NA	< 2500	< 560	< 2500	< 330	< 3300	< 1	< 2	< 1	< 830	< 1	< 63	< 10	< 330	< 6.7	< 2000	< 6.3	< 10	< 1	< 1
1,2,4-Trimethylbenzene	ug/L	NA	NA	NA	NA	NA	**170** J	NA	**240** J	NA	NA	NA	NA	NA	NA	NA	NA	**62** J	NA	NA	**8.3**	**8.9** J	NA	**0.41** J
1,3,5-Trimethylbenzene	ug/L	NA	NA	NA	NA	NA	**150** J	NA	**86** J	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	**3.2** J	**3** J	NA	NA
Vinyl chloride	ug/L	**2.6**	**0.45** J	**3.8**	NA	< 2500	< 560	< 2500	**200** J	< 3300	**1.1**	**6.4**	< 1	< 830	**3**	**480**	**24**	< 330	< 6.7	< 2000	< 6.3	< 10	< 1	< 1
Xylenes (total)	ug/L	< 2	< 2	< 2	NA	**3400** J	**4700**	**6100**	**2300**	**3800** J	< 2	< 4	< 2	**4500**	**2.1**	**180**	< 20	**1300**	**17**	**4800**	**68**	**67**	< 2	**2**
Semi-Volatile Organic Compounds																								
Acenaphthene	ug/L	< 0.19	NA	< 0.19	< 40	< 38	NA	< 38	< 38	< 24	< 0.19	< 0.19	< 0.19	**35**	< 0.19	**0.32**	< 0.19	**1.6**	**0.91**	< 76	**2**	**0.68**	NA	**1.4**
Acenaphthylene	ug/L	< 0.19	NA	< 0.19	**120**	**82**	NA	**210**	< 38	**150**	< 0.19	< 0.19	< 0.19	**9.5**	< 0.19	< 0.19	**0.84**	< 0.42	**2.6**	**370**	**16**	**4.4**	NA	**2**
Acetophenone	ug/L	< 0.95	NA	< 0.96	< 200	< 190	NA	< 190	< 190	< 120	< 0.95	< 0.95	< 0.95	< 48	< 0.95	< 0.95	< 0.95	**10**	< 0.95	< 380	< 0.95	< 0.95	NA	< 0.95
Anthracene	ug/L	< 0.19	NA	< 0.19	< 40	< 38	NA	< 38	< 38	**67**	< 0.19	< 0.19	< 0.19	< 9.5	< 0.19	< 0.19	< 0.19	< 0.42	**3.9**	< 76	**2.8**	**0.2**	NA	**0.33**
Benzo(a)anthracene	ug/L	< 0.19	NA	< 0.19	< 40	< 38	NA	< 38	< 38	**44**	< 0.19	< 0.19	< 0.19	< 9.5	< 0.19	< 0.19	< 0.19	< 0.42	**1.7**	< 76	< 0.19	< 0.19	NA	< 0.19
Benzo(a)pyrene	ug/L	< 0.19	NA	< 0.19	< 40	< 38	NA	< 38	< 38	**44**	< 0.19	< 0.19	< 0.19	< 9.5	< 0.19	< 0.19	< 0.19	< 0.42	**1.2**	< 76	< 0.19	< 0.19	NA	< 0.19
Benzo(b)fluoranthene	ug/L	< 0.19	NA	< 0.19	< 40	< 38	NA	< 38	< 38	**28**	< 0.19	< 0.19	< 0.19	< 9.5	< 0.19	< 0.19	< 0.19	< 0.42	**1.0**	< 76	< 0.19	< 0.19	NA	< 0.19
Benzo(ghi)perylene	ug/L	< 0.19	NA	< 0.19	< 40	< 38	NA	< 38	< 38	**25**	< 0.19	< 0.19	< 0.19	< 9.5	< 0.19	< 0.19	< 0.19	< 0.42	**0.74**	< 76	< 0.19	< 0.19	NA	< 0.19
Benzo(k)fluoranthene	ug/L	< 0.19	NA	< 0.19	< 40	< 38	NA	< 38	< 38	< 24	< 0.19	< 0.19	< 0.19	< 9.5	< 0.19	< 0.19	< 0.19	< 0.42	**0.39**	< 76	< 0.19	< 0.19	NA	< 0.19
1,1'-Biphenyl	ug/L	< 0.95	NA	< 0.96	< 200	< 190	NA	< 190	< 190	< 120	< 0.95	< 0.95	< 0.95	< 48	< 0.95	< 0.95	< 0.95	**1.7** J	< 0.95	< 380	**6.2**	**1.6**	NA	**4**
Bis(2-ethylhexyl)phthalate	ug/L	< 1.9	NA	**1.2** J	< 400	< 380	NA	< 380	< 380	< 240	**1.1** J	< 1.9	< 1.9	< 95	< 1.9	< 1.9	**0.88** J	< 4.2	< 1.9	< 760	< 1.9	**1.1** J	NA	< 1.9
Carbazole	ug/L	< 0.95	NA	< 0.96	< 200	< 190	NA	< 190	< 190	< 120	< 0.95	< 0.95	< 0.95	< 48	< 0.95	< 0.95	< 0.95	< 2.1	< 0.95	< 380	**0.31** J	< 0.95	NA	< 0.95
Chrysene	ug/L	< 0.19	NA	< 0.19	< 40	< 38	NA	< 38	< 38	**36**	< 0.19	< 0.19	< 0.19	< 9.5	< 0.19	< 0.19	< 0.19	< 0.42	**1.4**	< 76	< 0.19	< 0.19	NA	< 0.19
Dibenz(a,h)anthracene	ug/L	< 0.19	NA	< 0.19	< 40	< 38	NA	< 38	< 38	< 24	< 0.19	< 0.19	< 0.19	< 9.5	< 0.19	< 0.19	< 0.19	< 0.42	**0.12** J	< 76	< 0.19	< 0.19	NA	< 0.19
Dibenzofuran	ug/L	< 0.95	NA	< 0.96	< 200	< 190	NA	< 190	< 190	< 120	< 0.95	< 0.95	< 0.95	< 48	< 0.95	< 0.95	< 0.95	< 2.1	< 0.95	< 380	**0.31** J	< 0.95	NA	< 0.95
2,4-Dimethylphenol	ug/L	< 1.9	NA	< 1.9	< 400	< 380	NA	< 380	< 380	< 240	< 1.9	< 1.9	< 1.9	< 95	< 1.9	< 1.9	< 1.9	**2.7** J	< 1.9	< 760	< 1.9	< 1.9	NA	< 1.9
Fluoranthene	ug/L	< 0.19	NA	< 0.19	< 40	< 38	NA	< 38	< 38	**110**	< 0.19	< 0.19	< 0.19	< 9.50	< 0.19	< 0.19	< 0.19	**0.60**	**9.7**	< 76	**5**	**0.21**	NA	**0.86**
Fluorene	ug/L	< 0.19	NA	< 0.19	< 40	< 38	NA	**51**	< 38	**81**	< 0.19	< 0.19	< 0.19	< 0.95	< 0.19	< 0.19	< 0.19	**1.3**	**4.9**	< 76	**2.7**	**0.67**	NA	< 0.19
Indeno(1,2,3-cd)pyrene	ug/L	< 0.19	NA	< 0.19	< 40	< 38	NA	< 38	< 38	**17** J	< 0.19	< 0.19	< 0.19	< 0.95	< 0.19	< 0.19	< 0.19	< 0.42	**0.46**	< 76	< 0.19	< 0.19	NA	< 0.19
Isophorone	ug/L	< 0.95	NA	< 0.96	< 200	**650**	NA	< 190	< 190	**35** J	< 0.95	< 0.95	< 0.95	< 48	< 0.95	< 0.95	< 0.95	< 2.1	< 0.95	< 380	< 0.95	< 0.95	NA	< 0.95
2-Methylnaphthalene	ug/L	< 0.19	NA	< 0.19	**370**	**360**	NA	**610**	**200**	**390**	< 0.19	< 0.19	< 0.19	**130**	< 0.19	**0.82**	< 0.19	**4.7**	**0.58**	**330**	**6.4**	**6.9**	NA	**0.91**
2-Methylphenol	ug/L	< 0.95	NA	< 0.96	< 200	< 190	NA	< 190	< 190	< 120	< 0.95	< 0.95	< 0.95	< 48	< 0.95	< 0.95	< 0.95	**2.7**	< 0.95	< 380	< 0.95	< 0.95	NA	< 0.95
3 & 4-Methylphenol	ug/L	< 1.9	NA	< 1.9	NA	< 380	NA	< 380	< 380	< 240	< 1.9	< 1.9	< 1.9	< 95	< 1.9	< 1.9	< 1.9	**5.1**	< 1.9	< 760	< 1.9	< 1.9	NA	< 1.9
Naphthalene	ug/L	< 0.19	NA	< 0.19	**6000**	**6300**	NA	**7200**	**4600**	**5100**	< 0.19	< 0.19	< 0.19	**1800**	**0.88**	**11**	**0.29**	**52**	**0.25**	**9500**	**150**	**210**	NA	**2.8**
Phenanthrene	ug/L	< 0.19	NA	< 0.19	**55**	< 38	NA	< 38	< 38	**290**	< 0.19	< 0.19	< 0.19	**26**	< 0.19	< 0.19	< 0.19	**2.4**	**4.1**	< 76	**15**	**1.8**	NA	**0.38**
Phenol	ug/L	< 0.95	NA	< 0.96	< 200	< 190	NA	< 190	< 190	< 120	< 0.95	< 0.95	< 0.95	**33** J	< 0.95	**3.5**	< 0.95	**11**	< 0.95	< 380	< 0.95	< 0.95	NA	< 0.95
Pyrene	ug/L	< 0.19	NA	< 0.19	< 40	< 38	NA	< 38	< 38	**150**	< 0.19	< 0.19	< 0.19	< 9.5	< 0.19	< 0.19	< 0.19	**0.65**	**12**	< 76	**6.2**	**0.22**	NA	**0.66**

NOTES:

J-Estimated result. Result is less than the reporting limit.

B-Method blank contamination. The associated method blank contains the target analyte at a reportable level.

TABLE 1-2. NAPL Sampling Summary Table - CRS Site, Elyria, Ohio

Parameter	Units	MW-6 DNAPL 9/29/2010	MW-6 DNAPL 11/16/2010	MW-6 LNAPL 11/16/2010	MW-7A LNAPL 8/19/2011	MW-7A LNAPL 9/21/2011	MW-7A DNAPL 9/21/2011	MW-13 DNAPL 7/27/2011	MW-13 LNAPL 7/27/2011	MW-14A DNAPL 8/19/2011	MW-14A LNAPL 8/19/2011
Volatile Organic Compounds											
Acetone	mg/kg	< 2100	NA	< 2100	< 4	1.5 J	< 400	< 380	< 4	< 4	< 22000
Benzene	mg/kg	320 J	NA	< 520	24	63	710	< 94	< 1	5.9	2300 J
Cyclohexane	mg/kg	< 2100	NA	< 2100	< 2	< 4	< 200	40 J	< 2	0.310 J	< 11000
1,1-Dichloroethane	mg/kg	< 530	NA	< 520	0.730 J	0.990 J	< 100	< 94	< 1	0.490 J	< 5400
1,1-Dichloroethene	mg/kg	270 J	NA	< 520	0.570 J	0.760 J	< 100	< 94	< 1	0.420 J	< 5400
cis-1,2-Dichloroethene	mg/kg	4400	NA	< 260	37	47	210	< 94	< 1	27	6700
Ethylbenzene	mg/kg	2500	NA	< 520	0.930 J	2	300	550	0.33 J	1.5	6000
2-Hexanone	mg/kg	< 2100	NA	< 2100	0.630 J	1.6 J	< 400	< 380	< 4	< 4	< 22000
Isopropylbenzene	mg/kg	86 J	NA	< 1000	< 1	< 2	< 100	72 J	< 1	< 1	< 5400
4-Methyl-2-pentanone	mg/kg	< 2100	NA	< 2100	5.4	14	17 J	< 380	< 4	1 J	< 22000
Methylcyclohexane	mg/kg	360 J	NA	< 520	< 1	< 2	< 21 J	670	< 1	< 1	< 5400
Methylene chloride	mg/kg	< 530	NA	< 520	< 1	< 2	< 100	180	< 2 JB	< 1	< 5400
Naphthalene	mg/kg	92000 J	NA	28000	35	16	4700	NA	NA	47	230000
Styrene	mg/kg	960	NA	590	1.5	3.2	320	1900	1.5	5.3	18000
Tetrachloroethene	mg/kg	140 J	NA	< 520	0.400 J	< 2	29 J	48 J	< 1	0.420 J	< 5400
Toluene	mg/kg	32000	NA	440 J	39	62	2600	820	1.1	34	53000
1,1,1-Trichloroethane	mg/kg	4200	NA	< 520	3	2.9	85 J	40 J	< 1	2.1	2200 J
Trichloroethene	mg/kg	< 530	NA	< 520	18	13	440	61 J	< 1	14	17000
1,2,4-Trimethylbenzene	mg/kg	3400	NA	1000	NA	0.58 J	170	NA	NA	NA	NA
1,3,5-Trimethylbenzene	mg/kg	1200	NA	820 J	NA	NA	62 J	NA	NA	NA	NA
Vinyl Chloride	mg/kg	< 1000	NA	< 1000	0.120 J	< 4	< 200	< 190	< 2	< 2	< 11000
Xylenes (total)	mg/kg	7200	NA	430 J	2.7	5.8	780	4700	3.1	7.3	28000
Semi-Volatile Organic Compounds											
Acenaphthene	mg/kg	NA	52	NA	< 19	< 20	< 500	1300 J	28 J	< 20	1100 J
Acenaphthylene	mg/kg	NA	380	NA	< 19	1.5 J	270 J	12000	220	2.1 J	12000
Anthracene	mg/kg	NA	310	NA	< 19	1.3 J	260 J	6700 J	120 J	< 20	5400 J
Benzo(a)anthracene	mg/kg	NA	< 25	NA	< 19	< 20	150 J	3500 J	74 J	< 20	3500 J
Benzo(a)pyrene	mg/kg	NA	260	NA	< 19	< 20	160 J	4100 J	78 J	< 20	3200 J
Benzo(b)fluoranthene	mg/kg	NA	200	NA	< 19	< 20	120 J	2100 J	49 J	< 20	2200 J
Benzo(ghi)perylene	mg/kg	NA	150	NA	< 19	< 20	100 J	2500 J	51 J	< 20	1700 J
Benzo(k)fluoranthene	mg/kg	NA	110	NA	< 19	< 20	35 J	1700 J	29 J	< 20	1300 J
1,1'-Biphenyl	mg/kg	NA	130 J	NA	< 19	< 20	140 J	4200 J	79 J	< 20	2700 J
Chrysene	mg/kg	NA	< 25	NA	< 19	< 20	140 J	3900 J	72 J	< 20	3000 J
Fluoranthene	mg/kg	NA	600	NA	< 19	2 J	420 J	11000	200 J	1.5 J	8900 J
Fluorene	mg/kg	NA	280	NA	< 19	< 20	210 J	6200 J	120 J	< 20	5100 J
Indeno(1,2,3-cd)pyrene	mg/kg	NA	91	NA	< 19	< 20	64 J	1500 J	31 J	< 20	1200 J
2-Methylnaphthalene	mg/kg	NA	790	NA	< 19	3.2 J	650	22000	400	3 J	18000
Naphthalene	mg/kg	NA	3400	NA	14 J	26	3100	89000	1500	24	110000
Phenanthrene	mg/kg	NA	1200	NA	< 19	4.3 J	830	23000	450	3.6 J	20000
Pyrene	mg/kg	NA	850	NA	< 19	2.9 J	570	15000	300	2.1 J	12000

NOTES:

J-Estimated result.

B-Method blank contamination. The associated method blank contains the target analyte at a reportable level.

NA - Not analyzed

ATTACHMENT A

FIELD SAMPLING PLAN
FOR THE
REMEDIAL DESIGN / REMEDIAL ACTION
CHEMICAL RECOVERY SYSTEMS, INC. SITE

Prepared for

CRS Site RD/RA Performing Parties
Chemical Recovery Systems, Inc. (CRS) Site
Elyria, Ohio

September 2010

Revised May 2012

Prepared by:

Brown and Caldwell
4700 Lakehurst Court, Suite 100
Dublin, Ohio 43016

139452

TABLE OF CONTENTS

1. INTRODUCTION A-1
1.1 Background and History A-1
2. SAMPLING AND TESTING OBJECTIVES A-3
3. SAMPLING LOCATION AND FREQUENCY A-4
3.1 Investigation/Site Closure Activities A-4
3.1.1 Soil Boring/Monitoring Well Installation A-4
3.1.2 Groundwater Sampling A-7
3.1.3 Surface Water Sampling A-7
3.1.4 NAPL Sampling A-7
3.1.5 Monitoring Well Abandonment A-7
3.1.6 Soil Sampling A-7
3.1.7 Demolition A-8
3.1.8 Closure of On-Site Sumps A-8
3.2 Contingency Investigations A-8
4. SAMPLE DESIGNATION AND DOCUMENTATION A-9
4.1 Field Sample Designation A-9
4.2 Laboratory Sample Identification Number A-10
4.3 Field Sampling Documentation A-10
4.3.1 Sample Labels A-10
4.3.2 Field Logbook A-10
4.3.3 Field Forms A-11
5. SAMPLING EQUIPMENT AND PROCEDURES A-12
5.1 Sample Collection Equipment A-12
5.1.1 Equipment for Sampling Soils A-12
5.1.2 Equipment for Sampling Groundwater A-13
5.1.3 Equipment for Sampling Surface Water A-13
5.1.4 Equipment for Sampling NAPL A-14
5.2 Field Instrumentation A-14
5.2.1 Interface Probe A-14
5.2.2 Groundwater Multi-Parameter Meter A-14
5.2.3 Photoionization Detector A-15
5.2.4 Multi-Gas Detector A-15
5.3 Sample Collection Procedures A-15
5.3.1 Soil Sampling A-15
5.3.2 Groundwater Sampling A-16
5.3.3 Surface Water Sampling A-16
5.3.4 NAPL Sampling A-17

5.4 Field Quality Control Samples A-17
5.4.1 Trip Blank A-17
5.4.2 Equipment Blank A-17
5.4.3 Field Duplicates A-17
5.4.4 Matrix Spike/Matrix Spike Duplicates A-18
5.5 Environmental Conditions A-18
5.6 Decontamination Procedures A-18
5.7 Investigation Derived Waste A-18

6. SAMPLE HANDLING AND CUSTODY A-20
6.1 General Sampling Protocols A-20
6.1.1 Sample Containers A-20
6.2 Chain-of-Custody A-20
6.2.1 Sample Labels A-20
6.2.2 Chain-of-Custody Record A-20
6.2.3 Transfer of Custody A-21
6.3 Sample Packaging and Shipment A-21

7. REFERENCES A-22

FIGURES ..... Follows Text

Figure 1-1 Site Location

Figure 1-2 Site Layout

TABLES ..... Follows Text

Table 6-1 Analytical Sample Volume and Containers

ATTACHMENT A - SOPS AND ASTM/API STANDARDS ..... A

BCSOP No. 1 Monitoring Well Installation and Development

BCSOP No. 2 Monitoring Well Sampling

ASTM D1586 Standard Method for Standard Penetration Test (Spt) and Split Barrel Sampling of Soils

ASTM D1587 Standard Practice for Thin Walled Tube Sampling of Soils for Geotechnical Purposes

ASTM D4630-96 Standard Test Method for Determining Transmissivity and Storage Coefficient of Low Permeability Rocks by Insitu Measurements Using the Constant Head Injection Test

ASTM D5876 Standard Guide for Use of Direct Rotary Wireline Casing Advancement Drilling Methods for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices

ASTM D5079 Standard Practices for Preserving and Transporting Rock Core Samples

ASTM D425 Standard Test Method for Centrifuge Moisture Equivalent of Soils

ASTM D445	Standard Test Method for Kinematic Viscosity of Transparent And Opaque Liquids (and Calculation of Dynamic Viscosity)
ASTM D854	Standard Test Methods for Specific Gravity of Soil Solids by Water Pycnometer
ASTM D1481	Standard Test Method for Density and Relative Density (Specific Density) of Viscous Materials By Lipkin Bicapillary Pycnometer
ASTM D6836	Standard Test Methods for the Determination of Soil Water Characteristic Curve for Desorption Using Hanging Column, Pressure Extractor, Chilled Mirror Hygrometer, or Centrifuge
API RP40	Recommended Practices for Core Analysis
PTSSOP	PTS Laboratory Recommended Core Handling/Preservation Field Procedures

ATTACHMENT B - ODNR WELL ABANDONMENT GUIDANCE ..... B

1. INTRODUCTION

This document presents the Field Sampling Plan (FSP) for field activities associated with Remedial Design (RD), and Remedial Action (RA) at the Chemical Recovery Systems, Inc. (CRS) site (the Site). Updates to this FSP may be made periodically if field activities are required that are not currently anticipated. These activities will be undertaken on behalf of the CRS Site RD/RA Group Performing Parties (Performing Parties). This work is being performed to meet the requirement of the Consent Decree 1:10-cv-00996 (CD), signed by USEPA and the Performing Parties, and entered by the Court on July 18, 2010, the Record of Decision (ROD), and the Statement of Work (SOW) for the Site.

This FSP has been prepared in general conformance to relevant U.S. Environmental Protection Agency (USEPA) and the Ohio EPA (OEPA) guidance, including "Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA" (USEPA, 1988).

The overall approach and objectives of the remedial design activities are discussed in the RD Work Plan (RDWP) (Brown and Caldwell, 2010). The Data Quality Objectives (DQOs) and overall project Quality Assurance (QA) objectives and procedures are presented in the Quality Assurance Project Plan (QAPP), included as Appendix B to the RDWP. A site-specific Health and Safety Plan (HASP) developed for the field activities associated with the RD is included as Appendix C to the RDWP.

This FSP describes the project sampling rationale and procedures that will result in data which meets the project objectives. The FSP is organized into the following sections:

- Section 1.0 - Introduction
- Section 2.0 - Sampling and Testing Objectives
- Section 3.0 - Sampling Location and Frequency
- Section 4.0 - Sample Designation and Documentation
- Section 5.0 - Sample Equipment and Procedures
- Section 6.0 - Sample Handling and Custody
- Section 7.0 - References

1.1 Background and History

The Site is located at 142 Locust Street (formerly Maple Street) in Elyria, Lorain County, Ohio (Figure 1-1) and is located in a predominantly industrial area of Elyria, Ohio. The western boundary of the Site runs along the East Branch of the Black River (River). To the north and east, the Site is bordered by the BASF Corporation (formerly Engelhard Corporation and prior to that the Harshaw Chemical Company). Locust Street runs along the eastern boundary of the CRS Site. The M&M Aluminum Siding property to the south was recently acquired by BASF for an expansion project. Figure 1-2 is a Site layout drawing including on-site features and property.

Two buildings (located on the southeast corner of the Site) remain on the property. One building is the former warehouse and office building and the other is a portion of the Rodney Hunt Still Building. These buildings have historically been leased and utilized for storage of carpeting and other miscel-laneous items by M&M Improvement Company. A concrete slab foundation of a former building is located in the northwest

corner of the property. In addition, a concrete slab foundation for the Brighton Still building, and the secondary containment dike for a former AST farm remain in the northwestern corner of the Site (all tanks have been removed from the AST farm). Two sumps remain onsite, one sump is located in the Rodney Hunt Building and the other, is located in the former Brighton Still Building. Four pipes (subsurface conduits) are located along the western boundary of the property along the River. The primary subsurface conduit is a storm sewer pipe that runs from Locust Street under the Site to the River. A manhole on Locust Street provides access to the storm sewer, which drains run-off from BASF and other commercial and industrial sites comprising approximately one fourth of the downtown Elyria area. Cars, trucks, wood waste, and other debris are also located on the property.

The working area of the Site is essentially level and consists of a grassy cover with patches of gravel and asphalt. Surface drainage is westward, towards the East Branch of the Black River (the River). The western edge of the Site is heavily vegetated with a steep grade from the working area to the East Bank of the Black River. The property is fenced on the northern, eastern, and southern sides, restricting casual access to the Site.

Based on Sanborn Maps dating as far back as 1884, the Site has been in industrial use since the later 1800's. In 1960, Russell Obitts began operations by leasing lots from the Swiers Coal Company. On March 23, 1965, Dorothy K. Dubena purchased the parcels from the Swiers Coal Company. Dorothy Dubena married Russell Obitts and became Dorothy Obitts. Two companies were owned by Mr. Obitts and operated at this Site from 1960 through 1974. Obitts Chemical Services operated a solvent reclamation facility. Obitts Chemical Company sold the reclaimed solvents to industry. Obitts obtained used, "scrap" or "spent" organic solvents from various companies. After distilling the "dirty" solvents, the "cleaned" reclaimed solvents were repackaged and sold. The solvents were transported to and from the Site in 55-gallon drums or by tanker trucks.

From 1974 to 1981, CRS continued in the business of solvent reclamation and sales. The solvents continued to be stored in 55-gallon drums, ASTs, and tanker trucks. It is alleged that several spills and releases occurred during this time. In 1980, the USEPA filed a lawsuit against CRS, requiring the facility owners to abate environmental issues identified at the Site. In response to this lawsuit, CRS ceased the receipt, processing, and storage of spent solvents and removed tanks, drums, and other solvent containers from the Site in 1981. CRS ceased operations and filed for bankruptcy prior to 1983.

2. SAMPLING AND TESTING OBJECTIVES

The objectives of the sampling program are to gather data of adequate technical content, quality, and quantity to support the RD/RA for the Site. Samples will be collected at designated locations to ensure that a sufficient quantity of data is obtained to meet the goals of the RD/RA. The overall objective for each of the sampling tasks is to obtain data of sufficient quality and quantity to evaluate the current conditions of the Site and to verify the effectiveness of the remedy.

The QAPP, provided as a companion document, outlines the data quality objectives and the corresponding quality assurance standards by which all sample acquisition and testing will be conducted as part of the field program. The QAPP presents the detailed, site-specific laboratory protocols to be followed to ensure the quality and integrity of the remedial design activities data, along with the accuracy and precision of the testing/analyses and the representativeness and completeness of the data.

3. SAMPLING LOCATION AND FREQUENCY

This section discusses sampling locations and frequencies for the RD activities. Detailed sampling and field procedures are provided in Sections 4 and 5.

3.1 Investigation/Site Closure Activities

The following paragraphs summarize the proposed investigations to support the RD.

3.1.1 Soil Boring/Monitoring Well Installation

Several monitoring wells will be installed as part of the RD/RA. If additional monitoring wells or replacement wells are determined to be necessary, they will be installed consistent with those wells installed during the RI/FS. Wells will be constructed of two-inch diameter schedule 40 PVC well casing and screen. The well screen will include 0.010-slot well screen and a screen length of 5 to 15 feet, depending on the length of the zone to be monitored. The well will be installed such that the shallow water table is contained within the screened interval where a water table exists. Wells will be installed through 4.25-inch I.D. hollow stem augers (HSAs), if the well is placed in unconsolidated material, to create an approximately eight-inch diameter borehole. Well construction will include a sand/filter pack around the screened portion of the well to a height of one to two feet above the top of the screen, followed by a one- to two-foot bentonite seal, then grouted to ground surface. A measurement reference point will be marked on the top of the inner PVC well casing. Surface completion will include a steel, lockable, stick-up protective casing secured in a concrete surface pad.

During drilling in unconsolidated material, the soils will be continuously sampled via the SPT method (ASTM D1586) to establish the lithology of the subsurface and the location of the shallow water table. Following installation, the new monitoring well(s) will be developed. Monitoring well installation and development will be in accordance with those procedures described in SOP No. 1 in Attachment A. Soil cuttings and development water will be managed in accordance with procedures described in Section 5-7.

Wells to be completed in competent rock will utilize one or more rock drilling techniques such as air rotary, or wire-line. The upper, unconsolidated material will be cased off from the bedrock borings using HSA until refusal is encountered. A casing will be placed from the grade to the top of competent bedrock to ensure that contaminants present in the overburden or in perched water above bedrock does not enter the bedrock zone of the well. The casing will be grouted in place at the bedrock surface using a cement-bentonite grout. This grouting will be allowed to set for a minimum of 24-hours, after which point a borehole will be drilled through the hardened grout and to the targeted total depth of core boring in bedrock.

Rock at each coring location will be cored using a wire-line or similar drilling methodology following ASTM D5876. Core runs will be advanced in 10-foot increments using air. If circulation cannot be maintained solely through the use of air, the determination may be made to use potable water as a drilling fluid. If this is the case, the amount of potable water used will be kept to a minimum and the gallons used for each core run will be recorded in the boring log. Any water or air that will be used inside the borehole will be analyzed or filtered, respectively, to prevent addition of contaminated material into the borehole. Care will be exercised while drilling to limit the down pressure on the drill stem to avoid creating drilling induced fractures immediately adjacent to the borehole.

Each rock core will be stacked in stratigraphic order in a core box. Major core breaks, the start and stop of each run, and the time of each run will be recorded on the core box. The storage and transport of rock cores will be in conformance with ASTM D5079 Standard Practice for the Transporting and Storage of Rock Core Samples.

A field geologist will log each rock core noting the rock type, field hardness, weathering structure, color, texture, mineralogy, discontinuity spacing, coatings/infillings, attitude, shape, and any other identified discrete features. The percent recovery (sum total length of recovered core fitted together, divided by the length of attempted core run) and rock quality designation (RQD) (sum length of hard, sound core pieces 4 inches and longer, divided by the length of attempted core run) will be calculated and recorded on the log for each core run along with the starting/ending depths and times, amount of drilling water used during the core run, and results of a PID screen over the length of the core.

Rock core sections collected for petrophysical and non-aqueous phase liquid (NAPL) testing will be handled and tested in accordance with the following ASTM and/or American Petroleum Institute (API) published procedures contained in Attachment A:

- Grain Density (API RP40)
- Total Porosity (API RP40)
- Air-Filled Porosity (API RP40)
- Effective Porosity (Mod. ASTM D425)
- Pore Fluid Saturations (API RP40)
- Hydraulic Conductivity (API RP40/EPA 9100)
- Intrinsic Permeability (API RP40)
- Specific Gravity of Fluids (ASTM D854) or Fluid Density (ASTM D1481)
- Viscosity of Fluids (ASTM D445)
- Residual Saturation by Direct Centrifuge Method (API RP 40)
- Residual Saturation by LNAPL/Water Imbibition Capillary Pressure Package (API RP40 and ASTM 6836).

Subsamples of the core subjected to petrophysical testing will be sent to the chemical testing laboratory, preserved and crushed in the laboratory for chemical extraction and tested in accordance with standard analytical methodologies for volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), Volatile Petroleum Hydrocarbons (VPH) and Extractable Petroleum Hydrocarbons (EPH).

For those coreholes from which interval groundwater samples will be collected, following each 10 foot core run, the boring will be measured for total depth with a clean, weighted tape. A decontaminated, 5-foot, pre-packed screen with packer will be attached to the drill stem and lowered to the total depth of the boring to create a temporary well. The packer will be placed to isolate the water interval around the well screen. Once the packer is in place, the water level inside the well screen and in the annulus outside the drill stem will be measured and recorded. A Grundfos Redi-flo2 or equivalent pump will be lowered into the temporary well screen. Low-flow groundwater sampling (purge rate less than 500 milliliters per minute and ideally at 100 milliliters per minute) will be performed; the overfilling of the sample collection bottles will be prevented by controlling the flow rate of the pumping. The following water-quality parameters will be monitored during purging to verify stabilization prior to sampling: temperature, pH, and specific conductance. Turbidity, oxidation-reduction potential, and dissolved oxygen will be recorded but not considered parameters for stabilization. Water quality parameters will be recorded on standard groundwater-sampling data sheets along

with the time associated with each monitoring interval and the total volume removed. If any water has been added during drilling, a similar amount will be removed prior to sampling. Groundwater samples will be collected for VOC analysis once the water quality parameters have stabilized to the criteria specified in Ohio Environmental Protection Agency's (OEPA's) Technical Guidance Manual for Hydrogeologic Investigations. Each groundwater sample will be labeled with the core boring location followed by the screen interval in parentheses, the date and time of the sample, sample preservative, requested analysis, and sampler's initials. Samples will be packaged and placed on ice in a cooler for delivery to the laboratory under chain-of-custody procedures. All purge water will be containerized and transported to the onsite investigation derived waste (IDW) staging area.

During the drilling of certain bedrock borings/monitoring wells, or subsequent testing of existing open hole bedrock wells, insitu hydraulic conductivity/permeability, or packer, testing of the bedrock may be performed. Packer testing will be performed in general accordance with ASTM D4630, contained in Attachment A, by a qualified drilling subcontractor under the supervision of BC's field geologist. The packer testing procedure is summarized as follows:

- The downhole testing apparatus consists either of a metal pipe with an open end positioned between an inflatable rubber packer and the bottom of the corehole, or a perforated pipe positioned between two packers within the corehole:

- The packer test interval is typically between five and ten feet, although in some cases other intervals may be necessary to provide complete coverage of the total interval to be tested within a given well. For example, if the total length of bedrock to be tested is 12 feet, then the testing might consist of two six-foot long test intervals, or one five-foot interval and one seven-foot interval. The actual interval length in these situations will be chosen based on review of the boring log, well construction details, etc.;

- The packers are inflated through a flexible line with compressed nitrogen gas to seal off the test zone;

- Potable water is pumped at a constant rate/pressure from the surface into the test interval. The pumping rate, pressure and volume are recorded at regular intervals. The water volume which enters the bedrock under the the specified pressure is a function of the hydraulic conductivity;

- The parameters are recorded at one minute intervals for five minutes. If, during the five minute test period less than 0.1 gallons have flowed into the formation, the test will be continued for an additional five minutes to provide better resolution for low hydraulic conductivity intervals;

- The maximum pressures (in psi) to be used during the test will be determined prior to testing by multiplying the depth of the top of the test interval below ground surface by a factor of 0.7. Higher pressures have the potential to "lift" the tested rock, thus providing faulty values; and

- Upon completion of the test, the hydraulic conductivity will be calculated using the following equation:

  $K = Cp(Q/H)$ Where: K=hydraulic conductivity (ft/yr)

  Q=rate of flow developed at equilibrium (gpm)

  Cp=packer coefficient, from literature (1/ft)

  H=total calculated head (ft)

Following installation, the horizontal/vertical position of the boring/well will be surveyed by a licensed Ohio surveyor in accordance with the QA procedures described in the QAPP. The elevation of the ground surface and the measurement reference point on top of the inner PVC well casing will also be surveyed for elevation.

3.1.2 Groundwater Sampling

Groundwater sampling will be conducted in accordance with the QAPP and SOP No. 2 in Attachment A. Groundwater sampling will generally use "low-flow" purging and sampling techniques, depending on the recovery rates of the wells sampled; hand bailers may also be utilized to collect samples as well as other procedures discussed in SOP No. 2. Groundwater samples collected for analysis will be sent to the analytical laboratory to be analyzed for the specified parameters, which might include some combination of VOCs, SVOCs, metals, and polychlorinated biphenyls (PCBs). Field parameters measured during purging/sampling may include pH, temperature, dissolved oxygen (DO), specific conductance and turbidity. Purge water will be managed in accordance with the procedures described in Section 5-7.

3.1.3 Surface Water Sampling

Surface water sampling will be conducted in accordance with procedures described in Section 5.3.3 and the QAPP. Surface water samples collected for analysis will be sent to the analytical laboratory to be analyzed for the specified parameters, which might include some combination of VOCs, SVOCs, metals, and polychlorinated biphenyls (PCBs).

3.1.4 NAPL Sampling

Samples of NAPL recovered from wells at the Site may be analyzed for certain analytical procedures, including density, viscosity, VOCs, SVOCs, and average molecular weight. NAPL sampling will be conducted in accordance with procedures described in Section 5.3.4 and the QAPP.

3.1.5 Monitoring Well Abandonment

Monitoring well abandonment, if performed, will be in conformance with procedures described in the document entitled "State of Ohio Technical Guidance for Sealing Unused Wells", dated 1996 (Attachment B), and in accordance with OAC 3745-9. The Technical Guidance additionally calls for completing a form for each well abandoned and submitting it to the Ohio Department of Natural Resources (ODNR). Copies of the well abandonment form(s) will also be submitted to USEPA.

Well materials removed during well abandonment will be managed in accordance with procedures described in Section 5-7.

3.1.6 Soil Sampling

The extent of soil excavation that will be necessary in the northwest corner of the Site was defined during the RI. Post-excavation sampling will be conducted to document the concentrations of chemicals of concern (COCs) left in the soil, but will not be used to direct further soil removal. The rationale for determining the size of the excavation will be defined in the RD and will be based on existing data contained in the RI/FS Report and the limits defined in the SOW.

The precise post-excavation sampling strategy will be provided in the RD. These samples will be submitted to the laboratory for analysis. Analyses will likely include a combination of metals, VOCs, SVOCs, as well as PCBs. Analytical procedures will consistent with those described in the QAPP. The post-excavation sample data will be shared and discussed with U.S. EPA prior to initiating backfilling of the excavation.

3.1.7 Demolition

Two on-site buildings/structures (the office/warehouse building and the shell of the former Rodney Hunt still building) will be demolished. Metal, glass, and asbestos containing materials will be disposed of off-site. In addition, wood chips and other vegetative debris in the former aboveground storage tank area will be disposed of off-site. Other building materials may be crushed and used on site as fill material. U.S. EPA approved sampling methods will be utilized to characterize the demolition materials generated during demolition activities for off-site disposal or for use on-site, where appropriate, for leveling, filling, and grading.

3.1.8 Closure of On-Site Sumps

Two on-site sumps will be removed or properly sealed in place. The Brighton Still sump will be removed for offsite disposal. At least one soil sample will be collected from beneath the sump to characterize the soil after removal of this sump. The sample will be analyzed for metals, VOCs and SVOCs.

Samples of soil near the Rodney Hunt Still sump will be collected for metals, VOCs, and SVOCs analyses. Soil cleanup levels in the areas of the Rodney Hunt Still will be proposed in a demonstration to be protective of groundwater. If the demonstration is approved, the sump will be backfilled in accordance with the SOW. If the demonstration is not approved, the sump will be excavated and materials will be disposed of off-site. If removal is necessary, at least one soil sample will be collected from beneath the sump to characterize the soil after removal of this sump. The sample will be analyzed for metals, VOCs and SVOCs.

3.2 Contingency Investigations

In addition to those investigations defined above, additional, or contingency investigations may also be necessary during the RD/RA, depending on the results of the initial investigations and evaluations. These contingency investigations may include additional monitoring well installation, groundwater sampling, monitoring well abandonment, and post-excavation sampling. The field procedures associated with these contingency investigations are discussed in general terms in the above subsections, so that if any aspect of these investigations is realized, this FSP will not need significant revisions. Field procedures associated with any additional investigations will either be in accordance with procedures described in this FSP or specific procedures described in the work plan developed for the investigation.

4. SAMPLE DESIGNATION AND DOCUMENTATION

A key aspect of field sampling quality assurance involves the implementation of sampling documentation procedures. Proper development and implementation of these procedures ensures the integrity of a sample from collection to data reporting.

4.1 Field Sample Designation

Each sample collected during the pre-design field activities will receive a unique sample number. This sample number will be essential for tracking samples in the field and at the laboratory, and managing the resulting data.

In order to identify each sample collected, a sample-tracking scheme will be implemented which allows all samples to be traced to a specific location (e.g., well, soil boring, etc.), matrix, depth (as appropriate), and date of collection. These procedures are described in the QAPP and summarized below.

The method of identification of a sample depends on the type of measurement or analysis performed. When field screening measurements (e.g., pH, dissolved oxygen, or turbidity) are made, data are recorded directly in logbooks or on field investigation forms. Identifying information such as project name, station number, station location, date and time, name of sampler, field observations, remarks, or other pertinent information will be recorded.

Samples collected for laboratory analysis during the field investigation will be specifically designated for unique identification. Each sample will be designated by an alpha-numeric code which will identify the sampling location, type or as necessary depth.

Aqueous and solid samples collected as a part of the work will be assigned unique sample identifiers. The sample identifiers are required in order to identify and track each of the samples collected for analysis. Associated field QA/QC samples such as duplicates and field blanks will be further identified adding the date of collection to the sample name as shown below, and as needed a number will be added sequentially to distinguish the samples.

Each of the samples will be identified by a unique alpha-numeric code that indicates the particular sample type, location, and date. The format of the code is as follows:

The three codes that make up the sample identifier are described as follows:

1. The Site name – CRS (Chemical Recovery Systems, Inc.);
2. The medium/sample type codes are listed below:
   - MW - Groundwater sample taken from a monitoring well;
   - SW - Surface water sample;
   - SP - Seep sample;
   - SED - Sediment sample;
   - S - Soil sample
   - NAPL – NAPL sample
   - DW - Drummed waste sample;
   - W - Waste sample;

- SB - soil boring sample;
- TB - Trip Blank;
- FB - Field Blank sample;
- DUP - Duplicate sample; and
- MS/MSD – Matrix Spike/Matrix Spike Duplicate sample.

3. The location code will be keyed to the specific sample designation.

The following is an example of a sample identifier that will be used for samples collected for geotechnical laboratory analysis:

- CRS-SB-10B-2-4: indicating that this is the second soil sample was collected at soil boring location CRS-SB-10 from a depth of 2 to 4 feet below ground surface.

4.2 Laboratory Sample Identification Number

The analytical laboratories will assign a unique I.D. number to each sample submitted for testing. The laboratory will submit a deliverable to BC that provides a cross-reference between the laboratory sample I.D. and the field sample I.D.

4.3 Field Sampling Documentation

A critical aspect of data quality involves the development, use, and maintenance of a field sampling documentation system. A strict and thorough sampling documentation system will be utilized during sampling conducted at the Site. This documentation, which includes proper labeling of sample containers and field logbook entries, is essential for data tracking, sample custody, and overall data quality assurance.

4.3.1 Sample Labels

Sample labels are required on sample containers for the primary purpose of sample identification. Specific field data need not be recorded on the labels as they will be recorded on field data sheets and/or in the field logbook. The sample labels will contain the following information:

- Sample identification (see Section 4.1);
- Depth of sample (if appropriate);
- Analysis/test to be performed;
- An "up" arrow (Shelby tube samples only)
- Preservative (if required);
- Project name and number;
- Date and time of sample collection; and
- Initials of Sampler

4.3.2 Field Logbook

A bound weatherproof logbook, dedicated to the Site, will be kept by the sampling personnel. The Site logbook is a controlled document that records major on-site activities during the field investigation. At a minimum, the Site logbook will include a summary of sampling identifiers and shipment information, visitor's names and arrival/departure times, community contacts, and other site-specific information determined by the sampling personnel to be noteworthy. In addition, prior to field work each day, the Site personnel, the

proposed activities, and the weather will be recorded in the Site logbook. Discussions of program activities, field difficulties/problems, and deviations from the FSP, QAPP and/or other Site plans (with justification) must also be included in the logbook, along with corresponding times.

The sampling team or any individual performing a particular field investigation activity (e.g., soil or groundwater sampling) will be required to maintain a field logbook. Each logbook will be controlled and assigned a unique sequential identification by the sampling personnel.

Pertinent information to be recorded in field logbooks includes information that is necessary to reconstruct the investigative/sampling operations. Documentation of sample activities in the field logbook will be completed immediately after sampling at the location of sample collection. Logbook entries will contain sample information, including sample number, collection time, location, descriptions, field measurements, and other site- or sample-specific observations. Difficulties with sample recovery and other field observations (e.g., staining, visible contamination, etc.) will be noted if encountered.

If photographs are taken as part of the documentation procedure, the name of the photographer, the date, the time, and a description of the photo (including direction and scale, if applicable) will be entered sequentially in the field logbook as the photographs are taken. Once downloaded, the photographs will be numbered in correspondence to the logbook numbers, and the above information will be placed in a photographic log filed with the photographs and/or placed on the back of the photograph.

The field logbooks will have the name of the Site and project written on the cover and inside the cover. The date, page number and initials of the primary field note transcriber will be written atop each page during the day the entries are made. Corrections to the logbooks will consist of a single strike line through the incorrect entry, the new accurate information, the initials of the corrector, and the date of amendment. Any blank spaces/pages in the logbooks will be crossed out with a single strike mark and signed by the person making the notation.

4.3.3 Field Forms

During certain sampling events, specific field forms may be used to record field data. An example is for groundwater sampling where well-specific field forms may be used to record the purging and sampling data. However, basic sampling information will still be recorded in the field logbook.

5. SAMPLING EQUIPMENT AND PROCEDURES

5.1 Sample Collection Equipment

To ensure that data generated for the remedial design are valid, consistent, and usable, BC will implement specific sample collection requirements. These requirements focus on the use of appropriate sample collection equipment and set forth specific procedures for sample collection. Also discussed are the proposed decontamination procedures and management of IDW.

5.1.1 Equipment for Sampling Soils

Various pieces of field equipment that may be used to sample soils at the Site are described below.

- Scoops and Shovels

  Scoops and shovels are used to collect surface soils or sediments. Scoops and shovels are made of a variety of materials including stainless steel, wood, plastic, or Teflon®. Scoops or shovels may be used to collect shallow bulk samples for geotechnical testing.

- Hand Auger

  The hand-powered auger consists of a steel drill bit at the end of a bucket, a drill rod and a T-shaped handle. The auger is used to drill a borehole to a desired depth, usually not more than 4 feet below the surface (depending upon the material encountered). The soil sample is brought to the surface when the auger is withdrawn.

- Soil Corer

  The soil corer consists of a small diameter, hollow, thin-walled metal tube. Their length can vary from six inches to two feet. It can be used to obtain an undisturbed soil profile sample from the surface to a maximum depth of approximately eight feet. The soil corer can be driven either manually by a slide hammer, or using a gas or electric powered hammer drill. Soil corer and slide hammer manufacturers include AMS. Hammer drill manufacturers include Bosch.

- Split Spoon

  Split spoon samplers are used to collect soil samples from a wide variety of soil types at greater depths than obtainable with most other soil sampling devices. A split spoon sampler consists of a heavy steel sampling tube that can be split longitudinally into two equal halves. Split spoons and Shelby tubes (below) are most commonly used when drilling borings with a hollow stem auger drill rig. The split spoon is attached to a drill rod and is forced into the ground by hammering in accordance with ASTM Method D1586. After the desired depth is reached, the split spoon is extracted from the ground and opened to obtain a soil profile sample.

- Shelby Tube

  A Shelby tube is used to obtain an undisturbed sample of soil for determining geotechnical characteristics. It is constructed of metal and has an outside diameter of 2 to 5 inches and a length of 24 to 54 inches. The sampler is advanced through the soil without rotation in a smooth motion. When retrieved, the ends of the sampler are capped with wax, and the sample is sent to the geotechnical laboratory for testing. Shelby tube sampling will be performed in accordance with ASTM Method D1587.

- Macro-Core Sampler

  The macro-core sampler is used when drilling soil borings with the Geoprobe®. The sampler consists of a four-foot long, 2-inch diameter stainless steel barrel with a threaded cutting shoe at its base. Soil samples are collected by attaching the sampler to push rods and pushing or hammering the sampler into the subsurface using the hydraulics on the Geoprobe® rig. The soil samples are contained in disposable, clear acetate liners within the drive barrel. The liner is replaced after each sample is obtained.

- Large Core Sampler

  The large core sampler is also used with the Geoprobe® and is useful when caving of the boring occurs. The sampler consists of a two-foot long, 1-3/8-inch-diameter stainless steel barrel with a threaded cutting shoe at its base. It is a piston-type sampler that recovers soil from discrete depths. Soil samples are collected by attaching the sampler to push rods used to advance the sealed sampler to the top of the depth interval of interest, then releasing a stop pin from the drive head within the push rods. Once the stop pin is released, the sampler is advanced an additional 24 inches. As the sampler is advanced, the drive point/piston retracts allowing the soil to enter the sampler. The soil samples are contained in disposable, clear liners within the drive barrel.

5.1.2 Equipment for Sampling Groundwater

Various pieces of field equipment that may be used to sample groundwater at the Site are described below.

- Bailer

  A bailer is a cylindrical device with a check valve that is made of Teflon®, PVC or stainless steel. It is designed to collect liquid samples from groundwater monitoring wells, and may also be used to sample liquids in pits or tanks. Bailers may be dedicated, field cleaned, or disposable. For groundwater sampling at the Site, bottom-emptying, dedicated/disposable Teflon® bailers will be used to sample each well scheduled to be sampled.

- Peristaltic Pump and Tubing

  A peristaltic pump may be used to purge/sample the shallow wells at the Site. The pump consists of a circular series of rollers that successively squeeze a section of flexible tubing, creating a vacuum. A section of polyethylene tubing is connected to the pump's silicone tubing and lowered into the well to the desired depth. Flow is regulated with a rheostat-type controller located on the pump. The pumps run on internal batteries, or external AC or 12 volt DC power. Dedicated/disposable tubing will be used to sample each well at the Site.

- Submersible Pump and Tubing

  A Grundfos® 2-inch diameter submersible electric pump or similar DC submersible pump may be used to sample wells at the Site. The Grundfos® pump is constructed of stainless steel with a Teflon® impeller, and is easily decontaminated between wells. Other DC pumps that might be used will typically have a plastic or stainless steel body with plastic or Teflon impellers. Flow is regulated with a rheostat-type controller located at the surface. AC power is usually provided by a generator. DC power is typically supplied be a 12 volt battery. Polyethylene tubing connected to the pump will be dedicated/disposable and changed between well locations.

5.1.3 Equipment for Sampling Surface Water

Various pieces of field equipment that may be used to sample groundwater at the Site are described below.

- Extension Pole and Sample Bottle Cradle

Surface water samples will be collected directly into the appropriate laboratory bottles by attaching each unpreserved sample bottle to the sample bottle cradle affixed to the end of a 10- to 12-foot long extension pole.

- Hydrochloric Acid (HCl) and Litmus Paper

  The 40-ml sample vials destined for VOC analysis must be immediately preserved upon collection in the field to a pH less than 2.0. This will be accomplished using 1:1 HCL from a nalgene or glass dropper bottle and litmus paper/comparator chart.

5.1.4 Equipment for Sampling NAPL

Various pieces of field equipment that may be used to sample NAPL at the Site are described below.

- Peristaltic Pump and Tubing

  A peristaltic pump may be used to sample light non-aqueous Liquid (LNAPL) from shallow wells at the Site. The pump consists of a circular series of rollers that successively squeeze a section of flexible tubing, creating a vacuum. A section of polyethylene tubing is connected to the pump's silicone tubing and lowered into the well to just above the top of the LNAPL layer. Flow is regulated with a rheostat-type controller located on the pump. The pump runs on internal batteries, or external AC or 12 volt DC power. Dedicated/disposable polyethylene and silicon tubing will be used to sample LNAPL from wells at the Site.

5.2 Field Instrumentation

Brown and Caldwell may utilize various field instruments at the Site. All instruments will be operated according to the manufacturer's specifications. Instruments requiring calibration will be calibrated prior to daily use according to the manufacturer's recommendations. Calibration results will be documented in the field logbook.

5.2.1 Interface Probe

An interface probe (IP) will be used to measure liquid levels in monitoring wells and to measure LNAPL/DNAPL thickness, if any. Measurements obtained using an IP will be documented in the field logbook.

The depth to liquid(s) will be measured in existing monitoring wells using an IP. The probe will be lowered into the well until the meter indicates liquid has been contacted via emission of an audible tone. The IP emits different audible tones when encountering NAPL versus groundwater. Generally, a solid tone indicates NAPL, whereas an intermittent tone indicates groundwater. The depth from the surveyed reference mark atop the well inner casing to the top of the first liquid encountered will be measured on the tape at least three times or until a consistent value is obtained. The value will be recorded to the nearest 0.01 feet on field data sheets and/or the field logbook, along with the type of liquid encountered (LNAPL, dense non-aqueous phase liquid – DNAPL, or groundwater). The procedure will be repeated to measure the depth to the LNAPL/DNAPL/groundwater interface (i.e., the groundwater surface) where both phases are present. Due to the difficulties sometimes encountered in attempting to obtain an accurate depth to the LNAPL/DNAPL/groundwater interface, care will be taken when measuring this depth.

5.2.2 Groundwater Multi-Parameter Meter

Groundwater samples will be field tested with a multi-parameter meter in a flow-through cell. Groundwater will be tested during purging (in accordance with the procedure used for low-flow sampling if this technique is used) and just prior to sample collection to provide an indication of field parameter stability over time. The meter will

be used to measure pH, specific conductance, temperature, dissolved oxygen, and turbidity. A commercially available instrument will be used. All parameter values will be reported in the field logbook and/or field sample data sheets. Operation and calibration will be in accordance with instrument's owner manual. The instrument will be calibrated at a minimum of before each day of use.

5.2.3 Photoionization Detector

A photoionization detector (PID) may be used to detect total organic vapors during various field activities for the health and safety of field personnel. Use of the PID is described in the HASP.

5.2.4 Multi-Gas Detector

A multi-gas detector may be used to detect methane, hydrogen sulfide and the lower explosive limit (LEL) during various field activities for the health and safety of field personnel. Records will be maintained for all measurements taken including measurements taken during boring advancement. Use of the multi-gas detector is described in the HASP.

5.3 Sample Collection Procedures

5.3.1 Soil Sampling

Sampling during the investigation may include Geoprobe® soil borings, hollow-stem-auger (HSA) drill rig soil borings, and rock coring/drilling rigs. The sampling procedures are described in the following paragraphs. Upon completion of the field program, if necessary, each soil boring location will be surveyed for horizontal and vertical position by a licensed Ohio surveyor in accordance with the QA procedures described in the QAPP.

5.3.1.1 Geoprobe® Soil Borings

Geoprobe® soil borings will be sampled continuously from the ground surface to the total depth of the boring. Soil samples will be collected in four-foot-long macro-core samplers. An acetate liner will be placed within the sampler. Upon recovery, the sampler will be opened and the liner containing the soil will be removed.

The driller, or a BC representative, will slit the liner longitudinally and BC's field geologist will log the color, texture, and moisture content of the sample. This information, as well as the percent of recovery in the liner and sample depth, will be recorded in the field logbook. Any visual or other evidence of contamination such as staining or odors will also be recorded. Each boring will be clearly identified, and recorded.

Upon completion of each Geoprobe® boring, the boring will be backfilled with bentonite pellets or chips and hydrated. Soil boring cuttings will be spread on the ground surface unless they contain waste material or evidence of contamination (e.g., odor, staining, etc.), then they will be containerized as described in Section 5.7.

5.3.1.2 Soil Borings

Soil samples from select borings may be collected using standard hollow-stem-auger methods and split-spoon samplers. It is anticipated that any auger drilling will be performed with 4.25-inch inside-diameter, hollow-stem augers. The intervals identified for testing will be based on the lithology of the boring, and discussions with BC's Project Manager. The actual depths of the samples to be collected cannot be pre-determined in this FSP.

The boring at each location will be continuously sampled using split spoons in accordance with the SPT via ASTM Method D1586 (see Attachment A). This method consists of driving the split spoon sampler using a

140-pound weight (or auto-hammer) and 30-inch drop. The number of blows needed to drive the split spoon each six inches will be recorded.

Upon retrieving each split spoon, the SPT blow counts will be recorded in the field logbook, along with the percent recovery in the split spoon and the description of the soil, including depth of sample, color, texture, grain size, evidence of contamination, etc. A representative portion of each split spoon sample will be retained in a labeled zip-lock storage bag for head-space analysis.

Upon completion of each boring location, the boring will be backfilled with a bentonite/cement slurry via the tremie method to grade as the augers are removed. Soil boring cuttings will be spread on the ground surface unless they contain waste material or evidence of contamination (e.g., odor, staining, etc.), then they will be containerized as described in Section 5.7.

5.3.2 Groundwater Sampling

Groundwater sampling will be performed in accordance with SOP No. 2 in Attachment A. Groundwater sampling will consist of four tasks:

1. Water level measurement and immiscible layer detection;
2. Well purging;
3. Field testing for pH, specific conductance, temperature, dissolved oxygen, and turbidity; and
4. Sampling.

Before each well is sampled, the well will be inspected for integrity. Any non-dedicated sampling equipment will be decontaminated before sampling each well following the procedures described below. During a complete Site sampling event, a round of liquid level measurements will be obtained from all Site wells prior to purging/sampling begins. Measurements will be made consecutively in a minimal amount of time to avoid any effects of temporal fluctuation in water levels.

Wells will be purged and sampled with a submersible or peristaltic pump. Wells may also be sampled with a disposable bailer. Bottom-emptying bailers will be used when bailer sampling of VOCs is necessary.

During well purging, wells that recover in a reasonable amount of time will be purged of three well volumes and sampled as soon as possible thereafter. Low yielding wells will be purged to dryness. All wells will be sampled within 24 hours of purging. Field parameters such as pH, conductivity, temperature and turbidity of the groundwater will be monitored periodically during purging, and measurements will be noted in the field logbook and/or field forms.

Following purging, the well will be sampled according to SOP No. 2 in Attachment A which also discusses the prioritized order of collection of samples for various parameters. Samples for analysis for dissolved metals, if required, will be field filtered following the procedures specified in SOP No. 2.

5.3.3 Surface Water Sampling

Surface water samples will be collected directly into the appropriate laboratory bottles by attaching each unpreserved sample bottle to the sample bottle cradle affixed to the end of a 10- to 12-foot long extension pole. The pole is extended to the appropriate length from the river bank and the sample bottle is lowered below the water surface with the open end of the bottle facing down to limit the entrance of water until it reaches the desired sampling depth. This procedure will minimize disturbance of the water that enters the bottle and ensure the collection of representative samples. Once at the desired sample depth, the pole/bottle is slowly rotated to allow water to enter the sample bottle. When the bottle is fully rotated and is full of

water, it will be raised to the surface and retracted. This procedure will be repeated until the required laboratory sample bottles have been filled.

The 40-ml sample vials destined for VOC analysis must be immediately preserved upon collection in the field to a pH less than 2.0. Two drops of 1:1 HCl is generally sufficient to achieve the desired pH for most water samples. However, this will be verified by collecting an additional 40-ml sample vial of sample, adding two drops of 1:1 HCl then determining the pH with litmus paper/comparator chart to ensure the pH is less than 2.0. The number of drops of HCL will be adjusted accordingly to ensure that the pH of the VOC samples submitted to the laboratory is less than 2.0. This additional sample vial will be discarded and not submitted to the laboratory.

5.3.4 NAPL Sampling

A peristaltic pump may be used to sample light non-aqueous Liquid (LNAPL) from shallow wells at the Site. The pump consists of a circular series of rollers that successively squeeze a section of flexible tubing, creating a vacuum. A section of polyethylene tubing is connected to the pump's silicone tubing and lowered into the well to just above the top of the LNAPL layer. Flow is regulated with a rheostat-type controller located on the pump. The pump runs on internal batteries, or external AC or 12 volt DC power. The pump is activated at a low rate as the end of the tubing is slowing lowered into the LNAPL layer, taking care to pump only LNAPL and not water or air. The appropriate laboratory sample jars are filled directly from the discharge tubing. Dedicated/disposable polyethylene and silicon tubing will be used to sample each well at the Site.

5.4 Field Quality Control Samples

Field quality control samples that may be completed during the RD/RA or in subsequent work are discussed in the QAPP and summarized in the following subsections.

5.4.1 Trip Blank

One trip blank will be submitted to the laboratory with each shipment of samples for volatile organic analysis. The analysis of this blank will provide a baseline measurement of any contamination to which the samples may have been exposed during transport. A trip blank is comprised of a sample bottle provided by the laboratory filled with deionized, organic-free water, preserved, handled like a sample, and returned to the laboratory for analysis. Trip blanks will be prepared by the analytical laboratory and will be submitted for both liquid and solid samples.

5.4.2 Equipment Blank

One equipment blank will be collected per sampling episode per sampled medium or at a frequency equal to approximately 5% of the samples collected for each medium, whichever is larger. The analysis of these blanks serves to verify the cleanliness of the sampling equipment. An equipment blank is collected by rinsing decontaminated field equipment with water, transferring the water to a sample bottle, and submitting the sample for analysis. Deionized, organic-free water provided by the laboratory will be used for the field blanks. The equipment blank will be analyzed for the same parameters as the samples associated with that equipment.

5.4.3 Field Duplicates

Approximately 5% of all the samples will be collected in duplicate and submitted for laboratory analysis. Duplicates are two samples collected independently from one sampling location during a single episode of sampling. Duplicates provide information about sample variability.

5.4.4 Matrix Spike/Matrix Spike Duplicates

Matrix spike and matrix spike duplicates (MS/MSDs) are a quality control requirement performed by the laboratory. It is necessary to collect three times the usual required volume for aqueous samples to be analyzed as MS/MSDs. No additional sample volume is necessary for solid samples. At a minimum, additional volume for one sample will be provided to the laboratory for every group of 20 samples collected per medium and matrix.

5.5 Environmental Conditions

Sampling at the Site may be conducted at any time of year, regardless of the weather. Extreme heat or cold will require increased attention to health and safety issues, and may impact the duration of work. Equipment such as Geoprobe® or hollow-stem auger rigs require longer warm-up times in cold weather. Some areas of the Site may be impassable to vehicles and personnel during wet periods. Consideration shall be given to these and other relevant weather impacts before scheduling field work.

5.6 Decontamination Procedures

Sampling equipment decontamination procedures will vary depending on the field task. The various levels of decontamination to be performed between sampling locations for the various types of field activities are described below.

Level 1 (Split-Spoons, Trowels, macro-core samplers, extension pole, rock core barrels, etc.)

- Wash/scrub with potable water; and
- Potable water rinse.

Level 2 (IP, Water Level Meter and Probes)

- Rinse with laboratory detergent/distilled water solution; and
- Rinse with distilled water.

Level 3 (Sampling Equipment that comes in Direct Contact with the Analytical Sample Media)

- Wash/scrub with laboratory detergent/distilled water solution;
- Distilled water rinse,
- Laboratory-grade methanol rinse (if organic analyses are planned);
- Distilled water rinse;
- Laboratory-grade 10% nitric acid rinse (if metals analyses are planned); and
- Final distilled water rinse.

The waste water generated from the decontamination procedures will be containerized and managed as described in Section 5.7.

5.7 Investigation Derived Waste

Investigation derived waste (IDW) generated from field activities will be containerized in labeled 55-gallon DOT-approved steel drums and staged at a pre-designation location at the Site for characterization and disposal. Information contained on the label will include the drum contents, name, address and telephone number of generator, date(s) the material was placed in the drum, and a BC contact name/telephone number. Wastes will be separated based on type. For example, separate drums will be filled for solid landfill waste, contaminated soil, monitoring well development and purge water, decontamination wastewater,

LNAPL/DNAPL, used personal protective equipment (PPE), and general trash. If a sufficient quantity of general trash is expected, then a dumpster or roll-off might be staged for this material instead of drums.

For liquid wastes, characterization of the drummed IDW will consist of collecting a composite sample from drums of similar contents and sending the sample to the analytical laboratory to be analyzed for the parameters required by the designated waste receiving facility. Characterization sampling will occur as soon as possible following field activities to ensure compliance with all local, state and federal regulations regarding storage of IDW.

Investigation generated wastes will be disposed in accordance with applicable Federal, State, and Local regulations.

6. SAMPLE HANDLING AND CUSTODY

An important aspect of field sampling quality assurance involves proper sample handling and strict chain of custody protocol. Improper sample handling may lead to results that are not representative of the materials sampled. BC will take precautions while handling samples to maintain the integrity of the sample. Such precautions include employing proper sample transfer procedures, use of proper sample containers, use of proper sample preservation techniques, and observing sample holding times.

6.1 General Sampling Protocols

6.1.1 Sample Containers

Table 6-1 describes the sample containers needed for the various potential analytical analyses.

6.2 Chain-of-Custody

The chain-of-custody procedures are initiated in the field following sample collection. The procedures consist of: (1) preparing and attaching a unique sample label to each sample collected, (2) completing the chain-of-custody (COC) record, and (3) preparing and packing the samples for shipment. These procedures are briefly described in the following subsections.

6.2.1 Sample Labels

Field personnel are responsible for uniquely identifying and labeling all samples collected during the field investigation program. All labeling must be completed in indelible/waterproof ink and securely affixed to the sample container.

All sample containers will be affixed with label that will contain the following information:

- Sample identification (see Section 4.1); Depth of sample (if appropriate);
- Analysis/test to be performed;
- An "up" arrow (Shelby tube samples only)
- Preservative (if required);
- Project name and number;
- Date and time of sample collection; and
- Initials of Sampler

6.2.2 Chain-of-Custody Record

The COC record, an example shown in Figure 6-1, will be completed for the geotechnical samples submitted to the laboratory. These forms are maintained as a record of sample collection, transfer, shipment, and receipt by the laboratory. These forms also contain pertinent information concerning sampling location, date, and times, signatures of at least one team member; types of samples collected along with a unique sample identification

number; the number of samples collected and shipped for testing, the requested tests, the project number, and the name of the laboratory to which the samples are being sent.

6.2.3 Transfer of Custody

Samples will be accompanied by COC form during each step of custody, transfer, and shipment. When physical possession of samples is transferred, both the individual relinquishing the samples and the individual receiving them will sign, date, and record the time on the COC form.

6.3 Sample Packaging and Shipment

Following sample collection, all samples will be brought to an onsite location for batching and paperwork checks. Label information is checked to ensure there is no error in sample identification. The samples will be packaged to prevent breakage and/or leakage, and staged awaiting hand delivery to the geotechnical laboratory on a weekly basis. Rock core samples destined for petrophysical and chemical testing will be handled in accordance with the applicable ASTM/API methods and SOPs contained in Attachment A.

7. REFERENCES

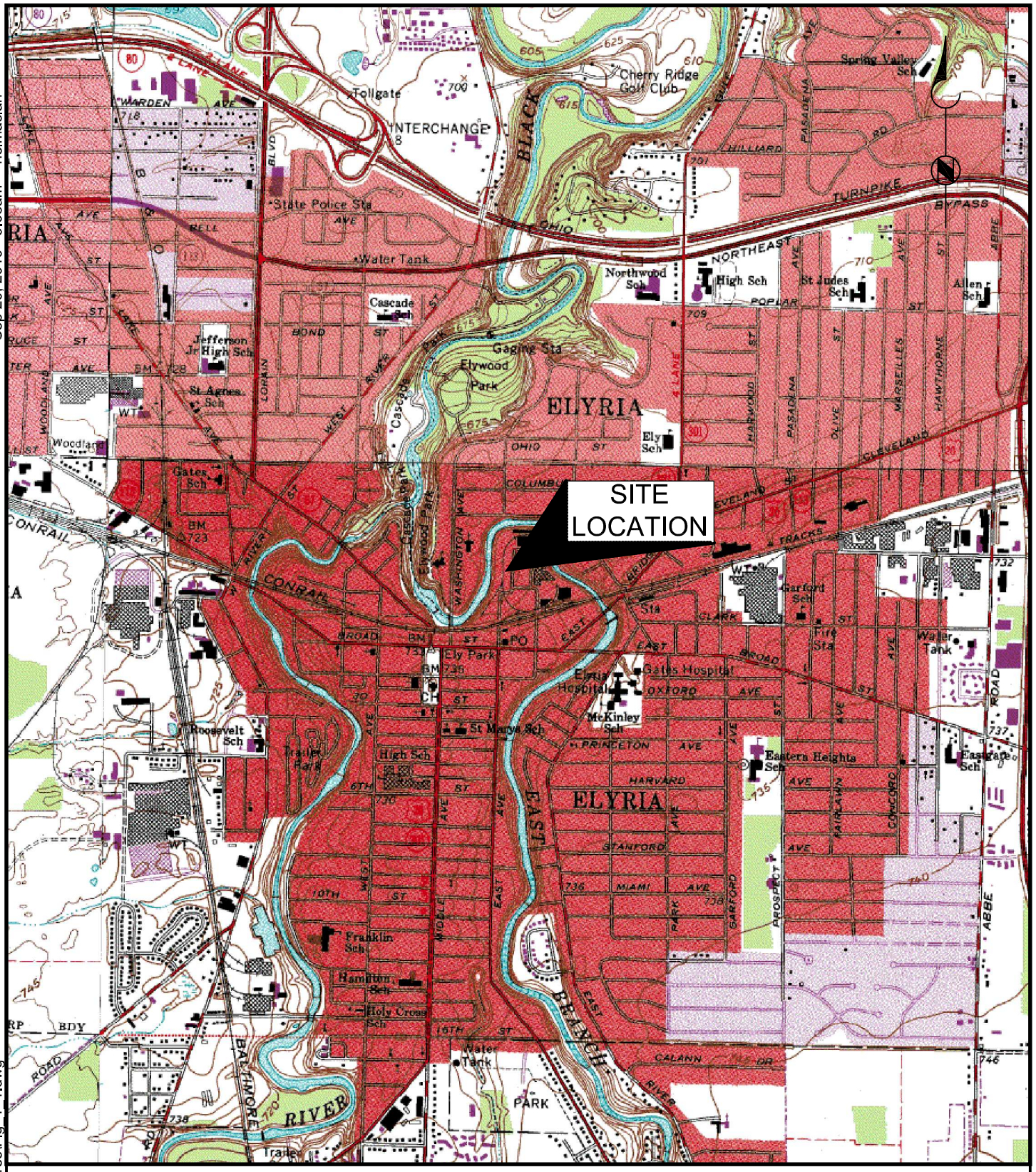
American Society of Testing Materials (ASTM), 2008. *Annual Book of ASTM Methods*, 2008.

Parsons, 2006. *Remedial Investigation Report – Chemical Recovery Systems, Inc., Revision 3, September, 2006.*

Parsons, 2006. *Feasibility Study – Chemical Recovery Systems, Inc., Revision 3, September 2006*

USEPA, 1988. *Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA, October, 1988*.

FIGURES



CHEMICAL RECOVERY SYSTEMS, INC. SITE
ELYRIA OHIO

SOURCE: BASE MAP USGS 7.5 MIN. QUAD., AVON, OH, 1994

2000 1000 0 2000

SCALE IN FEET

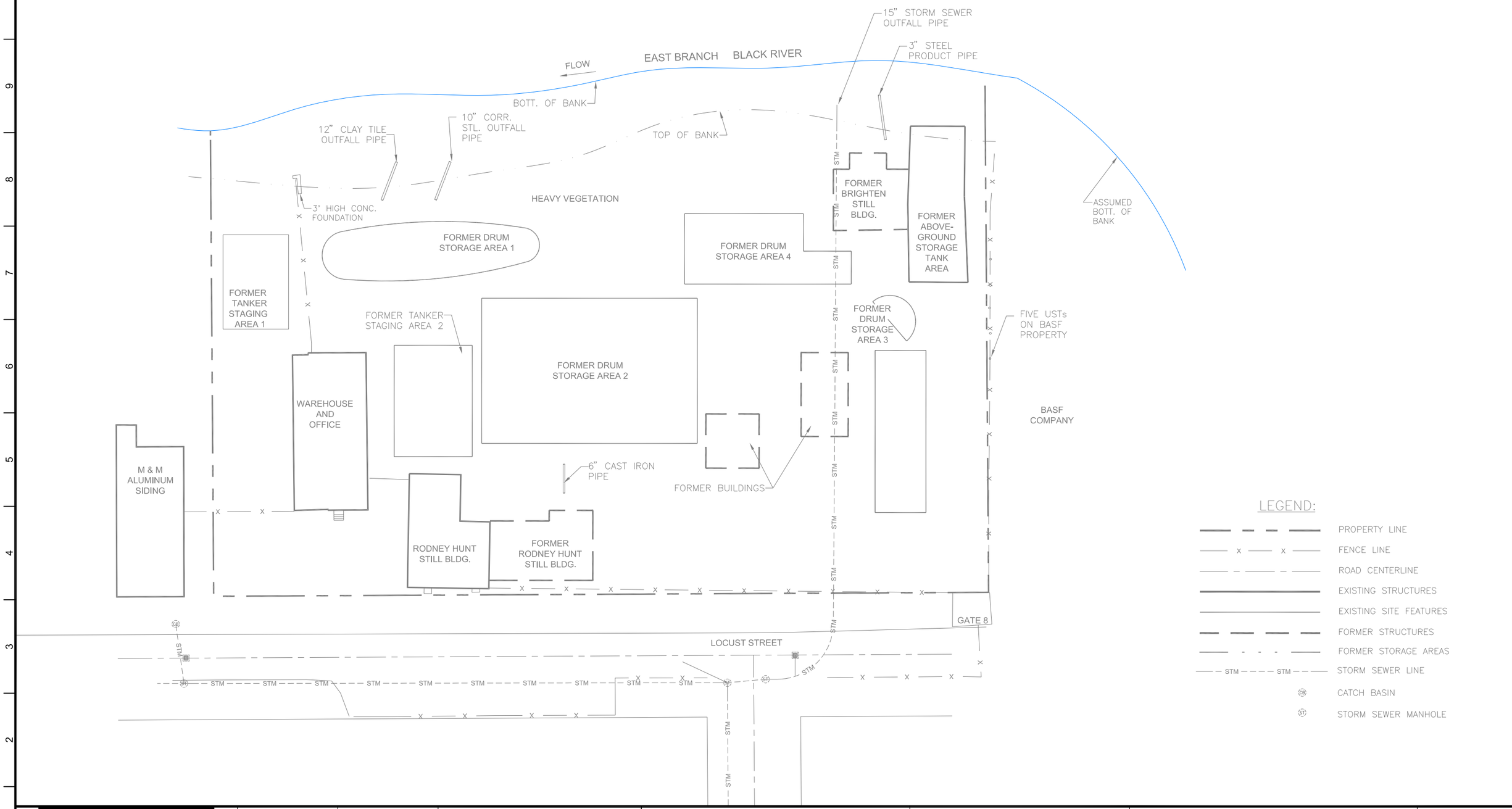
OHIO
QUADRANGLE LOCATION
LATITUDE 41° 22' 15"
LONGITUDE 82° 06' 12"

Sep 20, 2010 - 9:08am nelmasian

P:\CRS\Drawings\CAD\Figures\Fig_1_1.dwg

Brown AND Caldwell

Figure 1-1
SITE LOCATION



P:\CRS\DRAWINGS\CAD\FIGURES\FIGURES.DWG September 15, 2010

September 15, 2010

Brown AND Caldwell
COLUMBUS, OHIO

SUBMITTED: _____ DATE: _____
PROJECT MANAGER

APPROVED: _____ DATE: _____
BROWN AND CALDWELL

LINE IS 2 INCHES AT FULL SIZE
(IF NOT 2" - SCALE ACCORDINGLY)

DESIGNED:
DRAWN: NE
CHECKED:
CHECKED:
APPROVED: JP

EXTERNAL REFERENCE FILES

REVISIONS					
ZONE	REV.	DESCRIPTION	BY	DATE	APP.

0 25 50
SCALE IN FEET

CHEMICAL RECOVERY SYSTEMS SITE
ELYRIA, OHIO

SITE LAYOUT

FILENAME
FIGURES.DWG
BC PROJECT NUMBER
XXXXXX
CLIENT PROJECT NUMBER
FIGURE NUMBER
1-2

TABLES

Table 6-1

ANALYTICAL SAMPLE VOLUME AND CONTAINERS
Chemical Recovery Systems, Inc.
Elyria, Ohio

Analytical Method	Solid Matrix			Aqueous Matrix		
	Sample Container	Preservative	Holding Time	Sample Volume	Preservative	Holding Time
VOCs	2-ounce glass[1]	4° C	14 days	3 x 40=ml glass[2]	HCl to pH <2; 4°C	14 days
SVOCs	4-ounce glass[1]	4° C	14 days	2 x 1-liter amber glass[2]	4° C	7/40 days[3]
Metals	4-ounce glass[1]	4° C	180 days/ 28 days for Hg	500-ml glass or plastic	HNO_3 to pH<2; 4° C	180 days/28 days for Hg
PCBs	4-ounce glass[1]	4° C	14 days	2 x 1-liter amber glass[2]	4°C	7/40 days[3]

Notes:

1- Wide-mouth glass jar with PTFE-lined lid.

2-Glass with Teflon-lined cap

3-7 days until extraction and 40 days until analysis

ATTACHMENT A - SOPS AND ASTM/API STANDARDS

STANDARD OPERATING PROCEDURE (SOP) NO. 1

MONITORING WELL INSTALLATION AND DEVELOPMENT

Brown and Caldwell
Standard Operating Procedure

Groundwater Monitoring Well Installation and Development

Revision 1.0
Revision Date: October 31, 2000

Prepared/Revised by:	**Rebecca Frend**	October 31, 2000
	Name	Date
Senior QA Review:	Jay Lucas	10/26/01
	Name	Date
Regional Quality Officer:	Daniel Van Arnhem	October 26, 2001
	Name	Date

GROUNDWATER MONITORING WELL INSTALLATION

TABLE OF CONTENTS

1.0 OBJECTIVES 1
2.0 SCOPE AND APPLICABILITY 1
2.1 Health and Safety 2
3.0 RESPONSIBILITES 2
4.0 REQUIRED MATERIALS 3
5.0 METHODS 4
5.1 Drilling Methods 4
5.1.1 Hollow Stem Auger Methods 4
5.1.2 Mud Rotary Methods 5
5.1.3 Air Drilling Methods 5
5.1.4 Rotosonic Drilling 6
5.2 Borehole logging 7
5.3 MONITORING WELL CONSTRUCTION PROCEDURES 7
5.3.1 Backfilling. 8
5.3.2 Well Casing and Screen 9
5.3.3 Filter Pack 11
5.3.4 Well Sealing Material 13
5.3.5 Surface Completions 13
5.3.6 Monitoring Well Location and Surveying 14
5.4 Well Development 15
5.5 Disposal And Decontamination 17
6.0 QUALITY ASSURANCE/QUALITY CONTROL 18
7.0 RECORDS 18
8.0 REFERENCES 19
9.0 ATTACHMENTS 19

1.0 OBJECTIVES

The objective of this standard operating procedure (SOP) is to provide the methods to be used for the installation and development of groundwater monitoring wells and to provide standardized reporting formats for documentation of data. This SOP has been specifically designed with the objective of installing and developing wells for environmental investigations.

2.0 SCOPE AND APPLICABILITY

This procedure is intended for use by Brown and Caldwell for the installation, development, and documentation of monitoring wells that will be used for environmental investigations.

Specific monitoring well design and installation procedures depend on project-specific objectives and subsurface conditions and should be discussed in project-specific planning documents. The following aspects will need to be determined when planning a well installation:

- Borehole drilling method
- Construction materials
- Well depth
- Screen length
- Well construction materials
- Location, thickness, and composition of annular seals
- Well completion and protection requirements.

Groundwater monitoring well installation and development will be performed in accordance with applicable well standards for the area of the investigation, this SOP and the project-specific planning documents. Drilling methods employed to pilot the borehole for monitoring well installation will be dependent on the physical nature of

the subsurface materials (unconsolidated materials and/or consolidated materials) at the site. The drilling contractor shall be a licensed water well driller, in accordance with local and state requirements, and a qualified drilling contractor for the installation of groundwater monitoring wells for environmental investigations.

2.1 Health and Safety

Potential physical and chemical hazards will need to be addressed when planning monitoring well installation. A health and safety plan that addresses known and anticipated field conditions must be prepared prior to field work and be followed during well installation.

3.0 RESPONSIBILITES

The project manager is responsible for ensuring that the project involving monitoring well installation is properly planned and executed and that the safety of personnel from chemical and physical hazards associated with drilling and well installation is provided for.

The field geologist or engineer is responsible for directly overseeing the construction and installation of the monitoring wells by the driller and to ensure that the project specific well-installation specifications defined in the project-specific planning documents are followed and that pertinent data are recorded on appropriate forms and in the field notebook. Monitoring well construction and boring completion will be conducted under the supervision of an appropriately qualified and registered person as defined by local regulations.

The site safety officer, typically the field geologist or engineer, is responsible for overseeing the health and safety of Brown and Caldwell employees and for stopping work if necessary to fix unsafe conditions observed in the field. If a subcontracted

firm conducts installation and documentation activities, then the firm will designate a site safety officer.

4.0 REQUIRED MATERIALS

Many materials are required for successfully completing the installation and development of monitoring wells. The drilling Subcontractor often supplies much of the material. However, the field personnel should be aware of what is required to conduct the work so they have their own supplies and can provide complete Subcontractor oversight. The following is a general list of materials that are needed for performing the tasks outlined in this SOP.

Geologist

- Hand lens
- Health and Safety supplies (e.g., steel toed boots, gloves, hard hat, etc.)
- Lithologic Logs and Well completion forms
- Logbook
- Logging assistance tools (e.g., grain size charts, color charts)
- Measuring tapes (both long weighted cloth type and small measuring tape, preferably marked in tenths and hundredths of a foot)

Drilling Subcontractor

- Drilling equipment (depends upon the type of drilling, e.g., drill stem, auger, generators, compressors, steam cleaners, etc.)
- Well drilling supplies (drilling mud,
- Decontamination Pad construction supplies
- Well construction supplies (screen, well casing, sand pack, bentonite chips, bentonite, cement mixture, water).

- Health and safety records required for working on site
- Driver's licenses
- Ancillary support vehicles
- General tools

5.0 METHODS

The borehole diameter must be a minimum of four (4) inches greater than the outside diameter of the well screen or riser pipe used to construct the well. This is necessary so that sufficient annular space is available to install filter packs and grout seals. All boreholes will be cleared for shallow obstructions by following the SOP for Utility Clearance.

5.1 Drilling Methods

Several drilling methods are available for use in creating a borehole for well installation. These methods include hollow stem, air rotary, mud rotary, and cable tool, among others. The drilling method selected will be based on the physical properties of the subsurface materials.

5.1.1 Hollow Stem Auger Methods

Hollow stem auger uses continuous flight hollow stem auger with a bit on the bottom to drill and maintain an open borehole. The continuous flight auger drives the drill cuttings to the surface as drilling progresses. The walls of the auger minimize the amount of unconsolidated materials entering into the space inside the casing. Intact soil samples are collected by pounding a sampler ahead of the auger. The well casing, filter pack and seal are installed inside the auger. The auger is removed slightly ahead of backfilling as filter pack and grout are added. Careful recording of the amount of each material used should be recorded in the field logbook.

5.1.2 Mud Rotary Methods

Mud rotary drilling uses drilling fluids to circulate drill cuttings to the surface. Drilling fluid will consist of only uncontaminated air, water or uncontaminated water mixed with bentonite. Powdered bentonite or an approved equivalent will be used as an additive in the drilling fluid. Bentonite will be mixed into the drilling fluid using a mud mixer and a portable mud tank. Drilling fluid density and viscosity will be maintained at appropriate levels for the various lithology encountered and in accordance with material specifications.

A shale-shaker and de-sanding system will be used to maintain the density and viscosity of the drilling fluid. Sand content will be minimized to the degree possible by maintaining no greater than 4 percent sand by mud volume.

If water or other drilling fluids have been introduced into the borehole during drilling or well installation, samples of these fluids should be obtained and analyzed for chemical constituents that may be of interest at the site. In addition, an attempt should be made to recover the quantity of fluid or water introduced by flushing the borehole before well installation and/or by pumping the well during development.

5.1.3 Air Drilling Methods

The following are descriptions of air rotary, "down-the-hole", and dual-wall reverse circulation air rotary methods. Air rotary uses air as a primary means of transporting drill cuttings to the surface. A large compressor provides filtered air that is piped to the swivel hose connected to the top of the Kelly bushing or drill pipe. The air, forced down the drill pipe, escapes through small ports at the bottom of the drill bit, thereby lifting the cuttings and cooling the bit. The cuttings are blown out the top of the hole and are collected at the surface in a cyclone unit and a

two- to four-yard roll-off container. Injection of a small volume of clean water into the air system controls dust and lowers the temperature of the air so that the swivel is cooled. Air drilling is effective in semi-consolidated or consolidated materials.

A second direct rotary method using air is called the "down-the-hole" or percussion down hole hammer drilling system. A pneumatic drill operated at the end of the drill pipe rapidly strikes the rock while the drill pipe is slowly rotated. The percussive effect is similar to the blows delivered by a cable tool bit. Cuttings are removed continuously by the air used to drive the hammer.

A third direct air rotary method is called the Air Rotary Casing Hammer (ARCH) method is used where an outer steel casing is advanced slightly behind the drill bit. The drill bit reams material in front of the casing and then the casing is advanced with a pneumatic hammer down the hole to prevent hole collapse. Cuttings are collected in a tube system that conveys them into a cyclone at the surface.

Dual-wall reverse circulation air rotary method uses flush-jointed, double wall pipe in which the air moves by reverse circulation. The airflow is contained between the two walls of the dual-wall pipe and only contacts the walls of the borehole near the bit. Dual-wall pipe can be driven into place in loosely consolidated materials by a pile hammer as a drive bit is cutting the formation. Downhole air hammers and tricone bits can also be used to cut the formation. The air lifts the cuttings to the surface through the inner pipe. Dual-wall methods can be applied in consolidated and unconsolidated formations.

5.1.4 Rotosonic Drilling

This section to be added as needed later.

5.2 Borehole logging

Boreholes will be logged using cuttings and samples collected during drilling activities. Soil or rock samples will be collected as described in the SOP for Soil Sampling. Cuttings and soil and rock samples will be described at the frequency presented in the project-specific planning documents following the procedures outlined in SOP for Field Classification and Description of Soil and Rock.

After drilling has been completed, the field geologist/engineer will measure the total open depth of the borehole with a weighted, calibrated tape measure and document the depth. The field geologist will then collaborate with the supervising geologist by reviewing lithologic units encountered, water levels, if any, and other logged information to determine the well construction details.

Boreholes/well locations should be clearly designated in the field notes using notes and a hand sketched layout and should include the following information:

- Measurements of each boring/sample point relative to fixed objects (building, structures, etc),
- Boring/sample location with their identification number noted,
- North arrow or other compass directional indicator, and
- Other essential site features and/or investigation features (underground storage tanks, piping, above ground tanks, etc.).

5.3 MONITORING WELL CONSTRUCTION PROCEDURES

Monitoring wells will be constructed in accordance with state and local agency requirements, and will include at a minimum the following materials:

- Borehole backfill for overdrilled boreholes prior to well installation,
- Well casing and screen

- Filter pack materials
- Well sealing materials (e.g., bentonite pellets, cement, powdered bentonite), and
- Surface seals and materials for well surface completion (e.g., concrete, protective steel casing, steel posts, surface boxes).

A discussion of these materials and how they are used is provided in more detail in the following sections.

5.3.1 Backfilling.

If backfilling the borehole to the appropriate well installation depth is necessary, neat cement, bentonite grout, bentonite pellets or filter pack sand may be used. The backfill material selected for use will depend on site conditions, lithology, and project-specific requirements. Most often the borehole requires complete sealing with lower layers, so neat cement, bentonite grout, or bentonite pellets are used. The setup time should be a minimum of 48 hours for neat cement and 24 hours for bentonite grout and bentonite pellets prior to beginning well construction. Field personnel should remeasure and verify that the bottom of the bore hole is exactly where it should be set before proceeding with well construction. The necessary setup times may be reduced if manufacturer- approved additives are mixed with the grout to accelerate the cure time.

If neat cement or bentonite grout is used, a tremie pipe will be required to place the grout in the bottom of the hole. Grouting the borehole may be difficult to accomplish, if the portion of the borehole to be grouted is significantly lower than the groundwater level. Provisions will be necessary to support the screen and riser pipe to prevent them from sinking into the grout. Care will be taken to frequently measure the total borehole depth when adding grout to the bottom of the hole.

Grout should have thickened to a hardened state before proceeding. The thickness of the grout will be calculated based on depth readings and recorded. If a well has been backfilled too much it may require reaming to clear out the overfilled material.

Depending upon the lithology some distance should be planned between the fill in a borehole and the bottom of the screened interval. Unless this distance would result in a breach confining layer, or the well screen requires setting directly on the impermeable zone due to site requirements, the bottom of the well screen should be set at a maximum of 6 inches above the top of any backfill. The distance between the top of fill and the bottom of the well screen should be filled with a fine sand buffer.

Bentonite pellets should be carefully dropped into the borehole to minimize the risk of pellets sticking to the side of the borehole when dropped through a water column. Pellets are generally easier to place than bentonite chips because pellets do not hydrate as quickly, hence pellets are the preferred method for small backfill jobs where significant confining zones have not been breached.

5.3.2 Well Casing and Screen

The monitoring well will consist of factory-sealed commercially available well screen and casing. Well screens and casing will typically be constructed of polyvinyl chloride (PVC), a type of plastic, but may also be constructed of stainless steel or Teflon depending on subsurface conditions or other project requirements. Stainless steel casing shall meet one of the following standards: American Society For Testing Materials (ASTM) A-53-93A or B, A-589-93, or American Petroleum Institute 5L, March 1982 Edition to conform to the minimum standards given in Table A of that document.

Plastic casing and liners shall meet the requirements of ASTM Standard F480-94 and the National Sanitation Foundation (NSF) International Standard Number 14-1990, Plastic Piping System Components and Related Materials. Evidence of compliance shall be included in the current NSF listing and display of the NSF seal on each section of casing, and marking the casing in accordance with the requirements of ASTM Standard F-480-94. Plastic well casing and liners must be Standard Dimension Ratio (SDR)-rated and conform to the minimum requirements given in Table B of the above-referenced document.

Well screens shall be constructed of non-corrosive and non-reactive material. Well screens shall be permanently joined to the well casing and shall be centered in the borehole. The anticipated length of screen and the reasoning behind choosing the length of screen will be determined when developing the project-specific planning documents. Modification can be made in the field, but will be done in consultation with the PM, or their designee such as the Project Technical Manager or Responsible Geologist.

Screen slot type and size will be dependent on the sand pack material and the aquifer formation material. Casing will be connected by flush-threaded or coupled joints and will be completed with a bottom cap. A collection sump may be installed below the screen and will vary in length depending on lithology and project needs. The collection sump and bottom cap will be connected to the well screen by flush threaded or coupled joints. Plastic casing must have threaded joints and O-ring seals. Solvent, glue, or anti-seize compounds will not be used on the joints. With deep wells (greater than approximately 100 feet below grade), centralizers should be used to keep the well casing plume and straight in the borehole. Centralizers should be placed at approximately 20 foot intervals in the screen interval and 40 foot intervals throughout the blank casing interval.

For water table wells, well screens should be placed such that some of the screened interval is above the water table, and some section is below the water table. This allows for seasonal fluctations. The amount of split should be determined by the lead responsible geologist and be based upon local conditions.

Casing and screen (well string) must be clean, free of rust, grease, oil or contaminants and be composed of materials that will not affect the quality of the water sample. All casing shall be watertight. The casing shall be centered in the borehole, be free of any obstructions and allow sampling devices to be lowered into the well. The well string shall be hung in the borehole during installation so that the well is sufficiently plumbed and straight after completion.

5.3.3 Filter Pack

Monitoring wells installed in unconsolidated material will be constructed with filter packs. When used, the filter pack will be the only material in contact with the well screen. The filter pack will consist of sand or gravel. The sand or gravel used for filter pack material shall be sized to match the screen slot size and the surrounding lithology to prevent subsurface materials from penetrating through the sand or filter pack, and preventing the sand or filter pack from entering the well. Sizing of the filter pack material is often conducted using sieve analysis and following interpretative procedures outlined in Driscoll (1986). The sand or gravel shall be free of clay, dust, and organic material. Crushed limestone, dolomite, or any material containing clay or any other material that will adversely affect the performance of the monitoring well shall not be used as filter pack. The filter pack will extend a maximum of six (6) inches below the bottom of the screen to two (2) to three (3) feet above the top of screen. The filter pack material may be placed in the well by pouring the sand into the open borehole, or tremied into place depending upon site-specific criteria. However, in all cases, filter pack material should be

added carefully with continuous measurements by the field geologist to prevent bridging of the filter pack material.

Groundwater wells completed into competent bedrock material are often not completed with filter pack material, and can be completed as an open hole over the screened interval. Completion in this manner should be carefully considered and approved by regulatory agencies prior to field mobilization.

The well will be gently bailed and surged with a bailer and surge block after the filter pack has been added to the borehole and before the seal is placed in the annular space. A surge block consists of a rubber or leather and metal plunger attached to a rod or pipe of sufficient length to reach the bottom of the screen. Surging should be maintained for at least five minutes and the entire length of saturated screen will be surged to help settle the filter pack. The top of the filter pack will need to be gauged after surging and additional filter pack material may need to be added if settling has occurred.

Sometimes project specific requirements may identify that a transition sand be emplaced above the main filter pack. This transition sand is usually much smaller grain size than the filter pack, and is emplaced to provide added protection that grout invasion into the filter pack will not occur when deep wells (greater than 200 feet deep) are installed. Transition sands can be emplaced up to 10 or 20 feet above the regular sand pack interval. An alternative to transition sands is to use additional well seal material such as bentonite pellets.

5.3.4 Well Sealing Material

The wells will have an annular space seal that extends from the top of the filter pack to the surface. The annular sealing material above the filter pack will prevent the migration of fluids from the surface and between aquifers. Sealing material will be chemically compatible with anticipated contaminants. Hydrated bentonite chips or pellets are typically used as an annular seal directly above the filter pack. The annular seal should be a minimum of 3 feet thick unless site-specific requirements dictate otherwise. For example, as mentioned above, deep wells may require additional sealant material (10 to 20 feet thick versus 3 feet) between the sand pack and cement ground annular fill above to prevent grout invasion into the filter pack interval. Cement and/or bentonite grout are typically used as annular fill above the seal. Above the sealant material a bentonite grout mixture is often used as an annular fill to complete the well installation to within 2 feet of the surface. Grouting emplacement will occur using a tremie pipe so that the grout fills the annular space from the bottom to the surface without allowing air pockets to form in the filled zone.

5.3.5 Surface Completions

Above Grade or Monument Surface Well Head Completion

With above-grade well completions, the well casing will extend to 1 to 2 feet above the ground surface. A locking cap will be placed at the top of the casing and the cap will be watertight. The section of casing that sticks up above ground will be protected by a steel protective pipe, set at least 2-feet deep into a concrete surface seal. A concrete pad should be constructed around the protective steel pipe. The pad should be square, approximately 1.5-by-1.5 to 2-by 2-feet, sloped slightly away from the well, and the top of the pad should be approximately 4-inches off the ground. Specific client needs may differ from this construction, and such requirements

should be outlined in project specific planning documents. The top of the protective pipe will have a vented lockable cap. Protective steel posts will be installed in areas where the well could be struck by vehicles or heavy equipment. In addition, a "weep" hole should be drilled in the bottom of the protective steel pipe. In areas where freezing may occur, placement of the weep hole is critical; little volume should exist in the protective casing above the weep hole where water could accumulate and freeze thereby damaging the well. A "V" notch or other permanent mark will be placed at the north edge of the top of the well casing that will be used as the reference point for well elevation surveying and water level monitoring.

Ground or Grade Surface Well Head Completion

Monitoring well casing may terminate at the ground surface with a flush mounted traffic-rated road box. Road box installations must use a watertight well cap for the well riser pipe in addition to a watertight road box to prevent surface water from entering the well. The well casing should extend approximately 3 inches above the sealant in the bottom of the well box. The traffic-rated road box and surface concrete completion should meet Class A specifications, which meet a minimum 4000-pound compressive strength. The surface completion should provide positive drainage away from the well box to prevent ponding around the well. In traffic areas and sidewalks, this positive drainage slope away from the box should be minimized to prevent physical hazards. The surface seal around the box should be a minimum of 12 inches around the perimeter of the box. As discussed above a reference mark should be placed on the top of the well casing for well elevation surveying and water level monitoring.

5.3.6 Monitoring Well Location and Surveying

Monitoring wells will be located by parcel coordinates required by local permit requirements. Each well will be surveyed by a licensed surveyor in the state where

the well has been installed and tied to an established state or county benchmark, site conditions permitting. The vertical survey will be accurate to 0.01 foot relative to mean sea level. Both the top of casing and ground surface elevation near the well will be surveyed for vertical control. The "V" notch cut on the north side of each well casing will be used as the surveyor's reference mark. For horizontal control, each well will be tied to an existing site coordinate system and will be surveyed to a horizontal accuracy of 0.1 foot.

5.4 Well Development

Monitoring well development is necessary to ensure that complete hydraulic connection is made and maintained between the well and the aquifer material surrounding the well screen and filter pack. The appropriate development method will be selected for each project on the basis of the circumstances, objectives, and requirements of that project.

The appropriate development method will be selected for each project based on the lithology, objectives, and requirements of that project. Project-specific planning documents will identify the specific development method to be used. In general, most wells will be developed by using surge block and bailing methods to draw the coarse and/or fine material out of the sand pack. Other development methods that may be used include jetting, airlift, and submersible pump methods. These methods are discussed further below. Jetting is typically not used as a development method for environmental investigations, but is commonly used for water resource monitoring wells or production wells.

Well development should begin no sooner than 48 hours after well installation. However, if drilling muds are used during well installation, well development should occur approximately 24 hours following well installation so that the drilling mud does not set up in the well screen section.

Generally a phased process is used to develop wells, starting with a gentle bailing phase to remove sand, followed by a surging phase, then a pumping phase after the well begins to clear up. The following paragraphs provide more detailed information.

After a well is first installed, and in fact, often before the bentonite pellet seal is set, gentle bailing is used to remove water and sand from the well. The purpose of this technique is used to settle the sand pack. After further well sealant materials have been added and allowed to set for approximately 48 hours, bailing is resumed as part of well development. The purpose of bailing is to remove an fine material that may have accumulated in the well, and start pulling in natural material into the sand pack. Bailing is often conducted until the sand content in the removed water begins to decrease.

After the sand content begins to decrease, surging is conducted. A surge block is used to move sediments from the filter pack into the well casing. A surge block consists of a rubber (or leather) and metal plunger attached to a rod or pipe of sufficient length to reach the bottom of the well. All surge blocks will be constructed of materials that will not introduce contamination into the well. Surge blocks should have some manner of allowing pressure release to prevent casing collapse. The surge block is moved up and down the well screen interval and then removed, followed by a return to bailing to remove any sand brought into the well by the surging action. Care should be taken to not surge too strongly with subsequent casing deformation or collapse; the well screen interval is often the weakest part of a well. Surging should be followed by additional bailing to remove fine materials that may have entered the well during the surging effort.

After surging has been completed and the sand content of the bailed water has decreased, a submersible pump is used to continue well development. The pump

should be moved up and down the well screen interval until the obtained water is relatively clear. Well development will continue until the water in the well clarifies and monitoring parameters such as pH, specific conductivity, and temperature stabilize as defined in the project-specific planning documents. It should be noted that where very fine-grained formations are opposite the screened interval, continued well development until clear water is obtained might be impossible. Decisions regarding when to cease development where silty conditions exist should be made between the field supervisor and PM.

During well development pH, specific conductivity, temperature, and turbidity should be monitored frequently to establish natural conditions and evaluate whether the well has been completely developed. The main criteria for well development is clear water (Nephelometric turbidity units or NTU of less than 5). As mentioned above, clear water can often be impossible to obtain with environmental monitoring wells. A further criteria for completed well development is that the other water quality parameters mentioned above stabilize to within 10 percent between readings over one well volume.

The minimum volume of water purged from the well during development will be approximately a minimum of 3 borehole volumes (wells will typically not reach stabilization of water quality parameters before this condition is achieved and may not have reached stability even after this threshold has been achieved). The above is a general guideline for difficult well development - project-specific planning documents should address project constraints on well development. Development water will be stored in 55-gallon Department of Transportation (DOT) -approved drums and/or baker tanks depending upon the total volume of purge water removed from the newly installed wells.

5.5 Disposal And Decontamination

All drill cuttings and fluids generated during well installation and development will be containerized pending analytical results and determination of disposal options as outlined in the Investigation-Derived Waste Handling SOP unless project-specific requirements unless specify otherwise. Waste containment and disposal will occur in a manner that will not result in contamination of the immediate area or result in a hazard to individuals who may come in contact with these materials.

All drilling and well construction equipment that comes into contact with the borehole will be decontaminated by following the Equipment Decontamination SOP.

6.0 QUALITY ASSURANCE/QUALITY CONTROL

Borehole drilling and well construction details will be documented in detail in the field. Field documentation forms will consist of a lithologic borehole log, a well construction log, and daily field note forms. Examples of these forms are included in Attachment A. Deviations from project-specific planning documents will be documented and explained in daily field notes. The program manager will be contacted to discuss project deviations.

Field quality control can be maintained through 1) making sure employees are properly trained to conduct the work being implemented, and 2) performing routine field audits to evaluate how well employees are following procedures. These two aspects of QA/QC are detailed in the Quality Assurance Program documentation.

7.0 RECORDS

Field notes and logs will be submitted to the Project Manager or designate immediately following the field event for checking and revision purposes. The Project Manager or designate shall review and transmit the completed forms for incorporation into the project file.

8.0 REFERENCES

California Department of Water Resources, 1981, State of California Water Well Standards, Bulletin 74-81.

California Department of Water Resources, 1990, State of California Water Well Standards (modified), Bulletin 74-90.

Driscoll, F.G., 1986, Groundwater and Wells, Second Edition, Johnson Filtration Systems, Inc., St. Paul, MN.

United States Environmental Protection Agency, 1989, Handbook for Suggested Practices for the Design and Installation of Monitoring Wells, EPA 600/4-89/034, Reprinted by the National Ground Water Association.

9.0 ATTACHMENTS

Attachment A – Well Construction and Lithologic Log Form

ATTACHMENT A

WELL CONSTRUCTION AND LITHOLOGIC LOG FORM

ATTACHMENT A
EXAMPLE WELL COMPLETION FORM

BROWN AND CALDWELL

BOREHOLE/WELL LOG

LOCATION OF BORING

N
W E
S

SCALE:

CLIENT

LOCATION

PROJECT NAME

JOB NUMBER

BORING NUMBER

SHEET OF

DRILLING CONTRACTOR

DRILLING START TIME / DRILLING FINISH TIME

RIG TYPE (CIRCLE ONE) OTHER _____
HSA DP MUD ARCH NONE

DRILLING START DATE / DRILLING FINISH DATE

DRILLING METHOD, FLUID USED

SOIL SAMPLING METHOD (CIRCLE ONE) OTHER _____
SPLIT SPOON CC ACETATE NONE

WELL CONSTRUCTION START TIME / WELL CONSTRUCTION FINISH TIME

MONITORING INSTRUMENT (CIRCLE ONE) OTHER _____
PID FID LEL RAD NONE

WELL CONSTRUCTION START DATE / WELL CONSTRUCTION FINISH DATE

SURFACE CONDITIONS (CIRCLE ONE) OTHER _____
ASPHALT CONCRETE DIRT DRY WET

WELL CONSTRUCTION		SOIL SAMPLING									
ANNULUS	CASING	SAMPLER USED	BLOWS/6" INTERVAL	INTERVAL SAMPLED	RECOVERY	ANALYTICAL SAMPLE (A,B,C)	WATER LEVEL (INTIAL/STATIC)	DEPTH (FEET)	INSTRUMENT READING (ppm)	USCS GROUP SYMBOL	FINE GRAINED AND ORGANIC SOIL DESCRIPTION: GROUP NAME, MUNSELL COLOR, COLOR, SIZE DISTRIBUTION, PLASTICITY, DRY STRENGTH, DILATANCY, TOUGHNESS, MOISTURE, ODOR, STRUCTURE, CONSISTENCY, RELATIVE PERMEABILITY, LOCAL GEOLOGIC NAME, CONTACT DESCRIPTION COARSE GRAINED SOIL DESCRIPTION: GROUP NAME, MUNSELL COLOR, COLOR, GRADATION (g/s/f), SIZE DISTRIBUTION, PLASTICITY, SHAPE, ANGULARITY, MOISTURE, ODOR, STRUCTURE, CEMENTATION, RELATIVE PERMEABILITY, LOCAL GEOLOGIC NAME, MINERALOGY, CONTACT DESCRIPTION
											SAMPLE TYPE: INTERVAL: DESCRIPTION
								0			
								5			
								0			
								5			
								0			

ANALYTICAL SAMPLE ID (SEE ABOVE FOR LOCATION)

A _____ D _____
B _____ E _____
C F

STANDARD OPERATING PROCEDURE (SOP)
NO. 2

MONITORING WELL SAMPLING

Brown and Caldwell
Standard Operating Procedure

Groundwater Sample Collection

Revision 1.1
Revision Date: October 9, 2001

Prepared/Revised by:	**Wendy Linck**	October 9, 2001
	Name	Date
Senior QA Review:	Jay Lucas	10/26/01
	Name	Date
Regional Quality Officer:	Daniel Van Winkle	October 26, 2001
	Name	Date

GROUNDWATER SAMPLE COLLECTION

TABLE OF CONTENTS

TABLE OF CONTENTS i
1.0 OBJECTIVES 1
2.0 SCOPE AND APPLICABILITY 1
3.0 REQUIRED MATERIALS 1
4.0 RESPONSIBILITIES 2
5.0 METHODS 3
5.1 General Considerations 3
5.1.1 Equipment Decontamination 4
5.1.2 Dedicated and Disposable Equipment 4
5.1.3 Sequence of Sampling 5
5.1.4 Field QA/QC Samples 5
5.2 PURGING AND SAMPLING PROCEDURES 6
5.2.1 Planning 6
5.2.2 Preparatory Office Activities 9
5.2.3 Preparatory Field Activities 10
5.2.4 Well Purging 13
5.2.5 Groundwater Sampling 16
5.2.6 Equipment Instructions 17
5.2.7 Field Measurements 37
5.3 FIELD RECORDS 38
5.4 SAMPLE SHIPMENT 39
5.5 SPECIAL REGULATORY REQUIREMENTS 39
6.0 REFERENCES 39
7.0 ATTACHMENTS 42

1.0 OBJECTIVES

The primary objective of this standard operating procedure (SOP) is to establish a uniform method for the collection of representative groundwater samples from monitoring wells, and to reduce the potential variability associated with purging and sampling.

2.0 SCOPE AND APPLICABILITY

This SOP will be used to support groundwater monitoring programs and conducting the field groundwater sampling activities. Groundwater sampling involves two primary operations, purging stagnant water from a well followed by the collection of a sample from the same well. Groundwater sampling variables can be significantly controlled through the appropriate selection and use of purging and sampling equipment, and through the use of procedures that are described in this SOP.

3.0 REQUIRED MATERIALS

Materials required for conducting groundwater sampling are variable depending upon the method chosen to conduct the sampling. Therefore the listing of materials will be separated into two parts in this SOP. This section will present materials that are general in applicability – things that should be included regardless of purge or sampling method. In Section 5, where specific methods and approaches are discussed, additional materials will be listed. General materials that should be considered regardless of method are as follows:

- Health and safety records
- Personal protection equipment (as required by the Site Safety Plan)
- Health and safety monitoring equipment (e.g., PID)

- Well Completion Forms and Data from previous sampling efforts (if available)
- Water level indicator
- Decontamination supplies (5 gallon buckets, decontamination fluids, squirt bottles)
- Water quality monitoring equipment
- Purge pumps and control boxes
- Generator
- Twine
- Permanent marking pens
- Notebook
- Calculator
- Measuring tape
- IDW containers and labels
- Garbage bags
- Drinking water
- Phone, Camera
- Shipping labels and Chain of Custody records
- Coolers
- Ice
- Filters (0.45 μm), if appropriate
- Tubing

Checklists are included in the Readiness Review SOP for help in gathering and monitoring the equipment necessary to conduct groundwater purging and sampling

4.0 RESPONSIBILITIES

The Project Manager, or designee, will have the responsibility to oversee and ensure that groundwater purging and sampling procedures are implemented in accordance this SOP and any project- or site-specific planning documents.

The field personnel will be responsible for the understanding and implementation of this SOP during groundwater sampling activities, as well as, obtaining the appropriate field logbooks, forms and records necessary to complete the field activities.

5.0 METHODS

5.1 General Considerations

Groundwater sampling involves two primary operations. These include the purging of stagnant water from the well followed by the collection of a sample. Groundwater sampling variables can be significantly controlled through the appropriate selection and use of purging and sampling equipment, and through the use of procedures that are described below.

Good communication is essential to the ultimate success of a groundwater sampling project. This includes communication within the project team, as well as communication with the client and analytical laboratory, when establishing project objectives.

Good communication with the project team, laboratory, client, and, if appropriate, regulatory agencies, includes complete project specific planning documents such as field sampling plans, quality assurance plans, and scope of work documents for subcontracted laboratories. Plans should include detailed information with respect to site-specific requirements, with reference to SOPs wherever possible, and risk criteria that will be used to assess the data. The quality assurance plan and laboratory scope of work (of which the quality assurance plan can be part) should contain detailed information regarding what is expected from the laboratory regarding the methods to be used, quality assurance measures and calibrating corrective measures, and deliverables (especially electronic deliverable formats). A

detailed quality assurance plan is even more important in light of EPA's Performances Based Measurement Standard (PBMS) initiative.

In addition to good communication, the project plans should consider equipment decontamination, sampling equipment, sampling sequence, and field quality assurance/quality control (QA/QC) samples. These are described in the following sections.

5.1.1 Equipment Decontamination

Equipment that will be in contact with the sample must be decontaminated prior to each use. This is necessary to minimize inadvertent contamination of the sample. Specific methods for equipment cleaning are dependent upon a number of factors including the sample media, analytical parameters, the purpose of the investigation, the equipment to be cleaned, and the specific regulatory guidelines that may apply.

Equipment decontamination procedures are described in the Equipment Decontamination SOP. Any site specific decontamination procedures can be specified in the field sampling plan for each project.

5.1.2 Dedicated and Disposable Equipment

Use of dedicated and new, disposable purging and sampling equipment are preferable to decontamination of reusable sampling equipment. Dedicated equipment, and use of new, disposable equipment, can virtually eliminate cross-contamination between samples caused by incomplete decontamination. Dedicated equipment can also increase sampling efficiency through the elimination of the need to decontaminate equipment for successive sampling. Furthermore, dedicated equipment can also help to reduce the physical handling of the equipment that can cause sample contamination through contact with potentially contaminated

surfaces. New, disposable equipment may need to be decontaminated before use. Review project-specific planning documents regarding decontamination of disposable equipment.

5.1.3 Sequence of Sampling

The groundwater sampling operation should always be conducted in a sequence that proceeds from wells containing the lowest concentrations to wells containing the highest concentrations. Sampling in this order will further minimize the likelihood of sample cross-contamination that can be caused through improper handling or equipment cleaning. This type of sampling sequence should be used even for programs in which equipment is dedicated to minimize the cross-contamination that could result from exposure to contaminated garments or other equipment. If water quality is not known, the wells upgradient of a suspected source area should be sampled first, followed by the wells furthest away and cross-gradient or downgradient.

Sampling sequence also applies to the order that different analytes are collected. Typically these analytes that are volatile are collected first, followed by those that are sensitive to oxidation.

5.1.4 Field QA/QC Samples

Additional samples should be collected for the specific purpose of documenting the Quality Assurance/Quality Control (QA/QC) of the field sampling procedures. Field QA/QC samples provide technically and legally defensible data regarding the reproducibility and overall quality of the groundwater sample. Descriptions of the type and frequency of QA/QC sampling should be specified in the project-specific planning documents. Field QA/QC samples include field blanks, equipment blanks, trip blanks, and blind duplicates. These samples are collected in addition to the

laboratory QA/QC samples which may include method blanks, matrix spikes and matrix spike duplicate samples.

5.2 PURGING AND SAMPLING PROCEDURES

This section provides a description of the procedures to be used for groundwater sampling. These procedures include planning, preparatory office activities, preparatory field activities, well purging, well sampling, and post sampling activities. These activities are listed on Attachment A and are described in detail in the following sections. Many of these steps have record keeping components which are discussed in detail in Section 5.4.

5.2.1 Planning

The planning phase should include the selection of specific field methods, including the well purging strategy and planning for the proper disposal of the purge water. The sampling program should be discussed in project-specific planning documents.

Good communication with the analytical laboratory is essential to the success of a groundwater sampling project. The analytical requirements must be well defined and clearly communicated, prior to conducting the field work. Written communication is encouraged, in particular to document requirements for specific analytical methods, low detection limits, and other special needs. Written communication should include a detailed scope of work that includes the quality assurance plan for the project. These plans should specifically identify detection limits, with particular emphasis placed on how these limits relate to regulatory criteria or risk based criteria that have been developed for the project.

Purging and Sampling Equipment Selection

Some of the factors that should be considered in the selection of purging and sampling devices include:

- Well yield
- Depth to water
- Well diameter and depth
- Required material of construction
- Analytical parameters
- Regulatory requirements
- Cost

Attachment B presents a summary of purging equipment. Attachment C summarizes well sampling equipment. Attachment D presents a matrix indicating suitability of sampling and purging equipment for specific applications.

Purging Strategies

The strategy that will be employed for well purging should be determined prior to sampling and presented in project-specific planning documents. Several different strategies are commonly used in order to assess the completeness of well purging. The most common purging strategies are listed below.

- Purging is continued until stabilization of certain indicator parameters is observed in successive measurements over a specified time or volume. The most commonly used indicator parameters include pH, specific conductivity, turbidity, temperature, oxidation/reduction potential (ORP), and dissolved oxygen (DO).
- Purging 3 to 5 well volumes of water from the well.

- Purging low yield wells until the water level reaches the top of the well screen, and then allowing the well to partially recover. After partial recovery, the well is re-purged to the top of the well screen and allowing the well to partially recover a second time. It is recognized, however, that it may not be possible to avoid dewatering the well screen in many shallow wells.
- Very low yield wells are purged until dryness and allowing the well to partially recover (often 12 to 24 hours).
- Low or no-flow purging strategies. Generally, well is pumped at rates (<0.5 gallons per minute) that do not induce drawdown and ostensibly mirror flow rates in the aquifer. These are gaining acceptance in many areas because such techniques reduce generated wastes. Often regulatory agencies will require a comparison study before acceptance but these can be valuable when long term monitoring programs will be implemented.

Implementation of these strategies is discussed in more detail in Section 5.2.

Purge Water Disposal

The methods and responsibility for collection, containerization, treatment and disposal of purge water should be determined prior to initiation of any sampling project. Much of how to handle purge water is discussed in the SOP for Investigation Derived Waste. However, additional considerations for groundwater purging and sampling are included below.

Collection and contamination is often accomplished through use of 55 gallon drums, mobile storage tanks, or through use of vacuum trucks which can directly transport off site to a treatment facility or to larger storage tanks on site. If specifically allowed by the responsible agency, purge water may be reapplied to the ground surface. Treatment of purge water may be accomplished on site at facilities that

have wastewater treatment plants, or by using a mobile treatment unit. Responsibility for off-site disposal of containerized purge water must be determined prior to conducting the work.

5.2.2 Preparatory Office Activities

Equipment and containers should be organized in the office prior to embarking on a field sampling project to the extent practicable. The time spent in the field should be spent on sample collection, making field measurements and recording data.

Prepare Sampling and Purging Equipment

The purging and sample collection equipment and all required hardware should be obtained, organized and decontaminated prior to the initiation of the field sampling program. To accommodate waste generated during decontamination, these activities may be completed at the site prior to sampling.

Sample Containers and Preservatives

The appropriate sample containers and associated preservatives must be obtained. The containers and preservatives are normally, but not always, supplied by the laboratory that will be responsible for the analyses. Sample containers should be organized and inventoried several days prior to initiation of the sampling program in order to provide sufficient time to rectify any problems, should they occur. Whenever possible, pre-printed sample labels should be created prior to mobilization, if possible.

Initiation of Field Data Records

Field data sheets should be initiated prior to the start of sampling. Examples of initial data to be recorded include site and sampling location identification, well depth and construction, and purging and sampling collection methods. Additional discussion regarding field records is presented in Section 5.4. Example field data sheets are provided in Attachments E and F. These sheets can be combined in a bound field notebook as well. See SOPs on Sampling Handling, Sample Preservation, and Field Notes and Documentation regarding the requirements for storage/preservation and use of field records, respectively.

5.2.3 Preparatory Field Activities

The following procedures should be conducted in the field prior to well purging and sampling.

Well Maintenance Check

A well maintenance check should be performed that includes a visual inspection of the condition of the protective casing and surface seal. In addition, the well should be inspected for other signs of damage or unauthorized entry. Any problems should be documented.

It is recommended that the bottom of the well not be sounded each time the well is sampled. Routine sounding of the well can increase the risk of inadvertent well contamination because it is difficult to adequately decontaminate the tapes used for this purpose. Well depths obtained from well completion records are generally adequate for the purpose of the determination of well volume. Generally, the only reason to sound well depth is if a need to verify the depth arises, or if you suspect that sediment/soil has collected in the bottom of the well.

Preparation of Well Area

A suitable work area should be established around the perimeter of the well. Sampling equipment should be placed on a clean surface such that it will not become inadvertently contaminated. This work area can be prepared by placing new polyethylene (PE) sheeting on the ground around the well, taking care not to step on it. Alternatives also include the placement of a clean PE-lined trash can, a clean PE or aluminum covered table, or similar, adjacent to the well. Remember – a clean work area leaves a much more favorable impression than a dirty work area.

Water Level Measurements

The depth to water should measured prior to initiation of all sampling activities. The water level measurements should be made following the SOP for Groundwater Level and NAPL Measurements in Monitoring Wells.

Non-Aqueous Phase Liquid (NAPL) Determination

If their presence is suspected, each well should be evaluated for the presence of light or dense non-aqueous phase liquids (LNAPLs or DNAPLs) prior to well purging. The NAPL measurements should be made following the SOP for Groundwater Level and NAPL Measurements in Monitoring Wells.

Calculation of Well Purge Volume

The volume of water standing in the well should be calculated through the application of the depth to water data, the known well depth, and the well diameter using the constants presented below. Well depth information obtained from the well

completion records are generally sufficiently precise for the purpose of well volume calculations that would be used for subsequent purging determinations.

The following conversions allow quick calculation of well casing volumes:

Well Casing Diameter (inches)	Gallons per foot of water
1.0	0.041
2.0	0.163
3.0	0.367
4.0	0.653
6.0	1.469

Alternatively, the well casing volume may be calculated using the formula $V = CF*d^2h$, where

V = volume of water (gallons)

d = diameter of well (inches)

h = height of water column (feet)

CF = conversion factor (0.0408) that includes conversion of cubic feet to gallons, inches to feet, and diameter to radius.

In some states the volume of the entire well (not just the well casing, but entire borehole) is required when determining well volume. Check local state requirements to determine which method of well volume calculation applies to your site. This volume can be calculated in a manner similar to above with the following formula:

$$V = 0.0408\ d^2h + 0.0408\ (D^2-d^2)\ h\varnothing$$

where

V = volume of water in well (gallons)

D = diameter of borehole (inches)

d = diameter of well casing (inches)

h = height of water column (feet)

$\varnothing$ = porosity of sand pack (often approximated at 0.30)

Which of the above methods should be used should be discussed or defined in the project-specific planning documents.

5.2.4 Well Purging

Monitoring wells must be purged prior to the collection of aqueous phase samples. Specific instructions for the use of purging equipment are presented in Section 4.6.

The placement of a device (in most cases a pump) that will be used for well purging is critical in order to ensure a complete exchange of the entire water column. The intake of a device used for purging should be placed as high in the water column as is possible under pumping conditions. Optimum placement is to have the pump at the top of the water column. This is done so that purging will draw water from the formation into the screened area of the well, and up through the casing, so that the entire static water column can be removed.

If the monitoring well is a slow recharging well, then the pump should be placed near the surface and slowly lowered at a rate similar to groundwater withdrawal. As an alternative approach the pump could be set at no more than three to five feet below the water surface. If the recovery rate of the well is faster than the pump rate and no observable drawdown occurs, the pump can be raised until the intake is within one foot of the top of the water column for the duration of purging. If the pump rate exceeds the well recovery rate, the pump will have to be lowered as needed based upon the amount of drawdown.

Upon completion of purging, the device should continue to be operated as it is slowly withdrawn from the well to purge any water that remains above the pump intake. One should be very careful not to keep the pump on too long after removal, because

a pump can easily burn up without groundwater to cool it. It is recommended that pumps be fitted with backflow prevention valves so that purged water in the discharge tubing does not flow back into the well. Under no circumstances should the pump intake simply be placed at the bottom of the well without first making an effort to evaluate the pumping characteristics of the particular well.

Standard Purging Approach

Initially, groundwater withdrawal should occur no more than three to five feet below the water surface. If the recovery rate of the well is faster than the pump rate and no observable drawdown occurs, the pump should be raised until the intake is within one foot of the top of the water column for the duration of purging. If the pump rate exceeds the well recovery rate, the pump will have to be lowered as needed based upon the amount of drawdown.

An adequate purge is normally achieved when three to five times the volume of standing water in the well has been removed. After three well volumes have been removed, if the chemical parameters have not stabilized according to the criteria given below, additional well volumes may be removed. If the parameters have not stabilized within five volumes, it is at the discretion of the project manager whether or not to collect a sample or to continue purging.

Considering groundwater chemistry, an adequate purge is achieved when the pH, specific conductance, and temperature of the groundwater have stabilized and the turbidity has either stabilized or is below 10 Nephelometric Turbidity Units (NTUs). In very silty formations, the turbidity stabilization criteria given above may be impossible to reach and should be disregarded. Other parameters such as salinity, dissolved oxygen, and oxidation reduction potential also may be important criteria for stabilization, especially under low flow purging. Stabilization occurs when parameter measurements are within 10 percent between two readings spaced

approximately one well volume apart, or under low flow purging, between two readings determined in project planning documents. A water meter quality fitted with flow through cell, which allows continuous monitoring of the above parameters is recommended for these measurements.

Attempts should be made to avoid purging wells to dryness, as previously described. However, even with slow purge rates, a well may be purged dry. In those cases, this constitutes an adequate purge and the well can be sampled when recovery is sufficient (enough volume to fill the sample containers). Recovery criteria are often cited as 80 percent of the original well column height.

Low Stress or Low Flow Groundwater Purging

Sometimes it is desirable to collect representative samples while exerting minimum stress on the water-bearing formation. Typically this is accomplished by limiting the flow rate during purging to the range of 100 to 500 ml/min (0.025 to 0.13 gallons/min). For this procedure, the goal is to induce a steady flow rate while minimizing the drawdown.

It is important to insert the sampling equipment carefully, so as to prevent the re-suspension of silt and clay particles in the well. In order to minimize turbidity, it is preferable to use dedicated equipment, or to allow sufficient time after the installation of non-dedicated equipment to allow soil particles to re-settle before purging and sampling. Sufficient time for settling should be verified by turbidity measurements.

Initially, the purge flow rate should start at approximately 200 ml/min (0.053 gallons/min), and water level should be frequently monitored. Flow rate should be adjusted so that drawdown will not exceed 0.3 ft, or approximately 2 percent of the saturated thickness of low permeability formations, whichever is greater.

Indicator parameters which should be monitored for low stress groundwater purging include turbidity, specific conductance, temperature, pH, ORP, and DO. In-line analyzers and continuous readout displays are recommended, so that the sample is not exposed to air prior to measurement. The well is considered stabilized when three consecutive readings vary no more than 10% or are below 10 NTUs for turbidity, 3% for specific conductance, 0.1 SU for pH, 10 mV for ORP, and 10% for DO. Measurement of the indicator parameters should continue every three to five minutes until these measurements indicate stability in water quality. If these parameters have not stabilized after about an hour, the well should be purged until a minimum of three well volumes have been removed.

5.2.5 Groundwater Sampling

It is important that wells be sampled as soon as possible after purging. If adequate volume is available, the well should be sampled immediately as long as the well has recovered to 80 percent of the original water column height. If not, sampling should occur as soon as the well has recovered sufficiently to provide adequate volume. Specific instructions for the use of sampling equipment are presented in Section 5.2.6.

Standard Sampling Approach

As with purging equipment, there are a number of considerations in the selection of sample collection equipment. Furthermore, it is common to use a different device for sample collection than for purging. An example would be to purge with the use of submersible pump and to collect the sample with the use of a disposable bailer. A summary of well sampling equipment is provided in Attachment B.

As discussed previously, consideration should be given to the order in which sample containers are to be filled for various parameter groups. The order should be determined on the basis of parameter sensitivity to volatilization, pH change, or oxidation, and the priority for analytical data in cases where the water volume in the well is less than what is required for analysis. In general, volatile organic compounds are the most sensitive constituents to volatilization so the sample for these parameters should be containerized immediately. Likewise, pH change occurs rapidly in samples that are in contact with air, so pH measurements and the containerization of pH sensitive parameters, such as anions (e.g., nitrate, sulfate), or metals, (e.g., ferrous iron or Fe^{2+}), should also be implemented expeditiously.

Low Stress Groundwater Sampling

Sometimes it is desirable to collect representative samples while exerting minimum stress on the water-bearing formation. Typically this is accomplished by limiting the flow rate during sampling to the range of 100 to 250 ml/min (0.025 to 0.065 gallons/min). Sampling flow rate should not exceed the purge flow rate for which water quality indicator parameters stabilized. Sampling equipment must be the same equipment that was used for purging, and should not be moved between purging and sampling activities.

5.2.6 Equipment Instructions

This section provides specific instructions for the installation and use of various devices for both well purging and groundwater sample collection, and includes the following equipment:

- Bailer
- Bladder pump
- Small diameter (2-inch) electric submersible pump

- Large diameter (4-inch) electric submersible pump
- Compressed gas-driven piston pump
- Gasoline powered centrifugal pump
- Peristaltic suction pump
- Inertial lift pump.

It is recognized that a combination of the procedures may be employed. An example would be the use of a small diameter electric submersible pump for purging and a bailer for sample collection. The specific methods to be used for purging and sampling a well should be outlined in the project-specific planning documents.

Bailer

A bailer is one of the simplest groundwater sampling devices. The same bailer can be used for both purging and sample collection. Bailers consist of a rigid tube equipped with a bottom and/or top check valve that is lowered into the well on a flexible cord.

Required Equipment:

- Bailer of appropriate size and material
- New bailer cord of appropriate material
- New disposable gloves of appropriate material
- Clean trash can and supply of trash can liners, or new plastic sheeting
- Five gallon pail, graduated in minimum one gallon increments
- Bottom emptying device (optional)
- Water quality monitoring equipment

Purging Instructions:

- Determine the volume of water to be purged, as described previously.
- Place new polyethylene (PE) sheet on suitable surface adjacent to well, taking care not to step on the PE sheet.
- Don a new pair of gloves. Decontaminate equipment as outlined in decontamination SOP
- Attach a cord to the bailer.
- Lower the bailer into the well until it is completely submerged. The bailer should be lowered slowly so that it does not cause a splash that could aerate the water column.
- Pull the bailer out of the well while placing the cord on the PE sheet or in a PE-lined bucket. An alternate method is to wind the bailer cord between the hands. Care should be taken to prevent the bailer or the cord from contacting any surface other than the interior of the well or the plastic liner.
- Empty the bailer into the pail.
- Repeat the operation until the necessary volume of water has been purged from the well.
- Monitor indicator parameters as discussed previously.

Sampling Instructions:

- Prepare the well area and lower the bailer into the well following the method described previously for purging.
- Allow the bailer to fill slowly and then gently retrieve the bailer from the well while avoiding contact with the sides of the well. Care should be taken to prevent the bailer or the line from contacting any surface other than the interior of the well or the PE sheet or PE-lined bucket.
- Fill the sample containers slowly. The use of a bottom emptying device is preferred in that sample aeration and sample volume loss are minimized.

- Bottles should be filled in order of sensitivity to volatilization and oxidation as discussed in Section 5.1.3 or as outlined in project-specific planning documents. Care should be taken to ensure that no head space remains in the volatile organic vials. Certain other parameters may also require minimizing headspace (e.g., reduced or ferrous iron).
- Filtered samples can be obtained by filling a non-preserved sample receptacle, and then transferring the liquid through an in-line filter (typically a 0.45 μm disposable filter) into a preserved sample receptacle using a peristaltic pump. Other gravity feed filters also are available.

Bladder Pump

A bladder pump is one of the easiest devices to operate for the purpose of purging and sample collection. The bladder pump is often dedicated to the well and can be used in conjunction with an inflatable packer in order to minimize the purge volume necessary to accomplish effective purging.

Required Equipment:

- Bladder pump
- Tubing of appropriate type and length
- Bladder pump controller
- Compressed air source
- New disposable gloves of appropriate material
- New plastic sheeting
- Five gallon pail, graduated in minimum one gallon increments
- Water quality monitoring equipment (preferably a flow through cell).
-

Installation Instructions (initial installation or non-dedicated use):

- Place new polyethylene (PE) sheet on suitable surface adjacent to well, taking care not to step on the PE sheet.
- Don a new pair of gloves.
- Assemble the pump and tubing and lower into the well being careful not to contact any surface other than the interior of the well or the plastic sheeting.
- Decontaminate equipment and pump (if not dedicated) as outlined in decontamination SOP
- Install the air inlet and water discharge fittings on top of the well (for a dedicated installation).

Purging Instructions:

- Refuel the gasoline-powered compressor, if used, at a location that is remote from the well, being very careful not to spill any fuel on equipment or clothing that will be used at the well site.
- Place the gasoline-powered compressor as far from the well as possible in a down-wind direction to eliminate potential exhaust impact to sampling.
- Connect the compressed air source and pump controller to the pump as per manufacturer's instructions.
- Don a new pair of gloves after handling the gasoline-powered compressor.
- Determine the volume of water to be purged, as described previously.
- Start the pump by opening the regulator on the controller, which allows compressed air to flow into the system.
- The controller should be adjusted to maximize the flow rate while minimizing the rapid "jolting" of the tubing as water is drawn into pump.
- Direct the pump discharge to the five gallon pail and determine the pumping rate.
- Continue pumping until the necessary volume of water has been purged from the well.

- Monitor indicator parameters as discussed previously.

Sampling Instructions:

- Allow the well to recharge after completion of purging, if necessary.
- Resume pumping after adjusting the regulator to the minimum pressure that will still allow water to be pumped to the surface.
- Collect the samples by pumping directly into each of the required containers.
- Bottles should be filled in order of sensitivity to volatilization and oxidation as discussed in Section 5.1.3 or as outlined in project-specific planning documents. Care should be taken to ensure that no head space remains in the volatile organic vials. Certain other parameters may also require minimizing headspace (e.g., reduced or ferrous iron).
- Filtered samples can easily be obtained by installing an in-line, 0.45 μm disposable cartridge filter directly onto the pump discharge.

Small Diameter (2") Electric Submersible Pump

A small-diameter electric submersible pump (Grundfos Redi-Flo2 or equivalent) can be operated with a wide variety of pumping rates such that it is very versatile for both well purging and sample collection. This type of pump can be used in either a dedicated or non-dedicated mode. Collecting groundwater samples from these pumps is only appropriate if approved project-specific planning documents specifically include this technique for collecting samples.

Required Equipment:

- Small diameter electric submersible pump
- Pump shroud (when used in a six-inch or larger well to minimize turbulence, to keep motor cool)
- Teflon® jacketed power cable on a plastic reel

- Tubing of appropriate type and length
- Check valve (optional)
- Electric pump controller with appropriate power plug
- 230 volt, single phase, electric power source, >10 amps
- Tool kit including basic tools, tubing cutters, extra tubing connector bracket, electrical connectors, wire ties, etc.
- Ground fault interrupter (GFI)
- New disposable gloves of appropriate material
- New PE sheeting
- Five gallon pail, graduated in minimum one gallon increments
- Water quality monitoring equipment (preferably a flow through cell).

Installation Instructions:

- Place new polyethylene (PE) sheet on suitable surface adjacent to well, taking care not to step on the PE sheet.
- Don a new pair of gloves.
- Assemble the pump, tubing, optional check valve, and electric power cables.
- Decontaminate equipment and pump (if not dedicated) as outlined in decontamination SOP.
- Prime pump with deionized water by inverting the pump and removing fill cap.
- Lower pump slowly into the well, being careful not to contact any surface other than the interior of the well or the plastic sheeting. When lowering the pump be particularly sensitive to areas that suggest drag or problems in the well where the pump could get stuck. If a problem exists do not continue, but discuss ways to investigate with PM or senior technical personnel.
- Place the pump intake as discussed in Section 5.4.2. Monitor the pump discharge and well hydraulics as discussed previously.

Purging Instructions:

- Refuel the electric generator at a location that is remote from the well, being very careful not to spill any fuel on equipment or clothing that will be used at the well site.
- Place the gasoline-powered compressor as far from the well as possible in a down-wind direction to eliminate potential exhaust impact to sampling.
- Don a new pair of gloves after handling the generator.
- Connect electric power, GFI, and pump controller to the pump.
- Determine the volume of water to be purged, as described previoulsy.
- Start the pump.
- Direct the pump discharge to the five gallon pail and determine the pumping rate.
- Continue pumping until the necessary volume of water has been purged from the well.
- If the pump intake has been placed deeply down into the water column for some reason, slowly withdraw the pump upward through the water column while it is still running to purge all water standing above the pump unless the pump will be used for sample collection.
- Shut off the pump rapidly whenever the pump stops pumping water.
- Monitor indicator parameters as discussed previously.

Sampling Instructions:

- Allow the well to recharge after completion of purging, if necessary.
- Resume pumping and adjust the pumping rate to the slowest possible rate.
- Collect the samples by pumping directly into each of the required containers.
- Bottles should be filled in order of sensitivity to volatilization and oxidation as discussed in Section 5.1.3 or as outlined in project-specific planning documents. Care should be taken to ensure that no head space remains in

the volatile organic vials. Certain other parameters may also require minimizing headspace (e.g., reduced or ferrous iron).

- Filtered samples can easily be obtained by installing an in-line, 0.45 μm disposable cartridge filter directly onto the pump discharge.

Large Diameter (4") Electric Submersible Pump

A large-diameter electric submersible pump is most frequently used for purging large-diameter wells that cannot be efficiently purged using other methods. Large-diameter pumps can also be used for sample collection for parameters that are not particularly pH or pressure sensitive. This type of pump is usually not dedicated. Collecting groundwater samples from these pumps is only appropriate if approved project-specific planning documents specifically include this technique for collecting samples.

Required Equipment:

- Large-diameter, stainless steel electric submersible pump
- [Optional] Check valve, install internally within the pump or externally between the pump and the tubing
- Electric power cable on a plastic reel (Teflon® jacketed cable is preferred)
- Stainless steel or polypropylene support line
- 1/2" to 1" PE Tubing of appropriate length
- Flow control valve
- 120 or 240 volt electric power source (as appropriate)
- Ground fault interrupter (GFI)
- Tool kit including assorted basic tools, tubing cutters, wire cutters, hose clamps, waterproof connectors, wire ties, hose fittings, assorted pipe nipples and adapters, etc.
- New disposable gloves of appropriate material
- New plastic sheeting

- Five gallon pail, graduated in minimum one gallon increments or flow meter
- Bleeder tee (if the submersible pump is used for sample collection). This consists of a tee with a large diameter, valved pump discharge port and a Water quality monitoring equipment.
- small diameter, valved port equipped with a sample weephole.
- Water quality monitoring equipment (preferably a flow through cell).

Installation Instructions:

- Place new polyethylene (PE) sheet on suitable surface adjacent to well, taking care not to step on the PE sheet.
- Don a new pair of gloves.
- Decontaminate equipment and pump (if not dedicated) as outlined in decontamination SOP.
- Assemble the pump, tubing, flow control valve, electric power cables and support line and lower into well being careful not to contact any surface other than the interior of the well or the PE sheeting.
- The tubing, electrical power cable and support line should be fastened together at 15 to 20 foot intervals with the use of nylon wire ties (not with electrical tape) taking care to leave adequate slack in the electric power cable.
- The use of a check valve is recommended to prevent rapid backflow of water from the tubing after the pump is shut off. It is recognized, however, that the use of a check valve makes it more difficult to pull the pump from the well due to the added weight of the water in the tubing and can increase the chance of "sand locking" occurring in the pump.
- Lower pump slowly into the well, being careful not to contact any surface other than the interior of the well or the plastic sheeting. When lowering the pump be particularly sensitive to areas that suggest drag or problems in the well where the pump could get stuck. If a problem exists do not

continue, but discuss ways to investigate with PM or senior technical personnel.

- Place the pump intake as discussed in Section 5.4.2. Monitor the pump discharge and well hydraulics as discussed previously.

Purging Instructions:

- Refuel the electric generator at a location that is remote from the well, being very careful not to spill any fuel on equipment or clothing that will be used at the well site.
- Place the gasoline-powered compressor as far from the well as possible in a down-wind direction to eliminate potential exhaust impact to sampling.
- Don a new pair of gloves after handling the generator.
- Connect electric power and GFI to the pump.
- Determine the volume of water to be purged, as described previously.
- Start the pump.
- Determine the pumping rate either by directing the pump discharge to the five gallon pail or with the use of a flow meter.
- Adjust pumping rate to match the yield of the well.
- Continue pumping until the necessary volume of water has been purged from the well.
- If the pump intake has been placed deeply down into the water column for some reason, slowly withdraw the pump upward through the water column while it is still running to purge all water standing above the pump.
- Shut off the pump immediately whenever the pump stops pumping water.
- Dispose of the tubing and support line after use.
- Monitor indicator parameters as discussed previously.

Sampling Instructions:

- Allow the well to recharge after completion of purging, if necessary.
- Resume pumping and adjust the bleeder tee valves so that a slow, steady, non-aerated flow emanates from the bleeder tee weephole (approximately 100 ml/min).
- Collect the samples directly from the weephole into each of the required containers.
- Bottles should be filled in order of sensitivity to volatilization and oxidation as discussed in Section 5.1.3 or as outlined in project-specific planning documents. Care should be taken to ensure that no head space remains in the volatile organic vials. Certain other parameters may also require minimizing headspace (e.g., reduced or ferrous iron).
- Filtered samples can easily be obtained by installing an in-line, 0.45 μm disposable cartridge filter directly onto the pump discharge

Compressed Gas-Driven Piston Pump

A compressed gas-driven piston pump can be used for purging and sample collection in two-inch diameter wells at great depths. This type of pump is not typically dedicated. Collecting groundwater samples from these pumps is only appropriate if approved project-specific planning documents specifically include this technique for collecting samples

Required Equipment:

- Small diameter air driven pump
- Tubing of appropriate type and length
- Pump controller
- Compressed air source
- New disposable gloves of appropriate material
- New plastic sheeting

- Five gallon pail, graduated in minimum one gallon increments
- Water quality monitoring equipment (preferably a flow through cell).

Installation Instructions:

- Place new polyethylene (PE) sheet on suitable surface adjacent to well, taking care not to step on the PE sheet.
- Don a new pair of gloves.
- Decontaminate equipment and pump (if not dedicated) as outlined in decontamination SOP.
- Assemble the pump and tubing and lower into the well, being careful not to contact any surface other than the interior of the well or the plastic sheeting.
- Lower pump slowly into the well, being careful not to contact any surface other than the interior of the well or the plastic sheeting. When lowering the pump be particularly sensitive to areas that suggest drag or problems in the well where the pump could get stuck. If a problem exists do not continue, but discuss ways to investigate with PM or senior technical personnel.
- Place the pump intake as discussed in Section 5.4.2. Monitor the pump discharge and well hydraulics as discussed previously.

Purging Instructions:

- Connect the air source and pump controller to the pump.
- Determine the volume of water to be purged, as described above.
- Start the pump.
- Direct the pump discharge to the five gallon pail and determine the pumping rate.
- Continue pumping until the necessary volume of water has been purged from the well.

- If the pump intake has been placed deeply down into the water column for some reason, slowly withdraw the pump upward through the water column while it is still running to purge all water standing above the pump.
- Shut off the pump immediately whenever pump stops pumping water.
- Monitor indicator parameters as discussed previously.

Sampling Instructions:

- Allow the well to recharge after completion of purging, if necessary.
- Resume pumping and adjust to a slow pumping rate (approximately 100 ml/min).
- Collect the samples by pumping directly into each of the required containers.
- Bottles should be filled in order of sensitivity to volatilization and oxidation as discussed in Section 5.1.3 or as outlined in project-specific planning documents. Care should be taken to ensure that no head space remains in the volatile organic vials. Certain other parameters may also require minimizing headspace (e.g., reduced or ferrous iron).
- Filtered samples can easily be obtained by installing an in-line, 0.45 μm disposable cartridge filter directly onto the pump discharge.

Gasoline Powered Centrifugal Suction Pump

A two-cycle gasoline-engine-powered centrifugal pump provides an effective way to rapidly purge any well in which the depth to the pumped water level is less than about 20 to 25 feet. This type of pump is normally used with dedicated tubing. Groundwater sampling cannot be conducted using these pumps.

Required Equipment:

- Two-cycle gasoline-engine-powered centrifugal pump
- 1/2" to 3/4" PE tubing of appropriate length

- Flow control valve
- Check (foot) valve
- Supply of distilled or deionized water
- New disposable gloves of appropriate material
- New PE sheeting
- Five gallon pail, graduated in minimum one gallon increments
- Water quality monitoring equipment

Installation Instructions:

- Place new polyethylene (PE) sheet on suitable surface adjacent to well, taking care not to step on the PE sheet.
- Don a new pair of gloves.
- Decontaminate equipment such as hose (if not dedicated) as outlined in decontamination SOP.
- A check valve should be installed on the bottom of the tubing in order to prevent backflow of water into the well from the tubing and the pump after the pump is shut off. The check valve will also aid in priming the pump.
- Lower the tubing into well, being careful not to contact any surface other than the interior of the well or the PE sheeting. The outside of the tubing should be decontaminated following instructions in the Decontamination SOP.
- Place the intake of the tubing as high in the well as is possible but deep enough that it will not break suction.

Purging Instructions:

- Refuel the pump at a location that is remote from the well, being very careful not to spill any fuel on equipment or clothing that will be used at the well site. Pay strict attention to the required fuel mixture (gasoline/oil) that is specified by the pump manufacturer.

- Place the gasoline-powered compressor as far from the well as possible in a down-wind direction to eliminate potential exhaust impact to sampling.
- Connect the tubing to the pump.
- Don a new pair of gloves after handling the pump.
- Determine the volume of water to be purged, as described previously.
- Prime the pump using either distilled, deionized, or potable water. Use potable water only if the source has been tested analytically and found to be clear of the constituents of interest. Alternately, a pump equipped with a check valve on the tubing can be primed by working the tubing in an up and down motion, as with the intertial lift pump. This second approach should only be used if no other alternative is available for priming the pump.
- Start the pump.
- Direct the pump discharge to the five gallon pail and determine the pumping rate.
- Adjust the pumping rate to match the yield of the well.
- Continue pumping until the necessary volume of water has been purged from the well.
- If the pump intake tubing has been placed deeply down into the water column for some reason, slowly withdraw the tubing upward through the water column while it is still running to purge all water standing above the bottom intake.
- Dispose of the tubing after use, unless it is dedicated.
- Monitor indicator parameters as discussed previously.

Sampling Instructions:

- A centrifugal pump is not suitable and may not be used for sample collection.

Peristaltic Suction Pump

A peristaltic pump can be used to purge shallow, small diameter wells at a low to modest rate. The lift capacity is very limited with these pumps and often exceeded at groundwater depths of greater than 20 to 25 feet (depending upon pump size). This type of pump is normally used with dedicated tubing. Peristaltic pumps can also be used for sample collection for parameters that are not pH or pressure sensitive. Collecting groundwater samples from these pumps is only appropriate if approved project-specific planning documents specifically include this technique for collecting samples.

Required Equipment:

- Peristaltic pump, electric powered 12 VDC.
- 12 VDC power source, such as a sealed motorcycle battery or connection to vehicle battery.
- 3/8" to 1/2" PE, PP, or Teflon® tubing of appropriate length.
- 5/8" OD, 3/8" ID medical grade silicone tubing. Do not attempt to use Tygon® tubing.
- New disposable gloves of appropriate material.
- New PE sheeting.
- Five gallon pail, graduated in minimum one gallon increments.
- Water quality monitoring equipment (preferably a flow through cell).

Installation Instructions:

- Place new polyethylene (PE) sheet on suitable surface adjacent to well, taking care not to step on the PE sheet.
- Don a new pair of gloves.
- Replace the silicone tubing in the pump head if the pump will used for sample collection.
- Lower the tubing into the well, being careful not to contact any surface other than the interior of the well or the plastic sheeting.

- Place the intake of the tubing as high in the well as possible but deep enough that it will not break suction.

Purging Instructions:

- Connect the tubing to the pump.
- Determine the volume of water to be purged, as described above.
- Start the pump.
- Direct the pump discharge to the five gallon pail and determine the pumping rate.
- Continue pumping until the necessary volume of water has been purged from the well.
- If the pump intake tubing has been placed deeply down into the water column for some reason, slowly withdraw the tubing upward through the water column while it is still running to purge all water standing above the bottom intake.
- Dispose of the tubing after use.
- Monitor indicator parameters as discussed previously.

Sampling Instructions:

- Allow the well to recharge after completion of purging, if necessary.
- Resume pumping and adjust to a slow pumping rate, (approximately 100 ml/min) if the pump is equipped with a speed control.
- Collect the samples by pumping directly into each of the required containers.
- Bottles should be filled in order of sensitivity to volatilization and oxidation as discussed in Section 5.1.3 or as outlined in project-specific planning documents. Care should be taken to ensure that no head space remains in the volatile organic vials. Certain other parameters may also require minimizing headspace (e.g., reduced or ferrous iron).

- Filtered samples can easily be obtained by installing an in-line, 0.45 μm disposable cartridge filter directly onto the pump discharge.

Inertial Lift Pump

An inertial lift pump is a very simple device that can be used to purge and sample wells of almost any diameter to a depth of approximately 100 feet. This type of pump is normally dedicated to the well. Collecting groundwater samples from these pumps is only appropriate if approved project-specific planning documents specifically include this technique for collecting samples.

Required Equipment:

- 1/2" OD HDPE pump tubing
- Check valve
- Electric motor driven lift arm [OPTIONAL]
- Electric power source [OPTIONAL]
- New disposable gloves of appropriate material
- New plastic sheeting
- Five gallon pail, graduated in minimum one gallon increments
- Water quality monitoring equipment (preferably a flow through cell).

Installation Instructions:

- Place new polyethylene (PE) sheet on suitable surface adjacent to well, taking care not to step on the PE sheet.
- Don a new pair of gloves.
- Decontaminate equipment and pump (if not dedicated) as outlined in decontamination SOP.
- Install self threading valve on the bottom of the tubing

- Lower pump slowly into the well, being careful not to contact any surface other than the interior of the well or the plastic sheeting. When lowering the pump be particularly sensitive to areas that suggest drag or problems in the well where the pump could get stuck. If a problem exists do not continue, but discuss ways to investigate with PM or senior technical personnel.
- Place the tubing intake as high in the well as is possible but deep enough that it will not break suction. The tubing intake can be placed deeper in the well if the inertial lift pump will also be used for sample collection.

Purging Instructions:

- Refuel the electric generator, if used, at a location that is remote from the well, being very careful not to spill any fuel on equipment or clothing that will be used at the well site.
- Place the generator as far from the well as possible in a down-wind direction.
- Don a new pair of gloves after handling the generator.
- Connect the tubing to the pump arm, if used.
- Determine the volume of water to be purged, as described above.
- Start pumping by working the pump tubing in an up and down motion.
- Direct the pump discharge to the five gallon pail and determine the pumping rate.
- Continue pumping until the necessary volume of water has been purged from the well.
- If the pump intake tubing has been placed deeply down into the water column for some reason, slowly withdraw the tubing upward through the water column while it is still running to purge all water standing above the bottom intake. This should be accomplished by reconnecting the pump arm at progressively lower points on the tubing and allowing the pump to

operate for short periods of time. This step is not required if the inertial lift pump will be used for sample collection.

- Dispose of the tubing after use, unless it is left in the well.
- Monitor indicator parameters as discussed previously.

Sampling Instructions:

- Allow the well to recharge after completion of purging, if necessary.
- Resume pumping at a slow rate either by decreasing the speed of the lift arm unit or by operating the tubing manually.
- Collect the samples by pumping directly into each of the required containers.
- Bottles should be filled in order of sensitivity to volatilization and oxidation as discussed in Section 5.1.3 or as outlined in project-specific planning documents. Care should be taken to ensure that no head space remains in the volatile organic vials. Certain other parameters may also require minimizing headspace (e.g., reduced or ferrous iron).
- Filtered samples can easily be obtained by installing an in-line, 0.45 μm disposable cartridge filter directly onto the pump discharge.

5.2.7 Field Measurements

The final determination of pH, specific conductance, and temperature should be made immediately upon collection of the samples. It is preferred that these parameters be measured continuously using a water quality meter coupled with a "flow through" cell. Alternately, these measurements would be made in an aliquot contained in a disposable plastic cup.

5.3 FIELD RECORDS

Accurate field records must be maintained to document groundwater sampling activities. These records include technical field data, sample identification labels, and chain-of-custody information for each sample. These records are described in detail in the following sections, and discussed in the Field Documentation and Sampling Handling SOPs.

Specifically for groundwater sampling, the field sampling records should include, at a minimum, the following information:

- Sampling location
- Date and time
- Condition of the well
- Static water level (depth to water)
- Depth to the bottom of the well
- Calculated well volume
- Purging method
- Actual purged volume
- Sample collection method
- Sample description
- Field meter calibration data
- Water quality measurements
- General comments (weather conditions, etc.)

All data entries should be made using black indelible ink and should be written legibly. Entry errors should be crossed out with a single line, dated, and initialed by the person making the correction.

5.4 SAMPLE SHIPMENT

Shipment of samples to an analytical laboratory is usually required upon completion of sample collection. Proper packaging is necessary in order to protect the sample containers, to maintain the samples at or below a temperature of 4°C, and to comply with all applicable transportation regulations. See the Sampling Handling and Management SOP for further details.

5.5 SPECIAL REGULATORY REQUIREMENTS

A number of states and USEPA regions have specific groundwater sampling requirements that must be followed. These requirements must be determined in advance, and should be incorporated in to the project specific planning documents.

6.0 REFERENCES

American Society for Testing Materials. "D 4448, Standard Guide for Sampling Groundwater Monitoring Wells."

Armstrong, R. and F.R. McLaren, 1984. "The Suction Side Sample Catcher in Ground Water Quality Sampling," Ground Water Monitoring Review, Fall 1984, pp. 48-53.

Barcelona, M.J., J.P. Gibb, J.A. Helfrich and E.E. Garske, 1985. Practical Guide to Groundwater Sampling, Illinois State Geological Survey Contract Report 374, pp. 94.

Barcelona, M.J., and J.A. Helfrich, 1986. "Well Construction and Purging Effects on Ground-Water Samples," Environmental Science and Technology, vol. 20, no. 11, pp. 1179-1184.

Barcelona, M.J., J.A. Helfrich, and E.E. Garske, 1988. "Verification of Sampling Methods and Selection of Materials for Ground-Water Contamination Studies," Ground-Water Contamination: Field Methods, ASTM Special Technical Publication 963, pp. 221-231.

Barker, J.F. and R. Dickhout, 1988. "An Evaluation of Some Systems for Sampling Gas-Charged Ground Water for Volatile Organic Analysis," Ground Water Monitoring Review, Fall 1988, v. 8, no. 4, pp. 112-120.

Claassen, H.C., 1982. Guidelines and Techniques for Obtaining Water Samples That Accurately Represent the Water Chemistry of an Aquifer, U.S. Geological Survey, Open-File Report 82-1024, Lake, Colorado, pp. 49.

Clarke A.N., D. R. Davis and J. H. Clarke, "Environmental Project Definition with the Analytical Laboratory: The First Step Toward Total Quality Assurance," American Environmental Laboratory, October, 1990.

Ficken, J.H, 1988. "Recent Development of Downhole Water Samplers for Trace Organics," Ground-Water Contamination: Field Methods, ASTM Special Technical Publication 963, pp. 253-257.

Gibb, J.P., R.M. Schuller and R.A. Griffin, 1981. Procedures for the Collection of Representative Water Quality Data from Monitoring Wells. Illinois State Water Survey Cooperative Groundwater Report 7.

Gibb, J.P., R.M. Schuller, and R.A. Griffin, 1980. Monitoring Well Sampling and Preservation Techniques, EPA-600/9-80-101.

Gibs, Jacob, and Thomas E. Imbrigiotta, 1990. "Well-Purging Criteria for Sampling Purgeable Organic Compounds," Ground Water, vol. 28, no. 1.

Giddings, T., 1983. "Bore-Volume Purging to Improve Monitoring Well Performance: An Often-Mandated Myth," Proceedings of the Third National Symposium on Aquifer Restoration and Ground Water Monitoring, National Water Well Association, Dublin, Ohio, pp. 253-256.

Gillham, R.W., M.J.L. Robin, J.F. Barker and J.A. Cherry, 1983. Groundwater Monitoring and Sample Bias, Environmental Affairs Department, American Petroleum Institute, pp. 206.

Herzog, B.L., S.J. Chou, J.R. Valkenburg, and R.A. Griffin, 1988. "Changes in Volatile Organic Chemical Concentrations After Purging Slowly Recovering Wells," Ground Water Monitoring Review, Fall 1988, v. 8, no. 4, pp. 93-99.

Herzog, B., J. Pennino, and G. Nielsen, 1991. "Ground-Water Sampling," Practical Handbook of Ground-Water Monitoring, David M. Nielsen editor, Lewis Publishers, Chelsea, MI, pp. 449-499.

Ho, J.S.Y., 1983. "Effect of Sampling Variables on Recovery of Volatile Organics in Water," Journal of American Water Works, November 1983, pp. 583-586.

Imbrigiotta, T.E., J. Gibs, T.V. Fusillo, G.R. Kish, and J.J. Hochreiter, 1988. "Field Evaluation of Seven Sampling Devices for Purgeable Organic Compounds in Ground Water," Ground-Water Contamination: Field Methods, ASTM Special Technical Publication 963, pp. 258-273.

Kasper, R.B. and J.A. Serkowski, 1988. "Evaluation of Sampling Equipment for RCRA Monitoring in a Deep Unconfined Aquifer," Proceedings of the Second National Outdoor Action Conference on Aquifer Restoration and Ground Water Monitoring, National Water Well Association, Worthington, Ohio, pp. 445-455.

McAlary, T.A. and J.F. Barker, 1987. "Volatilization Losses of Organics During Ground Water Sampling from Low Permeability Materials," Ground Water Monitoring Review, Fall 1987, pp. 63-68.

NCASI, 1982. "A Guide to Groundwater Sampling", NCASI Technical Bulletin, No. 362, January 1982.

New Jersey Department of Environmental Protection, 1988. Field Sampling Procedures Manual, pp. 414

Nielsen, D.M. and G.L. Yeates, 1985. "A Comparison of Sampling Mechanisms Available for Small-Diameter Ground Water Monitoring Wells," Ground Water Monitoring Review, Spring 1985, pp. 83-99.

Panko, A.W. and P. Barth, 1988. "Chemical Stability Prior to Ground-Water Sampling: A Review of Current Well Purging Methods," Ground-Water Contamination: Field Methods, ASTM Special Technical Publication 963, pp. 232-239.

Rannie, E.H. and R.L. Nadon, 1988. "An Inexpensive, Multi-Use, Dedicated Pump for Ground Water Monitoring Wells," Ground Water Monitoring Review, Fall 1988, v. 8, no. 4, pp. 100-107.

Scalf, M.R., J.F. McNabb, W.J. Dunlap, R.L. Cosby, and J.S. Fryberger, 1981. Manual of Ground-Water Quality Sampling Procedures, September 1981, Robert S. Kerr Environmental Research Laboratory, Ada, Oklahoma, EPA-600/2-81-160.

Schalla, R., D.A. Myers, M.A. Simmons, J.M. Thomas, and A.P. Toste, 1988. "The Sensitivity of Four Monitoring Well Sampling Systems to Low Concentrations of Three Volatile Organics," Ground Water Monitoring Review, Summer 1988, pp. 90-96.

Schuller, R.M., J.P. Gibb, and R.A. Griffin, 1981. "Recommended Sampling Procedures for Monitoring Wells," Ground Water Monitoring Review, Spring 1981, pp. 42-46.

United States Environmental Protection Agency Region IV, 1996. Environmental Investigations and Standard Operating Procedures and Quality Assurance Manual, 1996, pp. 7-1 - 7-10.

United States Environmental Protection Agency, 1991. Compendium of ERT Groundwater Sampling Procedures, EPA/540/P-91/007, January 1991.

Unwin, J.P. and D. Huis, 1983. "A Laboratory Investigation of the Purging Behavior of Small-Diameter Monitoring Well," Proceedings of the Third Annual Symposium on Ground Water Monitoring and Aquifer Restoration, National Water Well Association, Dublin, Ohio, pp. 257-262.

Unwin, J. and V. Maltby, 1988. "Investigations of Techniques for Purging Ground-Water Monitoring Wells and Sampling Ground Water for Volatile Organic Compounds," Ground-Water Contamination: Field Methods, ASTM Special Technical Publication 963, pp. 240-252.

7.0 ATTACHMENTS

Attachment A - Groundwater Sampling Checklist

Attachment B - Summary of Well Purging Equipment

Attachment C - Summary of Well Sampling Equipment

Attachment D - Purging and Sampling Equipment Matrix

Attachment E – Example Groundwater Sampling Field Data Sheet

ATTACHMENTS

Attachment A. Groundwater Sampling Checklist

Planning

- ☐ Select well purging method
- ☐ Determine well purging strategy
  - ☐ Purging until of indicator parameters stabilize (±10%).
  - ☐ Purge predetermined number of well volumes (usually 3 to 5).
  - ☐ Low yield wells purged until dryness.
  - ☐ Others
- ☐ Determine containerization and disposal method for purge water
- ☐ Select well sampling method
- ☐ Determine groundwater sampling sequence

Preparatory Office Activities

- ☐ Obtain and decontaminate appropriate purging and sample collection equipment.
- ☐ Obtain appropriate sample containers and preservatives from laboratory.
- ☐ Inventory sample containers and preservatives.
- ☐ Pre-label sample containers.
- ☐ Initiate field data record for each well.
- ☐ Communicate sampling schedule with analytical laboratory and site contact or other parties, as required.

Preparatory Field Activities

- ☐ Perform well maintenance check.
- ☐ Prepare clean work area.
- ☐ Determine the depth to groundwater (±0.02 ft).
- ☐ Calculate the water volume standing in the well (±2%).

Well Purging

- ☐ Place purging device at proper depth to ensure complete purging of well (if device is not also used for sample collection).
- ☐ Purge well, following previously selected strategy.
- ☐ Handle and dispose of purge water using previously determined method.

Well Sampling

- ☐ Collect groundwater sample.
- ☐ Fill containers and made field determinations in order of decreasing sensitivity to volatilization and/or pH change.
- ☐ Fill all other sample containers.
- ☐ Record all technical data.
- ☐ Maintain chain of custody records.
- ☐ Pack and ship samples to prevent breakage, to maintain sample temperature of 4°C and to comply with Dangerous Goods regulations.

Attachment B. Summary of Well Purging Equipment

Description	Pumping Rate (gpm)	Well Diam. (inches)	Depth Limit (feet)	Can it be Dedicated?	Materials	Advantages	Disadvantages	Manufacturers
Bailer								
Tube with bottom and/or top check valve, suspended by line.	0.2 - 2	Unlimited	Unlimited	Yes	• PVC • Teflon ® • HDPE • SS	• Inexpensive • Simple • Can yield high quality samples • No power required • Easily cleaned	• Awkward to use • Can aerate sample if not used carefully • Increases likelihood of turbid samples • Labor intensive	Timco: Johnson: Voss:
Small Diameter (2") Electric Submersible Pump								
Electrically driven impeller pump.	0.1 - 9	2 - 4	75 - 300	Yes	• SS / Teflon ®	• Relatively high pump rate • Effective for deep well purging	• Requires 230 VAC power source • High capital cost • Deep sampling reduces efficiency	Grundfos Fultz
Large Diameter (4") Electric Submersible Pump								
Electrically driven impeller pump.	4 - 20	> = 4	Unlimited	Not typically	• SS	• High pump rate • Effective for deep wells • Moderate cost	• Difficult to handle & decon. • Requires AC power source	Grundfos
Peristaltic (Suction) Pump								
Elastic tubing that is sequentially squeezed by rollers.	1-3	< = 2	± 25	Yes, internal elastic tubing & pump tubing	• Teflon ® • HDPE • PP	• Flow easily controlled • Portable • Inexpensive • Relatively simple	• Requires power source • Slow purging rate	Isco, Masterflex

Notes:

SS: Stainless Steel
HDPE: High density polyethylene
PP: Polypropylene
VAC: Voltage AC Source
PVC: Polyvinyl Chloride
TSS: Total Suspended Solids
VOCs: Volatile Organic Compounds

Attachment C. Summary of Well Sampling Equipment

Description	Well Diam. (inches)	Depth Limit (feet)	Can it be Dedicated?	Materials	Reduce Flow to 100ml/m?	Acceptable for: VOC	Acceptable for: pH Sensitive	Advantages	Disadvantages	Manufacturers
Bailer										
Tube with bottom and/or top check valve, suspended by line.	Unlimited	Unlimited	Yes	• PVC • Teflon ® • HDPE • SS	Yes	Yes	Yes	• Simple, inexpensive • Can yield high quality samples if used carefully • No power required • Easily cleaned	• Awkward to use • Can aerate sample if not used carefully • Increases chance of turbid samples • Flow not continuous	Timco: Johnson: Voss:
Small Diameter (2") Electric Submersible Pump										
Electrically driven impeller pump.	2 - 4	400	Yes	• SS / Teflon ®	Yes	Yes	Yes	• Easy to operate • Easy to control • Continuous flow	• High capital cost • Awkward if not dedicated	Grundfos
Large Diameter (4") Electric Submersible Pump										
Electrically driven impeller pump.	>=4	unlimited	Not typically	• SS	No	No	No	• Continuous flow	• May not be compatible with VOC & pressure sensitive parameters	Grundfos
Peristaltic (Suction) Pump										
Elastic tubing that is sequentially squeezed by rollers.	<=2	± 25	Yes, internal elastic tubing & pump tubing	• Teflon ® • HDPE • PP	No	Yes		• Flow rate easily controlled, continuo • Portable • Inexpensive • No pump contact with sample	• Loss of VOC due to reduced pressure • Requires power source • May require priming • Can impede access	Isco, Masterflex

Notes:

SS: Stainless Steel VAC: Voltage AC Source VOCs: Volatile Organic Compounds

HDPE: High density polyethylene PVC: Polyvinyl Chloride PP: Polyprplene

Attachment D - Purging and Sampling Equipment Matrix

Methods	4 inch wells									2 inch wells										<2 inch wells								
	Water Level < 20'						W.L. > 20'			Water Level < 20'						W.L. > 20'				Water Level < 20'						W.L. > 20'		
	Cs>0.2			Cs<0.2						Cs>0.2			Cs<0.2							Cs>0.2			Cs<0.2					
	Sample			Sample			Sample			Sample			Sample			Sample				Sample			Sample			Sample		
	Purging	VOC/pH	Other	Purging	VOC/pH	Other	Purging	VOC/pH	Other	Purging	VOC/pH	Other	Purging	VOC/pH	Other	Purging	VOC/pH	Other		Purging	VOC/pH	Other	Purging	VOC/pH	Other	Purging	VOC/pH	Other
Methods Described in this SOP																												
Bailer	X	X	X	X	X	X	I	X	X	X	X	X	X	X	X	I	X	X	X	X	X	X	X	X	X	X	X	X
2" Electric Submersible Pump	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X										
4" Electric Submersible Pump	X	O	X	X	O	X	X	O	X																			
Peristaltic Pump	X	O	X							X	O	X							X	O	X							
Other Methods To Consider																												
Bladder Pump	X	X	X	X	X	X	I	X	X	X	X	X	X	X	X	X	X	X										
Gas Driven Piston Pump	X	X	X	X	X	X	I	X	X	X	X	X	X	X	X	X	X	X										
Centrifugal Pump	X									X									X									
Syringe Sampler		X	X		X	X		X	X		X	X		X	X		X	X										
Inertial Lift Pump	X	O	X	X	O	X	I	O	X	X	O	X	X	O	X	X	O	X	X	O	X	X	O	X	X	O	X	X

Notes: W.L. - Water Level
Cs - Specific Capacity (gallons per foot of drawdown)
VOC - Volatile Organic Compounds
X - Recommended method.
O - Conditionally acceptable Method, based upon technical concerns.
I - Inefficient method but technically acceptable.
- Not recommended or not feasible.

GROUNDWATER PURGE AND SAMPLING FIELD DATA SHEET

1. PROJECT INFORMATION

WELL ID: _____

Project Number:_____ Task Number:_____ Date:_____ Time:_____

Client:_____ Personnel:_____

Project Location:_____ Weather:_____

2. WELL DATA

Casing Diameter:_____ inches | Type of Casing: _____

Screen Diameter:_____ inches (d) | Type of Screen:_____ Screen Length : _____

Total Depth of Well from TOC:_____ feet

Depth to Static Water from TOC:_____ feet

Depth to Product from TOC:_____ feet

Length of Water Column (h):_____ feet Calculated Casing Volume:_____ gal (3 to 5 times one well volume)

Purge Volume Calculation (one casing volume = $0.041d^2h$):

Note: 2-inch well = 0.167 gal/ft 4-inch well = 0.667 gal/ft

3. PURGE DATA

Purge Method: _____

Materials: Pump/Bailer _____

Materials: Rope/Tubing _____

Was well purged dry? ❑ Yes ❑ No Pumping Rate: gal/min

Equipment Model(s)

1. _____

2. _____

Time	Cum. Gallons Removed	pH	Temp (Units)	Spec. Cond. (Units)	Eh (Units)	DO (Units)	Turbidity (NTU)	Other: _____	Comments	

4. SAMPLING DATA

Analyses Requested:

Method(s): _____

Materials: Pump/Bailer _____

Materials: Tubing/Rope _____

Depth to Water at Time of Sampling:_____ Field Filtered? ❑ Yes ❑ No

Sample ID:_____ Sample Time:_____ # of Containers:_____

Duplicate Sample Collected? ❑ Yes ❑ No ID:_____

5. COMMENTS

_____

Designation: D 1586 – 08a

Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils[1]

This standard is issued under the fixed designation D 1586; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method describes the procedure, generally known as the Standard Penetration Test (SPT), for driving a split-barrel sampler to obtain a representative disturbed soil sample for identification purposes, and measure the resistance of the soil to penetration of the sampler. Another method (Test Method D 3550) to drive a split-barrel sampler to obtain a representative soil sample is available but the hammer energy is not standardized.

1.2 Practice D 6066 gives a guide to determining the normalized penetration resistance of sands for energy adjustments of N-value to a constant energy level for evaluating liquefaction potential.

1.3 Test results and identification information are used to estimate subsurface conditions for foundation design.

1.4 Penetration resistance testing is typically performed at 5-foot depth intervals or when a significant change of materials is observed during drilling, unless otherwise specified.

1.5 This test method is limited to use in nonlithified soils and soils whose maximum particle size is approximately less than one-half of the sampler diameter.

1.6 This test method involves use of rotary drilling equipment (Guide D 5783, Practice D 6151). Other drilling and sampling procedures (Guide D 6286, Guide D 6169) are available and may be more appropriate. Considerations for hand driving or shallow sampling without boreholes are not addressed. Subsurface investigations should be recorded in accordance with Practice D 5434. Samples should be preserved and transported in accordance with Practice D 4220 using Group B. Soil samples should be identified by group name and symbol in accordance with Practice D 2488.

1.7 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D 6026, unless superseded by this test method.

1.8 The values stated in inch-pound units are to be regarded as standard, except as noted below. The values given in parentheses are mathematical conversions to SI units, which are provided for information only and are not considered standard.

1.8.1 The gravitational system of inch-pound units is used when dealing with inch-pound units. In this system, the pound (lbf) represents a unit of force (weight), while the unit for mass is slugs.

1.9 Penetration resistance measurements often will involve safety planning, administration, and documentation. This test method does not purport to address all aspects of exploration and site safety. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Performance of the test usually involves use of a drill rig; therefore, safety requirements as outlined in applicable safety standards (for example, OSHA regulations,[2] NDA Drilling Safety Guide,[3] drilling safety manuals, and other applicable state and local regulations) must be observed.

2. Referenced Documents

2.1 *ASTM Standards:* [4]

D 653 Terminology Relating to Soil, Rock, and Contained Fluids

D 854 Test Methods for Specific Gravity of Soil Solids by Water Pycnometer

D 1587 Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes

D 2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D 2487 Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)

D 2488 Practice for Description and Identification of Soils

[1] This method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.02 on Sampling and Related Field Testing for Soil Evaluations.

Current edition approved Oct. 1, 2008. Published October 2008. Originally approved in 1958. Last previous edition approved in 2008 as D 1586 – 08.

[2] Available from Occupational Safety and Health Administration (OSHA), 200 Constitution Ave., NW, Washington, DC 20210, http://www.osha.gov.

[3] Available from the National Drilling Association, 3511 Center Rd., Suite 8, Brunswick, OH 44212, http://www.nda4u.com.

[4] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

(Visual-Manual Procedure)
D 3550 Practice for Thick Wall, Ring-Lined, Split Barrel, Drive Sampling of Soils
D 3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction
D 4220 Practices for Preserving and Transporting Soil Samples
D 4633 Test Method for Energy Measurement for Dynamic Penetrometers
D 5434 Guide for Field Logging of Subsurface Explorations of Soil and Rock
D 5783 Guide for Use of Direct Rotary Drilling with Water-Based Drilling Fluid for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices
D 6026 Practice for Using Significant Digits in Geotechnical Data
D 6066 Practice for Determining the Normalized Penetration Resistance of Sands for Evaluation of Liquefaction Potential
D 6151 Practice for Using Hollow-Stem Augers for Geotechnical Exploration and Soil Sampling
D 6169 Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations
D 6286 Guide for Selection of Drilling Methods for Environmental Site Characterization
D 6913 Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis

3. Terminology

3.1 *Definitions:* Definitions of terms included in Terminology D 653 specific to this practice are:

3.1.1 *cathead*, *n*—the rotating drum or windlass in the rope-cathead lift system around which the operator wraps a rope to lift and drop the hammer by successively tightening and loosening the rope turns around the drum.

3.1.2 *drill rods*, *n*—rods used to transmit downward force and torque to the drill bit while drilling a borehole.

3.1.3 *N-value*, *n*—the blow count representation of the penetration resistance of the soil. The *N*-value, reported in blows per foot, equals the sum of the number of blows (*N*) required to drive the sampler over the depth interval of 6 to 18 in. (150 to 450 mm) (see 7.3).

3.1.4 *Standard Penetration Test (SPT)*, *n*—a test process in the bottom of the borehole where a split-barrel sampler having an inside diameter of either 1-1/2-in. (38.1 mm) or 1-3/8-in. (34.9 mm) (see Note 2) is driven a given distance of 1.0 ft (0.30 m) after a seating interval of 0.5 ft (0.15 m) using a hammer weighing approximately 140-lbf (623-N) falling 30 ± 1.0 in. (0.76 m ± 0.030 m) for each hammer blow.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *anvil*, *n*—that portion of the drive-weight assembly which the hammer strikes and through which the hammer energy passes into the drill rods.

3.2.2 *drive weight assembly*, *n*—an assembly that consists of the hammer, anvil, hammer fall guide system, drill rod attachment system, and any hammer drop system hoisting attachments.

3.2.3 *hammer*, *n*—that portion of the drive-weight assembly consisting of the 140 ± 2 lbf (623 ± 9 N) impact weight which is successively lifted and dropped to provide the energy that accomplishes the sampling and penetration.

3.2.4 *hammer drop system*, *n*—that portion of the drive-weight assembly by which the operator or automatic system accomplishes the lifting and dropping of the hammer to produce the blow.

3.2.5 *hammer fall guide*, *n*—that part of the drive-weight assembly used to guide the fall of the hammer.

3.2.6 *number of rope turns*, *n*—the total contact angle between the rope and the cathead at the beginning of the operator's rope slackening to drop the hammer, divided by 360° (see Fig. 1).

3.2.7 *sampling rods*, *n*—rods that connect the drive-weight assembly to the sampler. Drill rods are often used for this purpose.

4. Significance and Use

4.1 This test method provides a disturbed soil sample for moisture content determination, for identification and classification (Practices D 2487 and D 2488) purposes, and for laboratory tests appropriate for soil obtained from a sampler that will produce large shear strain disturbance in the sample such as Test Methods D 854, D 2216, and D 6913. Soil deposits containing gravels, cobbles, or boulders typically result in penetration refusal and damage to the equipment.

4.2 This test method provides a disturbed soil sample for moisture content determination and laboratory identification. Sample quality is generally not suitable for advanced laboratory testing for engineering properties. The process of driving the sampler will cause disturbance of the soil and change the engineering properties. Use of the thin wall tube sampler (Practice D 1587) may result in less disturbance in soft soils. Coring techniques may result in less disturbance than SPT sampling for harder soils, but it is not always the case, that is, some cemented soils may become loosened by water action during coring; see Practice D 6151, and Guide D 6169.

4.3 This test method is used extensively in a great variety of geotechnical exploration projects. Many local correlations and widely published correlations which relate blow count, or *N*-value, and the engineering behavior of earthworks and foundations are available. For evaluating the liquefaction potential of sands during an earthquake event, the *N*-value should be normalized to a standard overburden stress level. Practice D 6066 provides methods to obtain a record of normalized resistance of sands to the penetration of a standard sampler driven by a standard energy. The penetration resistance is adjusted to drill rod energy ratio of 60 % by using a hammer system with either an estimated energy delivery or directly measuring drill rod stress wave energy using Test Method D 4633.

NOTE 1—The reliability of data and interpretations generated by this practice is dependent on the competence of the personnel performing it

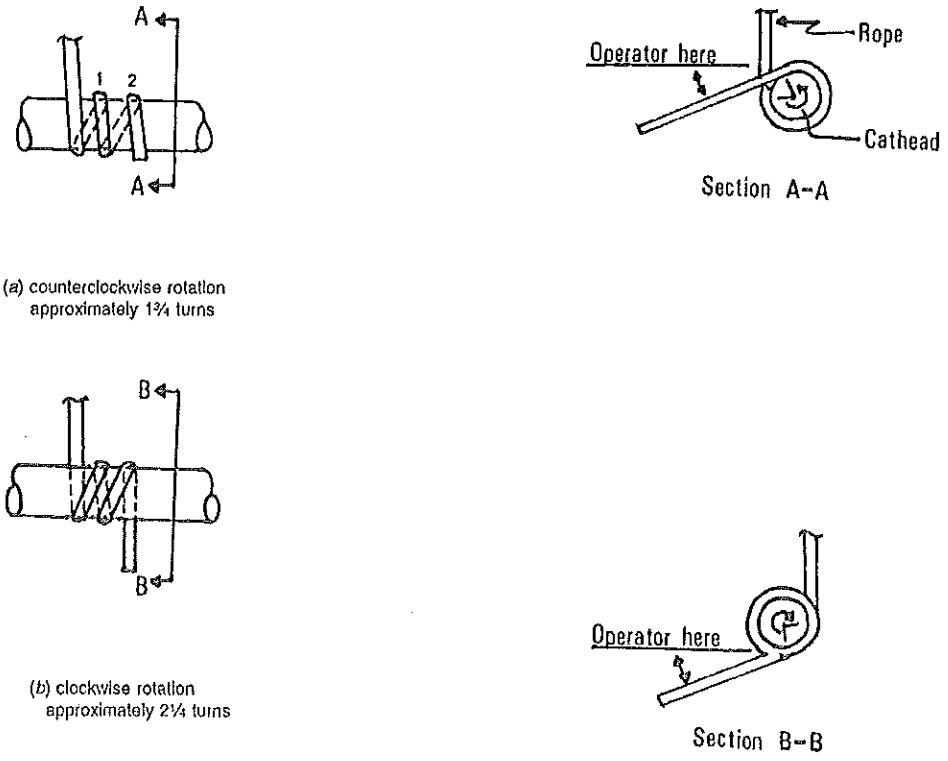


FIG. 1 Definitions of the Number of Rope Turns and the Angle for (*a*) Counterclockwise Rotation and (*b*) Clockwise Rotation of the Cathead

and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 generally are considered capable of competent testing. Users of this practice are cautioned that compliance with Practice D 3740 does not assure reliable testing. Reliable testing depends on several factors and Practice D 3740 provides a means of evaluating some of these factors. Practice D 3740 was developed for agencies engaged in the testing, inspection, or both, of soils and rock. As such, it is not totally applicable to agencies performing this practice. Users of this test method should recognize that the framework of Practice D 3740 is appropriate for evaluating the quality of an agency performing this test method. Currently, there is no known qualifying national authority that inspects agencies that perform this test method.

5. Apparatus

5.1 *Drilling Equipment*—Any drilling equipment that provides at the time of sampling a suitable borehole before insertion of the sampler and ensures that the penetration test is performed on intact soil shall be acceptable. The following pieces of equipment have proven to be suitable for advancing a borehole in some subsurface conditions:

5.1.1 *Drag, Chopping, and Fishtail Bits*, less than 6½ in. (165 mm) and greater than 2¼ in. (57 mm) in diameter may be used in conjunction with open-hole rotary drilling or casing-advancement drilling methods. To avoid disturbance of the underlying soil, bottom discharge bits are not permitted; only side discharge bits are permitted.

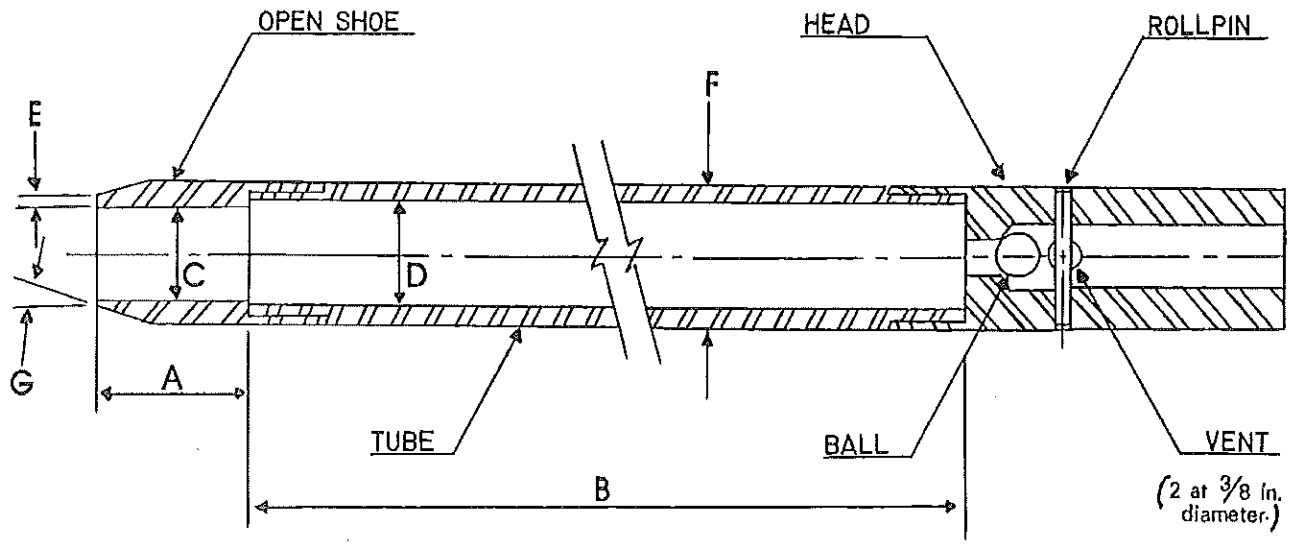
5.1.2 *Roller-Cone Bits*, less than 6½ in. (165 mm) and greater than 2¼ in. (57 mm) in diameter may be used in conjunction with open-hole rotary drilling or casing-advancement drilling methods if the drilling fluid discharge is deflected.

5.1.3 *Hollow-Stem Continuous Flight Augers*, with or without a center bit assembly, may be used to drill the borehole. The inside diameter of the hollow-stem augers shall be less than 6½ in. (165 mm) and not less than 2¼ in. (57 mm).

5.1.4 *Solid, Continuous Flight, Bucket and Hand Augers*, less than 6½ in. (165 mm) and not less than 2¼ in. (57 mm) in diameter may be used if the soil on the side of the borehole does not cave onto the sampler or sampling rods during sampling.

5.2 *Sampling Rods*—Flush-joint steel drill rods shall be used to connect the split-barrel sampler to the drive-weight assembly. The sampling rod shall have a stiffness (moment of inertia) equal to or greater than that of parallel wall "A" rod (a steel rod that has an outside diameter of 1-5/8 in. (41.3 mm) and an inside diameter of 1-1/8 in. (28.5 mm).

5.3 *Split-Barrel Sampler*—The standard sampler dimensions are shown in Fig. 2. The sampler has an outside diameter of 2.00 in. (50.8 mm). The inside diameter of the of the split-barrel (dimension D in Fig. 2) can be either 1½-in. (38.1



A = 1.0 to 2.0 in. (25 to 50 mm)
B = 18.0 to 30.0 in. (0.457 to 0.762 m)
C = 1.375± 0.005 in. (34.93 ± 0.13 mm)
D = 1.50 ± 0.05 – 0.00 in. (38.1 ± 1.3 – 0.0 mm)
E = 0.10 ± 0.02 in. (2.54 ± 0.25 mm)
F = 2.00 ± 0.05 – 0.00 in. (50.8 ± 1.3 – 0.0 mm)
G = 16.0° to 23.0°

FIG. 2 Split-Barrel Sampler

mm) or 1⅜-in. (34.9 mm) (see Note 2). A 16-gauge liner can be used inside the 1½-in. (38.1 mm) split barrel sampler. The driving shoe shall be of hardened steel and shall be replaced or repaired when it becomes dented or distorted. The penetrating end of the drive shoe may be slightly rounded. The split-barrel sampler must be equipped with a ball check and vent. Metal or plastic baskets may be used to retain soil samples.

NOTE 2—Both theory and available test data suggest that *N*-values may differ as much as 10 to 30 % between a constant inside diameter sampler and upset wall sampler. If it is necessary to correct for the upset wall sampler refer to Practice D 6066. In North America, it is now common practice to use an upset wall sampler with an inside diameter of 1½ in. At one time, liners were used but practice evolved to use the upset wall sampler without liners. Use of an upset wall sampler allows for use of retainers if needed, reduces inside friction, and improves recovery. Many other countries still use a constant ID split-barrel sampler, which was the original standard and still acceptable within this standard.

5.4 *Drive-Weight Assembly*:

5.4.1 *Hammer and Anvil*—The hammer shall weigh 140 ± 2 lbf (623 ± 9 N) and shall be a rigid metallic mass. The hammer shall strike the anvil and make steel on steel contact when it is dropped. A hammer fall guide permitting an unimpeded fall shall be used. Fig. 3 shows a schematic of such hammers. Hammers used with the cathead and rope method shall have an unimpeded over lift capacity of at least 4 in. (100 mm). For safety reasons, the use of a hammer assembly with an internal anvil is encouraged as shown in Fig. 3. The total mass of the hammer assembly bearing on the drill rods should not be more than 250 ± 10 lbm (113 ± 5 kg).

NOTE 3—It is suggested that the hammer fall guide be permanently marked to enable the operator or inspector to judge the hammer drop height.

5.4.2 *Hammer Drop System*—Rope-cathead, trip, semi-automatic or automatic hammer drop systems may be used, providing the lifting apparatus will not cause penetration of the sampler while re-engaging and lifting the hammer.

5.5 *Accessory Equipment*—Accessories such as labels, sample containers, data sheets, and groundwater level measuring devices shall be provided in accordance with the requirements of the project and other ASTM standards.

6. Drilling Procedure

6.1 The borehole shall be advanced incrementally to permit intermittent or continuous sampling. Test intervals and locations are normally stipulated by the project engineer or geologist. Typically, the intervals selected are 5 ft (1.5 m) or less in homogeneous strata with test and sampling locations at every change of strata. Record the depth of drilling to the nearest 0.1 ft (0.030 m).

6.2 Any drilling procedure that provides a suitably clean and stable borehole before insertion of the sampler and assures that the penetration test is performed on essentially intact soil shall be acceptable. Each of the following procedures has proven to be acceptable for some subsurface conditions. The subsurface conditions anticipated should be considered when selecting the drilling method to be used.

6.2.1 Open-hole rotary drilling method.

6.2.2 Continuous flight hollow-stem auger method.

6.2.3 Wash boring method.

6.2.4 Continuous flight solid auger method.

6.3 Several drilling methods produce unacceptable boreholes. The process of jetting through an open tube sampler and then sampling when the desired depth is reached shall not be

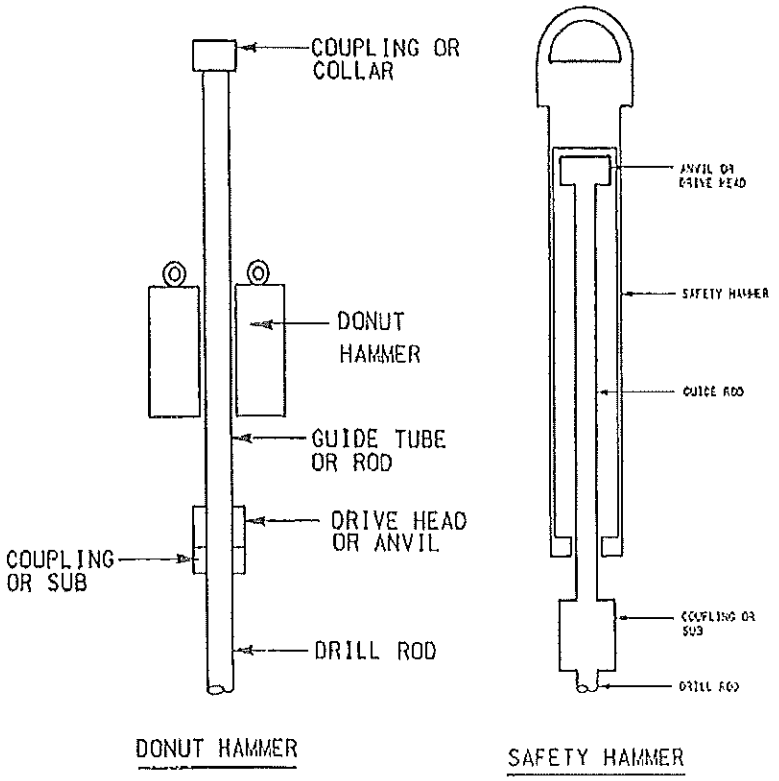


FIG. 3 Schematic Drawing of the Donut Hammer and Safety Hammer

permitted. The continuous flight solid auger method shall not be used for advancing the borehole below a water table or below the upper confining bed of a confined non-cohesive stratum that is under artesian pressure. Casing may not be advanced below the sampling elevation prior to sampling. Advancing a borehole with bottom discharge bits is not permissible. It is not permissible to advance the borehole for subsequent insertion of the sampler solely by means of previous sampling with the SPT sampler.

6.4 The drilling fluid level within the borehole or hollow-stem augers shall be maintained at or above the in situ groundwater level at all times during drilling, removal of drill rods, and sampling.

7. Sampling and Testing Procedure

7.1 After the borehole has been advanced to the desired sampling elevation and excessive cuttings have been removed, record the cleanout depth to the nearest 0.1 ft (0.030 m), and prepare for the test with the following sequence of operations:

7.1.1 Attach either split-barrel sampler Type A or B to the sampling rods and lower into the borehole. Do not allow the sampler to drop onto the soil to be sampled.

7.1.2 Position the hammer above and attach the anvil to the top of the sampling rods. This may be done before the sampling rods and sampler are lowered into the borehole.

7.1.3 Rest the dead weight of the sampler, rods, anvil, and drive weight on the bottom of the borehole. Record the sampling start depth to the nearest 0.1 ft (0.030 m). Compare the sampling start depth to the cleanout depth in 7.1. If excessive cuttings are encountered at the bottom of the borehole, remove the sampler and sampling rods from the borehole and remove the cuttings.

7.1.4 Mark the drill rods in three successive 0.5-foot (0.15 m) increments so that the advance of the sampler under the impact of the hammer can be easily observed for each 0.5-foot (0.15 m) increment.

7.2 Drive the sampler with blows from the 140-lbf (623-N) hammer and count the number of blows applied in each 0.5-foot (0.15-m) increment until one of the following occurs:

7.2.1 A total of 50 blows have been applied during any one of the three 0.5-foot (0.15-m) increments described in 7.1.4.

7.2.2 A total of 100 blows have been applied.

7.2.3 There is no observed advance of the sampler during the application of 10 successive blows of the hammer.

7.2.4 The sampler is advanced the complete 1.5 ft. (0.45 m) without the limiting blow counts occurring as described in 7.2.1, 7.2.2, or 7.2.3.

7.2.5 If the sampler sinks under the weight of the hammer, weight of rods, or both, record the length of travel to the nearest 0.1 ft (0.030 m), and drive the sampler through the remainder of the test interval. If the sampler sinks the complete interval, stop the penetration, remove the sampler and sampling rods from the borehole, and advance the borehole through the very soft or very loose materials to the next desired sampling elevation. Record the *N*-value as either weight of hammer, weight of rods, or both.

7.3 Record the number of blows (*N*) required to advance the sampler each 0.5-foot (0.15 m) of penetration or fraction

thereof. The first 0.5-foot (0.15 m) is considered to be a seating drive. The sum of the number of blows required for the second and third 0.5-foot (0.15 m) of penetration is termed the "standard penetration resistance," or the "*N*-value." If the sampler is driven less than 1.5 ft (0.45 m), as permitted in 7.2.1, 7.2.2, or 7.2.3, the number of blows per each complete 0.5-foot (0.15 m) increment and per each partial increment shall be recorded on the boring log. For partial increments, the depth of penetration shall be reported to the nearest 0.1 ft (0.030 m) in addition to the number of blows. If the sampler advances below the bottom of the borehole under the static weight of the drill rods or the weight of the drill rods plus the static weight of the hammer, this information should be noted on the boring log.

7.4 The raising and dropping of the 140-lbf (623-N) hammer shall be accomplished using either of the following two methods. Energy delivered to the drill rod by either method can be measured according to procedures in Test Method D 4633.

7.4.1 *Method A*—By using a trip, automatic, or semi-automatic hammer drop system that lifts the 140-lbf (623-N) hammer and allows it to drop 30 ± 1.0 in. (0.76 m ± 0.030 m) with limited unimpedence. Drop heights adjustments for automatic and trip hammers should be checked daily and at first indication of variations in performance. Operation of automatic hammers shall be in strict accordance with operations manuals.

7.4.2 *Method B*—By using a cathead to pull a rope attached to the hammer. When the cathead and rope method is used the system and operation shall conform to the following:

7.4.2.1 The cathead shall be essentially free of rust, oil, or grease and have a diameter in the range of 6 to 10 in. (150 to 250 mm).

7.4.2.2 The cathead should be operated at a minimum speed of rotation of 100 RPM.

7.4.2.3 The operator should generally use either 1-3/4 or 2-1/4 rope turns on the cathead, depending upon whether or not the rope comes off the top (1-3/4 turns for counterclockwise rotation) or the bottom (2-1/4 turns for clockwise rotation) of the cathead during the performance of the penetration test, as shown in Fig. 1. It is generally known and accepted that 2-3/4 or more rope turns considerably impedes the fall of the hammer and should not be used to perform the test. The cathead rope should be stiff, relatively dry, clean, and should be replaced when it becomes excessively frayed, oily, limp, or burned.

7.4.2.4 For each hammer blow, a 30 ± 1.0 in. (0.76 m ± 0.030 m) lift and drop shall be employed by the operator. The operation of pulling and throwing the rope shall be performed rhythmically without holding the rope at the top of the stroke.

NOTE 4—If the hammer drop height is something other than 30 ± 1.0 in. (0.76 m ± 0.030 m), then record the new drop height. For soils other than sands, there is no known data or research that relates to adjusting the *N*-value obtained from different drop heights. Test method D 4633 provides information on making energy measurement for variable drop heights and Practice D 6066 provides information on adjustment of *N*-value to a constant energy level (60 % of theoretical, N60). Practice D 6066 allows the hammer drop height to be adjusted to provide 60 % energy.

7.5 Bring the sampler to the surface and open. Record the percent recovery to the nearest 1 % or the length of sample recovered to the nearest 0.01 ft (5 mm). Classify the soil samples recovered as to, in accordance with Practice D 2488, then place one or more representative portions of the sample into sealable moisture-proof containers (jars) without ramming or distorting any apparent stratification. Seal each container to prevent evaporation of soil moisture. Affix labels to the containers bearing job designation, boring number, sample depth, and the blow count per 0.5-foot (0.15-m) increment. Protect the samples against extreme temperature changes. If there is a soil change within the sampler, make a jar for each stratum and note its location in the sampler barrel. Samples should be preserved and transported in accordance with Practice D 4220 using Group B.

8. Data Sheet(s)/Form(s)

8.1 Data obtained in each borehole shall be recorded in accordance with the Subsurface Logging Guide D 5434 as required by the exploration program. An example of a sample data sheet is included in Appendix X1.

8.2 Drilling information shall be recorded in the field and shall include the following:

8.2.1 Name and location of job,

8.2.2 Names of crew,

8.2.3 Type and make of drilling machine,

8.2.4 Weather conditions,

8.2.5 Date and time of start and finish of borehole,

8.2.6 Boring number and location (station and coordinates, if available and applicable),

8.2.7 Surface elevation, if available,

8.2.8 Method of advancing and cleaning the borehole,

8.2.9 Method of keeping borehole open,

8.2.10 Depth of water surface to the nearest 0.1 ft (0.030 m) and drilling depth to the nearest 0.1 ft (0.030 m) at the time of a noted loss of drilling fluid, and time and date when reading or notation was made,

8.2.11 Location of strata changes, to the nearest 0.5 ft (15 cm),

8.2.12 Size of casing, depth of cased portion of borehole to the nearest 0.1 ft (0.030 m),

8.2.13 Equipment and Method A or B of driving sampler,

8.2.14 Sampler length and inside diameter of barrel, and if a sample basket retainer is used,

8.2.15 Size, type, and section length of the sampling rods, and

8.2.16 Remarks.

8.3 Data obtained for each sample shall be recorded in the field and shall include the following:

8.3.1 Top of sample depth to the nearest 0.1 ft (0.030 m) and, if utilized, the sample number,

8.3.2 Description of soil,

8.3.3 Strata changes within sample,

8.3.4 Sampler penetration and recovery lengths to the nearest 0.1 ft (0.030 m), and

8.3.5 Number of blows per 0.5 foot (0.015 m) or partial increment.

9. Precision and Bias

9.1 *Precision*—Test data on precision is not presented due to the nature of this test method. It is either not feasible or too

costly at this time to have ten or more agencies participate in an in situ testing program at a given site.

9.1.1 The Subcommittee 18.02 is seeking additional data from the users of this test method that might be used to make a limited statement on precision. Present knowledge indicates the following:

9.1.1.1 Variations in *N*-values of 100 % or more have been observed when using different standard penetration test apparatus and drillers for adjacent boreholes in the same soil formation. Current opinion, based on field experience, indicates that when using the same apparatus and driller, *N*-values in the same soil can be reproduced with a coefficient of variation of about 10 %.

9.1.1.2 The use of faulty equipment, such as an extremely massive or damaged anvil, a rusty cathead, a low speed cathead, an old, oily rope, or massive or poorly lubricated rope sheaves can significantly contribute to differences in *N*-values obtained between operator-drill rig systems.

9.2 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

10. Keywords

10.1 blow count; in-situ test; penetration resistance; soil; split-barrel sampling; standard penetration test

APPENDIX

(Nonmandatory Information)

X1. Example Data Sheet

X1.1 See Fig. 4.

DRILLERS BORING LOG

Project _____ Project No: _____ Boring No _____

Location _____ Sheet _____ of _____

Date Started _____ Date Completed _____

Drill Crew: | Boring Location Station _____ Offset _____ | Elevation

Strata Depth		Soil Description and Remarks	Sample	No.	Depth		Recovery	N-Values		
From	To		Type		From	To		6"	6"	6"

Drill Rig Type _____

Method Of Drilling:

Auger _____ Size _____

Wash _____ Water _____ Mud _____

Hammer Type

Auto _____ Manual _____

Split-Spoon Type

Length _____ Liner Used _____

Boring Size _____ Bit Used _____

Casing Size _____ Length _____

Weather _____

Non-Drilling Time (Hrs.)

Boring Layout _____ Moving _____

Hauling Water _____ Standby _____

Water Level @ _____ Date _____ Time _____

@ _____ Date _____ Time _____

@ _____ Date _____ Time _____

Cave-In Depth

@ _____ Date _____ Time _____

FIG. 4 Example Data Sheet

SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (D 1586 – 08) that may impact the use of this standard. (Approved October 1, 2008.)

(*1*) Removed previous Figure 4.
(*2*) Replaced "undisturbed" with "intact" in Sections 5.1 and 6.2.

Committee D18 has identified the location of selected changes to this standard since the last issue (D 1586 – 99) that may impact the use of this standard. (Approved February 1, 2008.)

(*1*) There have been numerous changes to this standard to list them separately. From the most recent main ballot process, additional changes were requested and incorporated into this newest revision. Stated below is a highlight of some of the changes.
(*2*) Scope was completely revised.
(*3*) Referenced Documents updated to include new standards.
(*4*) Terminology: added section on Definitions.
(*5*) Significance and Use: clarified use of the SPT test.
(*6*) Apparatus: general editorial changes.
(*7*) Sampling and Testing Procedure: general editorial changes.
(*8*) Data Sheets/Forms: general editorial changes.
(*9*) Precision and Bias: added Sections 9.1.1.1 and 9.1.1.2.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).

Designation: D 1587 – 08

Standard Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes[1]

This standard is issued under the fixed designation D 1587; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This practice covers a procedure for using a thin-walled metal tube to recover relatively intact soil samples suitable for laboratory tests of engineering properties, such as strength, compressibility, permeability, and density. Thin-walled tubes used in piston, plug, or rotary-type samplers should comply with Section 6.3 of this practice which describes the thin-walled tubes.

NOTE 1—This practice does not apply to liners used within the samplers.

1.2 This Practice is limited to soils that can be penetrated by the thin-walled tube. This sampling method is not recommended for sampling soils containing gravel or larger size soil particles cemented or very hard soils. Other soil samplers may be used for sampling these soil types. Such samplers include driven split barrel samplers and soil coring devices (D 1586, D 3550, and D 6151). For information on appropriate use of other soil samplers refer to D 6169.

1.3 This practice is often used in conjunction with fluid rotary drilling (D 1452, D 5783) or hollow-stem augers (D 6151). Subsurface geotechnical explorations should be reported in accordance with practice (D 5434). This practice discusses some aspects of sample preservation after the sampling event. For information on preservation and transportation process of soil samples, consult Practice D 4220. This practice does not address environmental sampling; consult D 6169 and D 6232 for information on sampling for environmental investigations.

1.4 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.4.1 The tubing tolerances presented in Table 1 are from sources available in North America. Use of metric equivalent is acceptable as long as thickness and proportions are similar to those required in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.6 This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 653 Terminology Relating to Soil, Rock, and Contained Fluids

D 1452 Practice for Soil Investigation and Sampling by Auger Borings

D 1586 Test Method for Penetration Test (SPT) and Split-Barrel Sampling of Soils

D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)

D 3550 Practice for Thick Wall, Ring-Lined, Split Barrel, Drive Sampling of Soils

D 3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D 4220 Practices for Preserving and Transporting Soil Samples

[1] This practice is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.02 on Sampling and Related Field Testing for Soil Evaluations.

Current edition approved Oct. 1, 2008. Published October 2008. Originally approved in 1958. Last previous edition approved in 2007 as D 1587 – 00 (2007)$^{\varepsilon 1}$.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

TABLE 1 Dimensional Tolerances for Thin-Walled Tubes

Size Outside Diameter	Nominal Tube Diameters from Table 2[A] Tolerances					
	2 in.	50.8 mm	3 in.	76.2 mm	5 in.	127 mm
Outside diameter, D_o	+0.007 -0.000	+0.179 -0.000	+0.010 -0.000	+0.254 -0.000	+0.015 -0.000	0.381 -0.000
Inside diameter, D_i	+0.000 -0.007	+0.000 -0.179	+0.000 -0.010	+0.000 -0.254	+0.000 -0.015	+0.000 -0.381
Wall thickness	±0.007	±0.179	±0.010	±0.254	±0.015	±0.381
Ovality	0.015	0.381	0.020	0.508	0.030	0.762
Straightness	0.030/ft	2.50/m	0.030/ft	2.50/m	0.030/ft	2.50/m

[A] Intermediate or larger diameters should be proportional. Specify only two of the first three tolerances; that is, D_o and D_i, or D_o and Wall thickness, or D_i and Wall thickness.

D 5434 Guide for Field Logging of Subsurface Explorations of Soil and Rock

D 5783 Guide for Use of Direct Rotary Drilling with Water-Based Drilling Fluid for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices

D 6151 Practice for Using Hollow-Stem Augers for Geotechnical Exploration and Soil Sampling

D 6169 Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations

D 6232 Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities

3. Terminology

3.1 *Definitions:*

3.1.1 For common definitions of terms in this standard, refer to Terminology D 653.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *inside clearance ratio, %, n*—the ratio of the difference in the inside diameter of the tube, D_i, minus the inside diameter of the cutting edge, D_e, to the inside diameter of the tube, D_i expressed as a percentage (see Fig. 1).

3.2.2 *ovality, n*—the cross section of the tube that deviates from a perfect circle.

4. Summary of Practice

4.1 A relatively intact sample is obtained by pressing a thin-walled metal tube into the in-situ soil at the bottom of a boring, removing the soil-filled tube, and applying seals to the soil surfaces to prevent soil movement and moisture gain or loss.

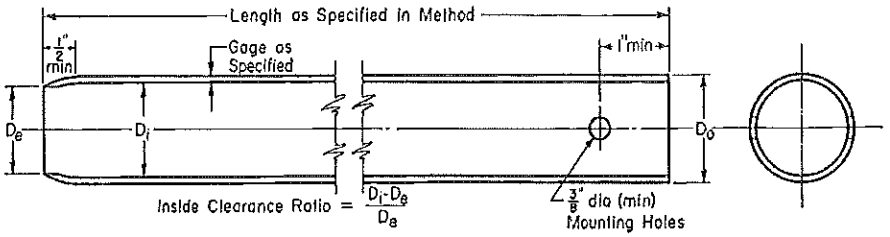
5. Significance and Use

5.1 This practice, or Practice D 3550 with thin wall shoe, is used when it is necessary to obtain a relatively intact specimen suitable for laboratory tests of engineering properties or other tests that might be influenced by soil disturbance.

NOTE 2—The quality of the result produced by this standard is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 are generally considered capable of competent and objective sampling. Users of this practice, are cautioned that compliance with Practice D 3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D 3740 provides a means of evaluating some of those factors.

6. Apparatus

6.1 *Drilling Equipment*—When sampling in a boring, any drilling equipment may be used that provides a reasonably clean hole; that minimizes disturbance of the soil to be sampled; and that does not hinder the penetration of the thin-walled sampler. Open borehole diameter and the inside



NOTE 1—Minimum of two mounting holes on opposite sides for D_o smaller than 4 in. (101.6 mm).
NOTE 2—Minimum of four mounting holes equally spaced for D_o 4 in. (101.6 mm) and larger.
NOTE 3—Tube held with hardened screws or other suitable means.
NOTE 4—2-in (50.8 mm) outside-diameter tubes are specified with an 18-gauge wall thickness to comply with area ratio criteria accepted for "intact samples." Users are advised that such tubing is difficult to locate and can be extremely expensive in small quantities. Sixteen-gauge tubes are generally readily available.

Metric Equivalent Conversions

in.	mm
3/8	9.53
1/2	12.7
1	25.4
2	50.8
3	76.2
4	101.6
5	127

FIG. 1 Thin-Walled Tube for Sampling

TABLE 2 Suitable Thin-Walled Steel Sample Tubes[A]

Outside diameter (D_o):			
in.	2	3	5
mm	50.8	76.2	127
Wall thickness:			
Bwg	18	16	11
in.	0.049	0.065	0.120
mm	1.24	1.65	3.05
Tube length:			
in.	36	36	64
m	0.91	0.91	1.45
Inside clearance ratio, %	<1	<1	<1

[A] The three diameters recommended in Table 2 are indicated for purposes of standardization, and are not intended to indicate that sampling tubes of intermediate or larger diameters are not acceptable. Lengths of tubes shown are illustrative. Proper lengths to be determined as suited to field conditions.

diameter of driven casing or hollow stem auger shall not exceed 3.5 times the outside diameter of the thin-walled tube.

6.2 *Sampler Insertion Equipment*, shall be adequate to provide a relatively rapid continuous penetration force. For hard formations it may be necessary, although not recommended, to drive the thin-walled tube sampler.

6.3 *Thin-Walled Tubes*, should be manufactured to the dimensions as shown in Fig. 1. They should have an outside diameter of 2 to 5 in. (50 to 130 mm) and be made of metal having adequate strength for the type of soil to be sampled. Tubes shall be clean and free of all surface irregularities including projecting weld seams. Other diameters may be used but the tube dimensions should be proportional to the tube designs presented here.

6.3.1 *Length of Tubes*—See Table 2 and 7.4.1.

6.3.2 *Tolerances*, shall be within the limits shown in Table 1.

6.3.3 *Inside Clearance Ratio*, should be not greater than 1 % unless specified otherwise for the type of soil to be sampled. Generally, the inside clearance ratio used should increase with the increase in plasticity of the soil being sampled, except for sensitive soils or where local experience indicates otherwise. See 3.2.1 and Fig. 1 for definition of inside clearance ratio.

6.3.4 *Corrosion Protection*—Corrosion, whether from galvanic or chemical reaction, can damage or destroy both the thin-walled tube and the sample. Severity of damage is a function of time as well as interaction between the sample and the tube. Thin-walled tubes should have some form of protective coating, unless the soil is to be extruded less than 3 days. The type of coating to be used may vary depending upon the material to be sampled. Plating of the tubes or alternate base metals may be specified. Galvanized tubes are often used when long term storage is required. Coatings may include a light coat of lubricating oil, lacquer, epoxy, Teflon, zinc oxide, and others.

NOTE 3—Most coating materials are not resistant to scratching by soils that contain sands. Consideration should be given for prompt testing of the sample because chemical reactions between the metal and the soil sample can occur with time.

6.4 *Sampler Head*, serves to couple the thin-walled tube to the insertion equipment and, together with the thin-walled tube, comprises the thin-walled tube sampler. The sampler head shall contain a venting area and suitable check valve with the venting area to the outside equal to or greater than the area through the check valve. In some special cases, a check valve may not be required but venting is required to avoid sample compression. Attachment of the head to the tube shall be concentric and coaxial to assure uniform application of force to the tube by the sampler insertion equipment.

7. Procedure

7.1 Remove loose material from the center of a casing or hollow stem auger as carefully as possible to avoid disturbance of the material to be sampled. If groundwater is encountered, maintain the liquid level in the borehole at or above ground water level during the drilling and sampling operation.

7.2 Bottom discharge bits are not permitted. Side discharge bits may be used, with caution. Jetting through an open-tube sampler to clean out the borehole to sampling elevation is not permitted.

NOTE 4—Roller bits are available in downward-jetting and diffused-jet configurations. Downward-jetting configuration rock bits are not acceptable. Diffuse-jet configurations are generally acceptable.

7.3 Lower the sampling apparatus so that the sample tube's bottom rests on the bottom of the hole and record depth to the bottom of the sample tube to the nearest 0.1-ft (0.03 m).

7.3.1 Keep the sampling apparatus plumb during lowering, thereby preventing the cutting edge of the tube from scraping the wall of the borehole.

7.4 Advance the sampler without rotation by a continuous relatively rapid downward motion and record length of advancement to the nearest 1 in. (25 mm).

7.4.1 Determine the length of advance by the resistance and condition of the soil formation, but the length shall never exceed 5 to 10 diameters of the tube in sands and 10 to 15 diameters of the tube in clays. In no case shall a length of advance be greater than the sample-tube length minus an allowance for the sampler head and a minimum of 3 in. (76 mm) for sludge and end cuttings.

NOTE 5—The mass of sample, laboratory handling capabilities, transportation problems, and commercial availability of tubes will generally limit maximum practical lengths to those shown in Table 2.

7.5 When the soil formation is too hard for push-type insertion, the tube may be driven or Practice D 3550 may be used. If driving methods are used, the data regarding weight and fall of the hammer and penetration achieved must be shown in the report. Additionally, that tube must be prominently labeled a "driven sample."

7.6 Withdraw the sampler from the soil formation as carefully as possible in order to minimize disturbance of the sample. The tube can be slowly rotated to shear the material at the end of the tube, and to relieve water or suction pressures (or both) and improve recovery. Where the soil formation is soft, a delay before withdraw of the sampler (typically 5 to 30 minutes) may improve sample recovery.

8. Sample Measurement, Sealing and Labeling

8.1 Upon removal of the tube, remove the drill cuttings in the upper end of the tube and measure the length of the soil sample recovered to the nearest 0.25 in. (6 mm) in the tube. Seal the upper end of the tube. Remove at least 1 in. (25 mm)

of material from the lower end of the tube. Use this material for soil description in accordance with Practice D 2488. Measure the overall sample length. Seal the lower end of the tube. Alternatively, after measurement, the tube may be sealed without removal of soil from the ends of the tube.

8.1.1 Tubes sealed over the ends, as opposed to those sealed with expanding packers, should be provided with spacers or appropriate packing materials, or both prior to sealing the tube ends to provide proper confinement. Packing materials must be nonabsorbent and must maintain their properties to provide the same degree of sample support with time.

8.1.2 Depending on the requirements of the investigation, field extrusion and packaging of extruded soil samples can be performed. This allows for physical examination and classification of the sample. Samples are extruded in special hydraulic jacks equipped with properly sized platens to extrude the core in a continuous smooth speed. In some cases, further extrusion may cause sample disturbance reducing suitability for testing of engineering properties. In other cases, if damage is not significant, cores can be extruded and preserved for testing (Practice D 4220). Bent or damaged tubes should be cut off before extruding.

8.2 Prepare and immediately affix labels or apply markings as necessary to identify the sample (see Section 9). Assure that the markings or labels are adequate to survive transportation and storage.

NOTE 6—Top end of the tube should be labeled "top."

9. Field Log

9.1 Record the information that may be required for preparing field logs in general accordance to Guide D 5434. This guide is used for logging explorations by drilling and sampling. Some examples of the information required include;

9.1.1 Name and location of the project,

9.1.2 Boring number,

9.1.3 Log of the soil conditions,

9.1.4 Surface elevation or reference to a datum to the nearest foot (0.5 m) or better,

9.1.5 Location of the boring,

9.1.6 Method of making the borehole,

9.1.7 Name of the drilling foreman and company, and

9.1.8 Name of the drilling inspector(s).

9.1.9 Date and time of boring-start and finish,

9.1.10 Depth to groundwater level: date and time measured,

9.2 Recording the appropriate sampling information is required as follows:

9.2.1 Depth to top of sample to the nearest 0.1 ft. (.03 m) and number of sample,

9.2.2 Description of thin-walled tube sampler: size, type of metal, type of coating,

9.2.3 Method of sampler insertion: push or drive,

9.2.4 Method of drilling, size of hole, casing, and drilling fluid used,

9.2.5 Soil description in accordance with Practice D 2488,

9.2.6 Length of sampler advance (push), and

9.2.7 Recovery: length of sample obtained.

10. Keywords

10.1 geologic investigations; intact; sampling; soil exploration; soil investigations; subsurface investigations

SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (D 1587 – 00 (2007)$^{\varepsilon 1}$) that may impact the use of this standard. (Approved October 1, 2008.)

(*1*) Replaced "undisturbed" with "intact" in Sections 1.1, 4.1, 5.1, 10, and Note 4 of Fig. 1.

(2) Editorial changes made in Sections 1.4, 7.3, 7.4.1, 7.6, 8.1, and 8.1.2.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).

Designation: D4630 – 96 (Reapproved 2008)

Standard Test Method for Determining Transmissivity and Storage Coefficient of Low-Permeability Rocks by In Situ Measurements Using the Constant Head Injection Test[1]

This standard is issued under the fixed designation D4630; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a field procedure for determining the transmissivity and storativity of geological formations having permeabilities lower than $10^{-3}\mu m^2$ (1 millidarcy) using constant head injection.

1.2 The transmissivity and storativity values determined by this test method provide a good approximation of the capacity of the zone of interest to transmit water, if the test intervals are representative of the entire zone and the surrounding rock is fully water-saturated.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 *transmissivity, T*—the transmissivity of a formation of thickness, b, is defined as follows:

$$T = K \cdot b \tag{1}$$

where:
K = hydraulic conductivity.

The hydraulic conductivity, K, is related to the permeability, k, as follows:

$$K = k\rho g/\mu \tag{2}$$

where:
ρ = fluid density,
μ = fluid viscosity, and
g = acceleration due to gravity.

2.1.2 *storage coefficient, S*—the storage coefficient of a formation of thickness, b, is defined as follows:

$$S = S_s \cdot b \tag{3}$$

where:
S_s = specific storage.

The ebrss is the specific storage of a material if it were homogeneous and porous over the entire interval. The specific storage is given as follows:

$$S_s = \rho g\,(C_b + nC_w) \tag{4}$$

where:
C_b = bulk rock compressibility,
C_w = fluid compressibility, and
n = formation porosity.

2.2 *Symbols:*

2.2.1 C_b—bulk rock compressibility ($M^{-1}LT^2$).
2.2.2 C_w—compressibility of water ($M^{-1}LT^2$).
2.2.3 G—dimensionless function.
2.2.4 K—hydraulic conductivity (LT^{-1}).
2.2.4.1 *Discussion*—The use of symbol K for the term hydraulic conductivity is the predominant usage in groundwater literature by hydrogeologists, whereas the symbol k is commonly used for this term in the rock and soil mechanics and soil science literature.
2.2.5 P—excess test hole pressure ($ML^{-1}T^{-2}$).
2.2.6 Q—excess water flow rate (L^3T^{-1}).
2.2.7 Q_o—maximum excess water flow rate (L^3T^{-1}).
2.2.8 S—storativity (or storage coefficient) (dimensionless).
2.2.9 S_s—specific storage (L^{-1}).
2.2.10 T—transmissivity (L^2T^{-1}).
2.2.11 b—formation thickness (L).
2.2.12 e—fracture aperture (L).
2.2.13 g—acceleration due to gravity (LT^{-2}).
2.2.14 k—permeability (L^2).
2.2.15 n—porosity (dimensionless).
2.2.16 r_w—radius of test hole (L).
2.2.17 t—time elapsed from start of test (T).
2.2.18 α—dimensionless parameter.
2.2.19 μ—viscosity of water ($ML^{-1}T^{-1}$).

[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.21 on Groundwater and Vadose Zone Investigations.

Current edition approved Sept. 15, 2008. Published October 2008. Originally approved in 1986. Last previous edition approved in 2002 as D4630 – 96 (2002). DOI: 10.1520/D4630-96R08.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:33:00 MDT

2.2.20 ρ—density of water (ML^{-3}).

3. Summary of Test Method

3.1 A borehole is first drilled into the rock mass, intersecting the geological formations for which the transmissivity and storativity are desired. The borehole is cored through potential zones of interest, and is later subjected to geophysical borehole logging over these intervals. During the test, each interval of interest is packed off at top and bottom with inflatable rubber packers attached to high-pressure steel tubing.

3.2 The test itself involves rapidly applying a constant pressure to the water in the packed-off interval and tubing string, and recording the resulting changes in water flow rate. The water flow rate is measured by one of a series of flow meters of different sensitivities located at the surface. The initial transient water flow rate is dependent on the transmissivity and storativity of the rock surrounding the test interval and on the volume of water contained in the packed-off interval and tubing string.

4. Significance and Use

4.1 *Test Method*—The constant pressure injection test method is used to determine the transmissivity and storativity of low-permeability formations surrounding packed-off intervals. Advantages of the method are: (*1*) it avoids the effect of well-bore storage, (*2*) it may be employed over a wide range of rock mass permeabilities, and (*3*) it is considerably shorter in duration than the conventional pump and slug tests used in more permeable rocks.

4.2 *Analysis*—The transient water flow rate data obtained using the suggested test method are evaluated by the curve-matching technique described by Jacob and Lohman (**1**)[2] and extended to analysis of single fractures by Doe *et al.* (**2**). If the water flow rate attains steady state, it may be used to calculate the transmissivity of the test interval (**3**).

4.3 *Units*:

4.3.1 *Conversions*—The permeability of a formation is often expressed in terms of the unit darcy. A porous medium has a permeability of 1 darcy when a fluid of viscosity 1 cp (1 mPa·s) flows through it at a rate of 1 cm^3/s ($10–6$ m^3/s)/1 cm^2 ($10–4$ m^2) cross-sectional area at a pressure differential of 1 atm (101.4 kPa)/1 cm (10 mm) of length. One darcy corresponds to 0.987 μm^2. For water as the flowing fluid at 20°C, a hydraulic conductivity of 9.66 μm/s corresponds to a permeability of 1 darcy.

5. Apparatus

NOTE 1—A schematic of the test equipment is shown in Fig. 1.

5.1 *Source of Constant Pressure*—A pump or pressure intensifier shall be capable of providing an additional amount of water to the water-filled tubing string and packed-off test interval to produce a constant pressure of up to 1 MPa in magnitude, preferably with a rise time of less than 1 % of one half of the flow rate decay ($Q/Q_o = 0.5$).

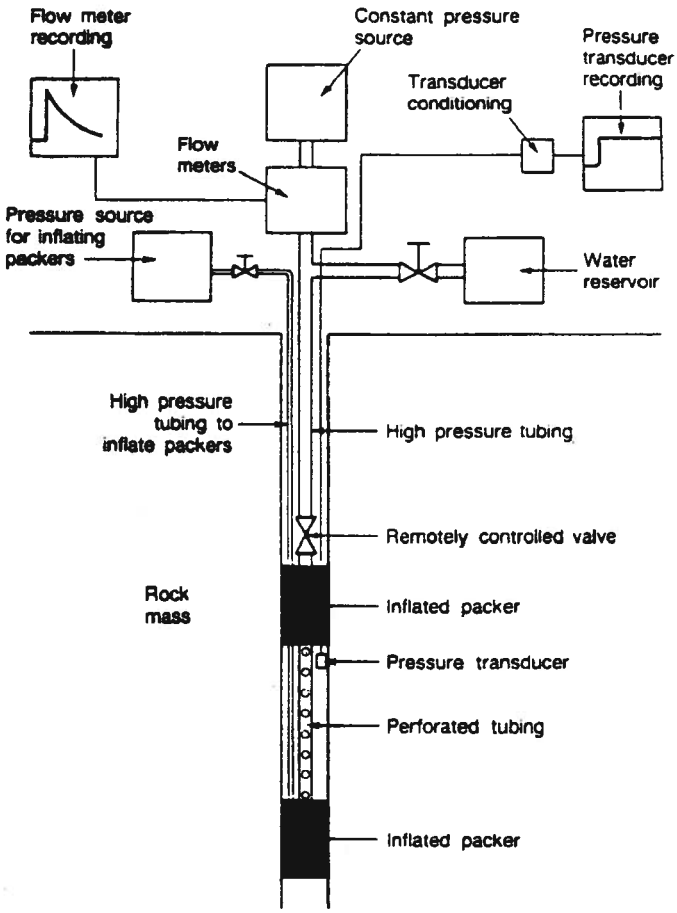


FIG. 1 Equipment Schematic

5.2 *Packers*—Hydraulically actuated packers are recommended because they produce a positive seal on the borehole wall and because of the low compressibility of water they are also comparatively rigid. Each packer shall seal a portion of the borehole wall at least 0.5 m in length, with an applied pressure at least equal to the excess constant pressure to be applied to the packed-off interval and less than the formation fracture pressure at that depth.

5.3 *Pressure Transducers*—The pressure shall be measured as a function of time, with the transducer located in the packed-off test interval. The pressure transducer shall have an accuracy of at least ±3 kPa, including errors introduced by the recording system, and a resolution of at least 1 kPa.

5.4 *Flow Meters*—Suitable flow meters shall be provided for measuring water flow rates in the range from 10^3 cm^3/s to 10^{-3} cm^3/s. Commercially available flow meters are capable of measuring flow rates as low as 10^2 cm^3/s with an accuracy of ±1 % and with a resolution of 10^{-5} cm^3/s; these can test permeabilities to 10^{-3} md based on a 10-m packer spacing. Positive displacement flow meters of either the tank type (Haimson and Doe (**4**) or bubble-type (Wilson, et al (**3**) are capable of measuring flow rates as low as 10^{-3} cm^3/s; these can test permeabilities to 10^{-4} md based on a 10-m packer spacing.

5.5 *Hydraulic Systems*—The inflatable rubber packers shall be attached to high-pressure steel tubing reaching to the surface. The packers themselves shall be inflated with water using a separate hydraulic system. The pump or pressure

[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:33:00 MDT

intensifier providing the constant pressure shall be attached to the steel tubing at the surface. A remotely controlled down-hole valve, located in the steel tubing immediately above the upper packer, shall be used for shutting in the test interval and for instantaneous starting of tests.

6. Procedure

6.1 *Drilling Test Holes*:

6.1.1 *Number and Orientation*—The number of test holes shall be sufficient to supply the detail required by the scope of the project. The test holes shall be directed to intersect major fracture sets, preferably at right angles.

6.1.2 *Test Hole Quality*—The drilling procedure shall provide a borehole sufficiently smooth for packer seating, shall contain no rapid changes in direction, and shall minimize formation damage.

6.1.3 *Test Holes Cored*—Core the test holes through zones of potential interest to provide information for locating test intervals.

6.1.4 *Core Description*—Describe the rock core from the test holes with particular emphasis on the lithology and natural discontinuities.

6.1.5 *Geophysical Borehole Logging*—Log geophysically the zones of potential interest. In particular, run electrical-induction and gamma-gamma density logs. Whenever possible, also use sonic logs and the acoustic televiewer. Run other logs as required.

6.1.6 *Washing Test Holes*—The test holes must not contain any material that could be washed into the permeable zones during testing, thereby changing the transmissivity and storativity. Flush the test holes with clean water until the return is free from cuttings and other dispersed solids.

6.2 *Test Intervals*:

6.2.1 *Selection of Test Intervals*—Determine test intervals from the core descriptions, geophysical borehole logs, and, if necessary, from visual inspection of the borehole with a borescope or TV camera.

6.2.2 *Changes in Lithology*—Test each major change in lithology that can be isolated between packers.

6.2.3 *Sampling Discontinuities*—Discontinuities are often the major permeable features in hard rock. Test jointed zones, fault zones, bedding planes, and the like, both by isolating individual features and by evaluating the combined effects of several features.

6.2.4 *Redundancy of Tests*—To evaluate variability in transmissivity and storativity, conduct three or more tests in each rock type, if homogeneous. If the rock is not homogeneous, the sets of tests should encompass similar types of discontinuities.

6.3 *Test Water*:

6.3.1 *Quality*—Water used for pressure pulse tests shall be clean, and compatible with the formation. Even small amounts of dispersed solids in the injection water could plug the rock face of the test interval and result in a measured transmissivity value that is erroneously low.

6.3.2 *Temperature*—The lower limit of the test water temperature shall be 5°C below that of the rock mass to be tested. Cold water injected into a warm rock mass causes air to come out of solution, and the resulting bubbles will radically modify the pressure transient characteristics.

6.4 *Testing*:

6.4.1 *Filling and Purging System*—Once the packers have been set, slowly fill the tubing string and packed-off interval with water to ensure that no air bubbles will be trapped in the test interval and tubing. Close the downhole valve to shut in the test interval, and allow the test section pressures (as determined from downhole pressure transducer reading) to dissipate.

6.4.2 *Constant Pressure Test*—Pressurize the tubing, typically to between 300 and 600 kPa above the shut-in pressure. This range of pressures is in most cases sufficiently low to minimize distortion of fractures adjacent at the test hole, but in no case should the pressure exceed the minimum principal ground stress. It is necessary to provide sufficient volume of pressurized water to maintain constant pressure during testing. Open the downhole valve, maintain the constant pressure, and record the water flow rate as a function of time. Then close the downhole valve and repeat the test for a higher value of constant test pressure. A typical record is shown in Fig. 2.

7. Calculation and Interpretation of Test Data

7.1 The solution of the differential equation for unsteady state flow from a borehole under constant pressure located in an extensive aquifer is given by Jacob and Lohman (**1**) as:

$$Q = 2\pi TP\, G(\alpha)/\rho g, \tag{5}$$

where:

Q = water flow rate,
T = transmissivity of the test interval,
P = excess test hole pressure,
ρ = water density,
g = acceleration due to gravity, and
$G(\alpha)$ = function of the dimensionless parameter α:

$$\alpha = Tt/Sr_w^2 \tag{6}$$

where:

t = time elapsed from start of test,
S = storativity, and
r_w = radius of the borehole over the test interval.

NOTE 2—For bounded aquifers, the reader is referred to Hantush (**5**).

7.1.1 In Fig. 2, the flow rate in the shut-in, packed-off interval is considered constant. In those cases where the response of the shut-in interval is time dependent, interpretation of the constant pressure test is unaffected, provided the time dependency is linear.

7.2 To determine the transmissivity, T, and storativity, S, data on the water flow rate at constant pressure as a function of time are plotted in the following manner (**1**).

7.2.1 First, plot a type curve log of of the function $G(\alpha)$ versus α where values of $G(\alpha)$ are given in Table 1.

7.2.2 Second, on transparent logarithmic paper to the same scale, plot values of the log of flow rate, Q, versus values of the log of time, t at the same scale as the type curve.

7.2.3 Then, by placing the experimental data over the theoretical curve, the best fit of the data to the curve can be made.

7.2.4 Determine the values of transmissivity, T, and storativity, S, using Eq 5 and Eq 6 from the coordinates of any point in both coordinate systems.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS
Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:33:00 MDT

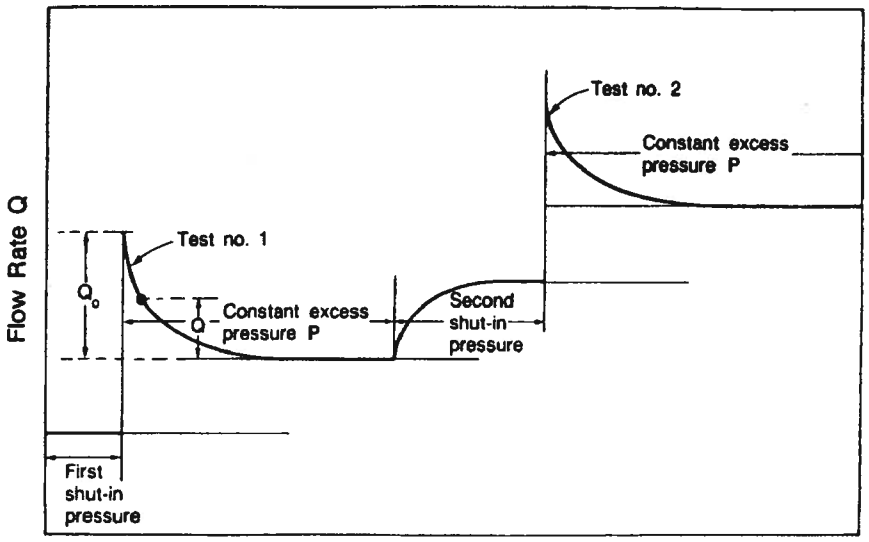


FIG. 2 Typical Flow Rate Record

TABLE 1 Values of G(α) for Values of α Between 10^{-4} and 10^{12} [A]

	10^{-4}	10^{-3}	10^{-2}	10^{-1}	1	10	10^{2}	10^{3}
1	56.9	18.34	6.13	2.249	0.985	0.534	0.346	0.251
2	40.4	13.11	4.47	1.716	0.803	0.461	0.311	0.232
3	33.1	10.79	3.74	1.477	0.719	0.427	0.294	0.222
4	28.7	9.41	3.30	1.333	0.667	0.405	0.283	0.215
5	25.7	8.47	3.00	1.234	0.630	0.389	0.274	0.210
6	23.5	7.77	2.78	1.160	0.602	0.377	0.268	0.206
7	21.8	7.23	2.60	1.103	0.580	0.367	0.263	0.203
8	20.4	6.79	2.46	1.057	0.562	0.359	0.258	0.200
9	19.3	6.43	2.35	1.018	0.547	0.352	0.254	0.198
10	18.3	6.13	2.25	0.985	0.534	0.346	0.251	0.196
	10^{4}	10^{5}	10^{6}	10^{7}	10^{8}	10^{9}	10^{10}	10^{11}
1	0.1964	0.1608	0.1360	0.1177	0.1037	0.0927	0.0838	0.0764
2	0.1841	0.1524	0.1299	0.1131	0.1002	0.0899	0.0814	0.0744
3	0.1777	0.1479	0.1266	0.1106	0.0982	0.0883	0.0801	0.0733
4	0.1733	0.1449	0.1244	0.1089	0.0968	0.0872	0.0792	0.0726
5	0.1701	0.1426	0.1227	0.1076	0.0958	0.0864	0.0785	0.0720
6	0.1675	0.1408	0.1213	0.1066	0.0950	0.0857	0.0779	0.0716
7	0.1654	0.1393	0.1202	0.1057	0.0943	0.0851	0.0774	0.0712
8	0.1636	0.1380	0.1192	0.1049	0.0937	0.0846	0.0770	0.0709
9	0.1621	0.1369	0.1184	0.1043	0.0932	0.0842	0.0767	0.0706
10	0.1608	0.1360	0.1177	0.1037	0.0927	0.0838	0.0764	0.0704

[A] From Jacob and Lohman (1).

8. Report

8.1 The report shall include the following:

8.1.1 *Introduction*—The introductory section is intended to present the scope and purpose of the constant pressure test program, and the characteristics of rock mass tested.

8.1.1.1 *Scope of Testing Program*:

8.1.1.1.1 Report the location and orientation of the boreholes and test intervals. For tests in many boreholes or in a variety of rock types, present the matrix in tabular form.

8.1.1.1.2 Rationale for test location selection, including the reasons for the number, location, and size of test intervals.

8.1.1.1.3 Discuss in general terms limitations of the testing program, stating the areas of interest which are not covered by the testing program and the limitations of the data within the areas of application.

8.1.1.2 *Brief Description of the Test Intervals*—Describe rock type, structure, fabric, grain or crystal size, discontinuities, voids, and weathering of the rock mass in the test intervals. A more detailed description may be needed for certain applications. In a heterogeneous rock mass or for several rock types, many intervals may be described; a tabular presentation is then recommended for clarity.

8.1.2 *Test Method*:

8.1.2.1 *Equipment and Apparatus*—Include a list of the equipment used for the test, the manufacturer's name, model number, and basic specifications for each major item, and the date of last calibration, if applicable.

8.1.2.2 *Procedure*—State the steps actually followed in the procedure for the test.

8.1.2.3 *Variations*—If the actual equipment or procedure deviates from this test method, note each variation and the reasons. Discuss the effects of any deviations upon the test results.

8.1.3 *Theoretical Background*:

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:33:00 MDT

8.1.3.1 *Data Reduction Equations*—Clearly present and fully define all equations and type curves used to reduce the data. Note any assumptions inherent in the equations and type curves and any limitations in their applications and discuss their effects on the results.

8.1.3.2 *Site Specific Influences*—Discuss the degree to which the assumptions contained in the data reduction equations pertain to the actual test location and fully explain any factors or methods applied to the data to correct for departures from the assumptions of the data reduction equations.

8.1.4 *Results*:

8.1.4.1 *Summary Table*—Present a table of results, including the types of rock and discontinuities, the average values of the transmissivity and storativity, and their ranges and uncertainties.

8.1.4.2 *Individual Results*—Present a table of results for individual tests, including test number, interval length, rock types, value of constant pressure transmissivity and storativity, and flow rate as a function of time.

8.1.4.3 *Graphic Data*—Present water flow rate versus time curves for each test, together with the appropriate type curves used for their interpretation.

8.1.4.4 *Other*—Other analyses or presentations may be included as appropriate, for example: (*1*) discussion of the characteristic of the permeable zones, (*2*) histograms of results, and (*3*) comparison of results to other studies or previous work.

8.1.5 *Appended Data*—Include in an appendix a completed data form (Fig. 3) for each test.

9. Precision and Bias

9.1 *Error Estimate*:

9.1.1 Analyze the results using standard statistical methods. Calculate all uncertainties using a 95 % confidence interval.

9.1.2 *Measurement Error*—Evaluate the errors in transmissivity and storativity associated with a single test. This includes the combined effects of flow rate determination, measurement of time, and type curve matching.

9.1.3 *Sample Variability*—For each rock or discontinuity type, calculate, as a minimum, the mean transmissivity and storativity and their ranges, standard deviations, and 95 % confidence limits for the means. Compare the uncertainty associated with the transmissivity and storativity for each rock type with the measurement uncertainty to determine whether measurement error or sample variability is the dominant factor in the results.

10. Keywords

10.1 borehole; constant head testing; faultzones; field testing; flow; flow and flow rate; in situ; permeability; pressure testing; rock; saturation; storativity; transmissivity; viscosity; water; water saturation

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:33:00 MDT

Data Sheet

Project _____ Test No. _____
Test Location _____ Borehole No. _____
Rock Type _____ Borehole Dip and Dip Direction _____
Date _____ Measured Depth of Test to Top Packer, m _____
Testing by _____ Borehole Diameter, mm _____
Rock Temperature, °C _____

Equipment Description	Serial No.	Date of last Calibration

Length of Packed-off Interval, m _____ Packer Pressure, kPa _____
Length of Tubing Above Top Packer, m _____ Tubing ID, mm _____
Water Temperature, °C _____ Shut-in Borehole Pressure, kPa _____
Constant Water Pressure, kPa _____

FIG. 3 Data Sheet for In Situ Measurement of Transmissivity and Storativity Using the Constant Head Injection Test

REFERENCES

(1) Jacob, C. E., and Lohman, S. W., "Non-Steady Flow to a Well of Constant Drawdown in an Extensive Aquifer," *Trans. American Geophys. Union*, Vol 33, 1952, pp. 559–569.

(2) Doe, T. W., Long, J. C. S., Endo, H. K., and Wilson, C. R., "Approaches to Evaluating the Permeability and Porosity of Fractured Rock Masses," *Proceedings of the 23rd U.S. Symposium on Rock Mechanics*, Berkeley, 1982, pp. 30–38.

(3) Wilson, C. R., Doe, T. W., Long, J. C. S., and Witherspoon, P. A., "Permeability Characterization of Nearly Impermeable Rock Masses for Nuclear Waste Repository Siting," *Proceedings*, Workshop on Low Flow, Low Permeability Measurements in Largely Impermeable Rocks, OECD, Paris, 1979, pp. 13–30.

(4) Haimson, B. C., and Doe, T. W., "State of Stress, Permeability, and Fractures in the Precambrian Granite of Northern Illinois," *Journal of Geophysics Research*, Vol 88, 1983, pp. 7355–7371.

(5) Hantush, M. S., "Non-Steady Flow to Flowing Wells in Leaky Aquifers," *Journal of Geophysics Research*, Vol 64, 1959, pp. 1043–1052.

(6) Zeigler, T.,"Determination of Rock Mass Permeability," Tech. Rep. S-76-2, U.S. Army Eng. Waterways Exp. Stn., Vicksburg, MI, 1976, 85 pp.

(7) Earlougher, R. C., "Advances in Well Test Analysis," *Society of Petroleum Engineers of A.I.M.E.*, New York, NY 1977.

(8) Freeze, R. A., and Cherry, J. A., *Groundwater*, Prentice-Hall, Englewood Cliffs, NJ, 1979.

(9) Shuri, F. S., Feves, M. L., Peterson, G. L., Foster, K. M., and Kienle, C. F., Public Draft: "Field and *In Situ* Rock Mechanics Testing Manual," Office of Nuclear Waster Isolation, Document ONWI-310, Section F: "*In Situ* Fluid Properties," GT-F.1 *In Situ* Permeability Measurement of Rock Using Borehole Packers, 1981.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the ASTM website (www.astm.org/COPYRIGHT/).

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:33:00 MDT

Designation: D5876 – 95 (Reapproved 2005)

Standard Guide for Use of Direct Rotary Wireline Casing Advancement Drilling Methods for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices[1]

This standard is issued under the fixed designation D5876; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers how direct (straight) wireline rotary casing advancement drilling and sampling procedures may be used for geoenvironmental exploration and installation of subsurface water-quality monitoring devices.

NOTE 1—The term "direct" with respect to the rotary drilling method of this guide indicates that a water-based drilling fluid or air is injected through a drill-rod column to rotating bit(s) or coring bit. The fluid or air cools the bit(s) and transports cuttings to the surface in the annulus between the drill rod column and the borehole wall.

NOTE 2—This guide does not include all of the procedures for fluid rotary systems which are addressed in a separate guide, Guide D5783.

1.2 The term "casing advancement" is sometimes used to describe rotary wireline drilling because at any time, the center pilot bit or core barrel assemblies may be removed and the large inside diameter drill rods can act as a temporary casing for testing or installation of monitoring devices. This guide addresses casing-advancement equipment in which the drill rod (casing) is advanced by rotary force applied to the bit with application of static downforce to aid in the cutting process.

1.3 This guide includes several forms of rotary wireline drilling configurations. General borehole advancement may be performed without sampling by using a pilot roller cone or drag bit until the desired depth is reached. Alternately, the material may be continuously or incrementally sampled by replacing the pilot bit with a core-barrel assembly designed for coring either rock or soil. Rock coring should be performed in accordance with Practice D2113.

1.4 The values stated in both inch-pound and SI units are to be regarded separately as the standard. The values given in parentheses are for information only.

1.5 Direct rotary wireline drilling methods for geoenvironmental exploration will often involve safety planning, administration, and documentation. This guide does not purport to specifically address exploration and site safety.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This guide offers an organized collection of information or a series of options and does not recommend a specific course of action. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this guide may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D1452 Practice for Soil Exploration and Sampling by Auger Borings

D1586 Test Method for Penetration Test (SPT) and Split-Barrel Sampling of Soils

D1587 Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes

D2113 Practice for Rock Core Drilling and Sampling of Rock for Site Investigation

D3550 Practice for Thick Wall, Ring-Lined, Split Barrel, Drive Sampling of Soils

[1] This guide is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.21 on Groundwater and Vadose Zone Investigations.

Current edition approved Nov. 1, 2005. Published December 2005. Originally approved in 1995. Last previous edition approved in 2000 as D5876 – 95 (2000). DOI: 10.1520/D5876-95R05.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:35:11 MDT

D4630 Test Method for Determining Transmissivity and Storage Coefficient of Low-Permeability Rocks by In Situ Measurements Using the Constant Head Injection Test
D4631 Test Method for Determining Transmissivity and Storativity of Low Permeability Rocks by In Situ Measurements Using Pressure Pulse Technique
D5088 Practice for Decontamination of Field Equipment Used at Waste Sites
D5092 Practice for Design and Installation of Ground Water Monitoring Wells
D5099 Test Methods for Rubber—Measurement of Processing Properties Using Capillary Rheometry
D5782 Guide for Use of Direct Air-Rotary Drilling for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices
D5783 Guide for Use of Direct Rotary Drilling with Water-Based Drilling Fluid for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices

3. Terminology

3.1 *Definitions*—Terminology used within this guide is in accordance with Terminology D653 with the addition of the following:

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *bentonite*—the common name for drilling fluid additives and well-construction products consisting mostly of naturally occurring montmorillonite. Some bentonite products have chemical additives that may affect water-quality analyses.

3.2.2 *bentonite pellets*—roughly spherical- or disk-shaped units of compressed bentonite powder (some pellet manufacturers coat the bentonite with chemicals that may affect the water-quality analysis).

3.2.3 *cleanout depth*—the depth to which the end of the drill string (bit or core barrel cutting end) has reached after an interval of cutting. The cleanout depth (or drilled depth as it is referred to after cleaning out of any sloughed material in the bottom of the borehole) is usually recorded to the nearest 0.1 ft (0.03 m).

3.2.4 *coefficient of uniformity*— $C_u(D)$, the ratio D_{60}/D_{10}, where D_{60} is the particle diameter corresponding to 60 % finer on the cumulative particle-size distribution curve, and D_{10} is the particle diameter corresponding to 10 % finer on the cumulative particle-size distribution curve.

3.2.5 *drill hole*—a cylindrical hole advanced into the subsurface by mechanical means. Also known as a borehole or boring.

3.2.6 *drill string*—the complete rotary drilling assembly under rotation including bit, sampler/core barrel, drill rods, and connector assemblies (subs). The total length of this assembly is used to determine drilling depth by referencing the position of the top of the string to a datum near the ground surface.

3.2.7 *filter pack*—also known as a gravel pack or primary filter pack in the practice of monitoring-well installations. The gravel pack is usually granular material, having selected grain-size characteristics, that is placed between a monitoring device and the borehole wall. The basic purpose of the filter pack or gravel envelope is to act as: a nonclogging filter when the aquifer is not suited to natural development or, as a formation stabilizer when the aquifer is suitable for natural development.

3.2.7.1 *Discussion*—Under most circumstances, a clean, quartz sand or gravel should be used. In some cases, a prepacked screen may be used.

3.2.8 *head space*—on a double- or triple-tube wireline core barrel it is the spacing adjustment made between the pilot-shoe leading edge and the inner kerf of the outer-tube cutting bit. Spacing should be about 1/16 in. or roughly, the thickness of a matchbook. (The head-space adjustment is made by removing the inner-barrel assembly, loosening the lock nut on the hanger-bearing shaft and either tightening or loosening the threaded shaft until the inner barrel is moved the necessary distance, up or down, to obtain the correct setting. Reassemble the inner- and outer-barrel assemblies, attach the barrel to the drill rod or a wireline and suspend vertically allowing the inner-barrel assembly to hang freely inside the outer barrel on the inner hanger-bearing assembly. Check the head space. It is imperative that the adjustment is correct to ensure that the inner barrel is free to rotate without contacting the outer barrel. If incorrectly adjusted, the inner barrel will "hang up" and rotate with the outer barrel as the core is being cut. This will cause the core to break and block entry of core into the inner barrel.)

3.2.9 *grout shoe*—a drillable "plug" containing a check valve that is positioned within the lowermost section of a casing column. Grout is injected through the check valve to fill the annular space between the casing and the borehole wall or another casing.

3.2.9.1 *Discussion*—The composition of the drillable "plug" should be known and documented.

3.2.10 *grout packer*—an inflatable or expandable annular plug that is attached to a tremie pipe, usually positioned immediately above the discharge end of the pipe.

3.2.11 *intermittent sampling devices*—usually barrel-type samplers that are driven or pushed below the bottom of a borehole with drill rods or with a wireline system to lower, drive, and retrieve the sampler following completion of an increment of drilling. The user is referred to the following standards relating to suggested sampling methods and procedures: Practice D1452, Test Method D1586, Practice D3550, and Practice D1587.

3.2.12 *in-situ testing devices*—sensors or probes, used for obtaining mechanical- or chemical-test data, that are typically pushed, rotated, or driven below the bottom of a borehole following completion of an increment of drilling. However, some in-situ testing devices (such as electronic pressure transducers, gas-lift samplers, tensiometers, and so forth) may require lowering and setting of the device(s) in preexisting boreholes by means of a suspension line or a string of lowering rods or pipes. Centralizers may be required to correctly position the device(s) in the borehole.

3.2.13 *lead distance*—the mechanically adjusted length or distance that the inner-barrel cutting shoe is set to extend beyond the outer core-barrel cutting bit in order to minimize possible core-erosion damage that can be caused by the circulating drilling-fluid media. Lead distance is checked by vertically suspending the entire core-barrel assembly from a

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:35:11 MDT

wireline or from a section of drill rod so that the inner-barrel can hang freely from the upper inner-barrel swivel assembly. The cutting shoe extension below the outer core-barrel cutting bit can then be checked. The "stiffer" or more competent the formation to be cored, the less the extension of the inner-barrel cutting shoe is necessary to avoid core erosion.

3.2.14 *overshot*—a latching mechanism located at the end of the hoisting line. It is specially designed to latch onto or release pilot bit or core-barrel assemblies.

3.2.15 *pilot bit assembly*—design to lock into the end section of drill rod for drilling without sampling. The pilot bit can be either drag, roller cone, or diamond plug types. The bit can be set to protrude from the rod coring bit depending on formation conditions.

3.2.16 *sub*—a substitute or adaptor used to connect from one size or type of threaded drill rod or tool connection to another.

3.2.17 *subsurface water-quality monitoring device*— an instrument placed below ground surface to obtain a sample for analyses of the chemical, biological, or radiological characteristics of subsurface pore water or to make in-situ measurements.

3.2.18 *wireline drilling*—a rotary drilling process which uses special enlarged inside diameter drilling rods with special latching pilot bits or core barrels which are raised or lowered inside the rods with a wireline and overshot latching mechanism.

4. Summary of Practice

4.1 Wireline drilling is a rotary drilling process that uses special enlarged inside diameter drilling rods with special latching pilot bits or core barrels which are raised or lowered inside the rods with a wireline and overshot latching mechanism. The bottom section of rod has either a diamond or carbide coring bit at the end and is specially machined to accommodate latching of either pilot bits or core barrels. The overshot mechanism is designed to latch and unlatch bit or barrel assemblies. Bit cutting is accomplished by application of the combination rotary and static down forces to the bit. General drill-hole advancement may be performed without sampling by using either a pilot roller cone or drag bit until the desired depth is reached. Alternately, the material may be continuously or incrementally sampled by replacing the pilot bit with a core-barrel assembly designed for coring either rock or soil.

4.2 The pilot bit or core barrel can be inserted or removed at any time during the drilling process and the large inside diameter rods can act as a temporary casing for testing or installation of monitoring devices.

5. Significance and Use

5.1 Wireline casing advancement may be used in support of geoenvironmental exploration and for installation of subsurface monitoring devices in both unconsolidated and consolidated materials. Use of direct-rotary wireline casing-advancement drilling methods with fluids are applicable to a wide variety of consolidated or unconsolidated materials as long as fluid circulation can be maintained. Wireline casing-advancement drilling offers the advantages of high drilling-penetration rates in a wide variety of materials with the added benefit of the large-diameter drilling rod serving as protective casing. Wireline coring does not require tripping in and out of the hole each time a core is obtained. The drill rods need only be removed when the coring bit is worn or damaged or if the inner core barrel becomes stuck in the outer barrel.

5.1.1 Wireline casing advancers may be adapted for use with circulating air under pressure for sampling water-sensitive materials where fluid exposure may alter the core or in cavernous materials or lost circulation occurs (**1, 2**).[3] Several advantages of using the air-rotary drilling method over other methods may include the ability to drill rather rapidly through consolidated materials and, in many instances, not require the introduction of drilling fluids to the borehole. Air-rotary drilling techniques are usually employed to advance the borehole when water-sensitive materials (that is, friable sandstones or collapsible soils) may preclude use of water-based rotary-drilling methods. Some disadvantages to air-rotary drilling may include poor borehole integrity in unconsolidated materials when casing is not used and the possible volatilization of contaminants and air-borne dust. Air drilling may not be satisfactory in unconsolidated or cohesionless soils, or both, when drilling below the groundwater table. In some instances, water or foam additives, or both, may be injected into the air stream to improve cuttings-lifting capacity and cuttings return. Use of water or other additives, or both, should be documented. The use of air under high pressures may cause fracturing of the formation materials or extreme erosion of the borehole if drilling pressures and techniques are not carefully maintained and monitored. If borehole damage becomes apparent, other drilling method(s) should be considered.

5.1.2 When air is used as the circulating fluid, the user should consult Refs (**1, 2**) and Guide D5782.

5.2 The application of wireline casing advancement to geoenvironmental exploration may involve sampling of groundwater, soil, or rock; or in-situ or pore-fluid testing; or installation of other casings for subsequent drilling activities in unconsolidated or consolidated materials.

5.3 The wireline drill rod can act as a temporary casing and may be used to facilitate the installation of a monitoring device. The monitoring device may be installed as the drill rod is removed from the drill hole.

NOTE 3—The user may install a monitoring device within the same drill hole wherein sampling or in-situ testing was performed.

5.4 Wireline casing-advancement rotary-drilling methods use fluid or air circulation to lubricate cutting bits and for removal of drill cuttings. In many cases, additives are added to improve circulation, cuttings return, borehole wall stabilization, and sealing of the borehole wall from fluid loss. The use of fluid or air under high pressures may allow for damage to formation materials by fracturing or excessive erosion if drilling conditions are not carefully maintained and monitored. If undesirable formation damage is occurring or evident, other drilling method(s) should be considered.

[3] The boldface numbers given in parentheses refer to a list of references at the end of the text.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:35:11 MDT

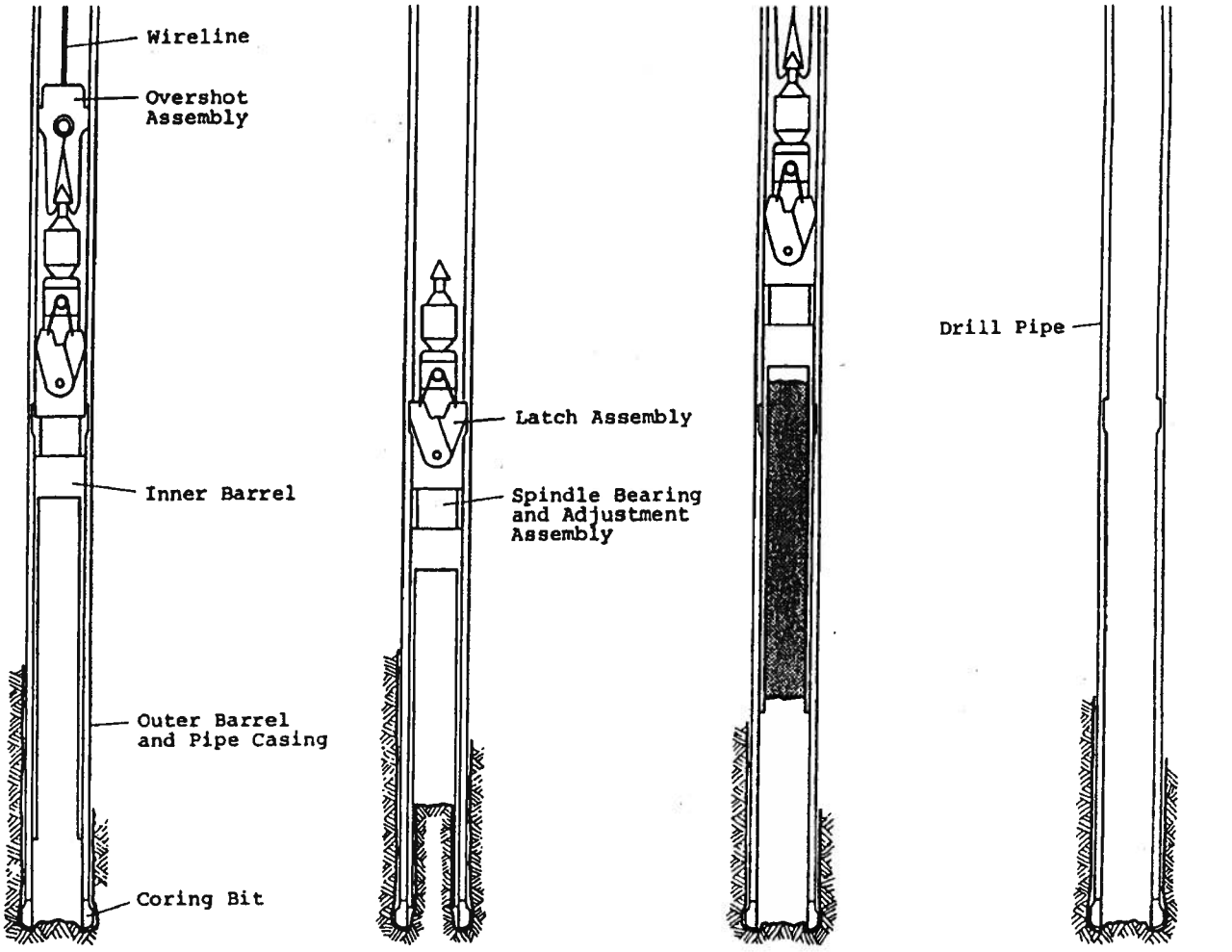
6. Apparatus

6.1 *General*—Direct rotary wireline casing advancement systems and procedures used for geoenvironmental exploration and subsurface water-quality monitoring device installations include direct air or mud-rotary drilling using wireline drill rods. The wireline drill rod has a large inside diameter and is equipped with either a wireline-retrievable center pilot bit for general hole advancement, or a rock- or soil-core barrel for sampling the borehole as it is advanced. Fig. 1(*a* through *d*) shows basic schematics of the components of a wireline-drilling assembly using a rock core-barrel assembly to sample formations as drilling progresses.

6.2 The basic mechanical components of wireline casing-advancement drilling systems include the drill rig with either hollow spindle or top-head drive, drill rods, coring or casing bits, overshot assembly, pilot bit, or core barrel. Water-based fluid circulation systems require drill fluid, mud pit, suction hose, drill fluid circulation pump, pressure hose, and swivel. Air circulation systems require an air compressor, dust collector, air cleaning device, pressure hose, and swivel.

6.2.1 *Drill Rig*—Most top-head drive or hollow-spindle drills are suitable for performing rotary wireline drilling. Rock-coring drills with smooth hydraulic operation and high RPM capability are desirable for rock-coring operations. Rotary table and kelly drills generally are not acceptable for wireline-drilling use due to difficulty or inability to raise and lower wireline assemblies. The drill unit should have the ability to rotate a drill-rod column and apply a controllable axial force on the drill bit appropriate to the drilling and sampling requirements and the geologic conditions.

6.2.2 *Drill Rods*, transfer force and rotation from the drill rig to the bit or core barrel. When rotary drilling is stopped, the large inside diameter wireline drill rod acts can as casing, that is, by preventing against hole collapse—to allow for testing or installation of monitoring devices for hole protection. Drill rods conduct drilling fluid to the bit or core barrel. Individual drill rods (that is, drill stem, drill string, drill pipe) should be straight so they do not contribute to excessive vibrations or "whipping" of the drill-rod column. All threaded connections should be in good repair and not leak significantly at the



a) INNER BARREL BEING LOWERED INTO POSITION

b) OBTAINING CORE, OUTER BARREL ROTATING, INNER BARREL STATIONARY

c) LIFTING INNER BARREL AND CORE OUT OF HOLE

d) OUTER BARREL AND CORING BIT REMAIN IN HOLE

FIG. 1 Schematics of Wire-Line Drilling Assembly

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:35:11 MDT

internal fluid pressure required for drilling. Drill rods should be made up securely by wrench tightening at the threaded joint(s) at all times to prevent rod damage.

6.2.2.1 Wireline drill rod dimensions are not fully standardized. The available sizes depend on the manufacturing sources (**3**). General hole diameter available from most manufacturers follows the Diamond Core Drill Manufacturers Association (DCDMA) size conventions of A (48-mm), B (59.9-mm), N (75.7-mm), H (96.3-mm), and P (122.6-mm) sizes (**4**). Inside diameter varies depending on the manufacturer.

NOTE 4—Sizes of casings, casing bits, drill rods, and core barrels are standardized by the DCDMA and the American Petroleum Institute (API). Refer to Ref (**4**) for available sizes and capacities of common use drilling equipments for soil and rock exploration.

6.2.2.2 The wireline lead rod contains shoulders for latching of pilot bits or core barrels. All wireline lead-rod sections are equipped with coring or casing bits. There are many configurations of wireline drilling equipment possible depending on the manufacturer. With rock-coring systems, the wireline lead rod is equipped with a reaming shell to ensure circulation and act as a stabilizer. Some multipurpose systems allow for latching of pilot bit, rock-core barrel, or soil-core barrel to the lead wireline rod section. In most coring operations the lead wireline rod is considered to be the outer barrel in a double- or triple-tube core barrel design (see Practice D2113). The bit is referred to as a core bit.

6.2.2.3 The wireline rod size is usually selected to provide a drill hole of sufficient diameter for the required sampling, testing, or insertion of instrumentation-device components, such as, the screened intake and filter pack and installation devices such as a tremie pipe. The inside diameter of the wireline rod should permit easy insertion and retraction of a sampler or a pipe with a sufficiently large inside diameter to accommodate the placement of completion materials adjacent to the screened intake and riser of an instrumentation device. Coring bits are selected to provide adequate required hole or core diameter, or both. Selection of protective casings, bits, and core barrels is made by considering size requirements listed above combined with: annulus circulation capabilities of drill rod used, and the need for tapering from larger to smaller diameters casings ("nesting" of casing) if difficult drilling conditions occur (lost circulation, zones of contamination, and so forth) requiring these problem zones in the borehole to be cased off.

NOTE 5—Drill rods usually require lubricants on the threads to allow easy threading and unthreading of the drill-rod tool joints. Some lubricants have organic or metallic constituents, or both, that could be interpreted as contaminants if detected in a sample. Various lubricants are available that have components of known chemistry. The effect of drill-rod lubricants on chemical analyses of samples should be considered and documented when using direct-rotary drilling. The same consideration and documentation should be given to lubricants used with water swivels, hoisting swivels, or other devices used near the drilling axis.

6.2.3 The casing bit or core bit provides the material cutting capability. In coring operations, the bit is referred to as a core bit. Rock coring should be performed in accordance with Practice D2113. Soil sampling or coring methods, some of which are listed in 2.2, can also be used to obtain samples. When drilling in a casing-advancement mode using a pilot bit, without sampling, the bit is referred to as a casing bit.

6.2.3.1 Numerous coring or casing bits can be selected depending on the properties of the formation to be drilled or cored. Since it is undesirable to remove the outer bit, design of this bit is extremely important. When coring, particularly in unconsolidated materials, it is important that the bit cuts the material and not merely tears it and pushes it aside. Some bit types successfully used include either carbide inserts coring bits or diamond core bits. In rock-coring operations the kerf design inner gage of bit, matrix cutting capacity, and location of drilling-fluid circulation ports are important. The inner gage of the bit can be selected so that the core is slightly undercut, thereby allowing it to move freely up the inner tube and not cause core blockage. If the core is over cut and air is present in the barrel, consideration should be given to possible alteration of the core that may occur during subsequent sealing and storage of the core obtained. It is beyond the scope of this guide to recommend bit styles. Bit selection can be aided by review of literature (**1, 2, 3, 5, 6**) and consultation with manufacturers.

6.2.3.2 The dimensions of the coring or casing bits often control the maximum diameter of testing or sampling device that can be inserted through the wireline drill rods. As mentioned previously, the bit size is usually selected to provide a drill hole of sufficient diameter for required borehole sampling or testing to be accomplished or for insertion of instrumentation device components such as the screened intake, riser pipe, filter pack, and well-completion installation devices such as a tremie pipe in the borehole.

6.2.4 *Wireline Retrievable Pilot-Bit Assembly*, used when no borehole coring/sampling is desired. The assembly is equipped with a receiver for pickup by the overshot latching assembly. Several pilot-bit styles are available including roller cone and drag bit configurations. Bit selection can be aided by review of literature (**1, 2, 3, 5, 6**) and consultation with manufacturers.

NOTE 6—Bottom-discharge bits are those having drill-fluid circulation discharge ports in direct contact with the base of the hole. If these bits are used to drill loose cohesionless materials, jetting or excessive erosion of the test intervals could occur.

6.2.5 *Overshot*, a latching retrieval assembly that is lowered into the hole with a wireline hoist cable to either retrieve or lower core-barrel inner assemblies or bits equipped with an upper retrieval spear and downhole latching assemblies.

NOTE 7—When lowering a latching bit assembly or retrievable inner-core barrel assembly into a dry hole, a retrievable dry-hole lowering tool should be employed to prevent damaging the outer bit kerf, matrix, or latching assemblies, or combination thereof. Inner tools should not be allowed to free-fall down the drill rod in a dry hole.

6.2.6 *Wireline Core Barrels*, available for obtaining continuous samples of soil or rock. The barrels for use in coring rock vary in design and manufacture from those barrels used for coring unconsolidated materials. The best core recovery in rock usually requires a double- or triple-tube, swivel-type design. The inner tube of the rock core barrel consists of a core-lifter case and core lifter that threads onto the lower end of the inner tube. On the upper end of the inner tube is a removable threaded inner-tube head swivel-bearing assembly

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:35:11 MDT

with an inner-tube latching device and release mechanism. The inner-tube latching device locks into a complementary recess in the wall of the lead outer drill rod such that the drill rod may be rotated while the inner tube remains stationary. The use of split inner tubes or split inner-tube liners inside a solid inner-tube barrel facilitates easier handling, inspection, and removal of the core from the core barrel.

6.2.6.1 Several types of soil core barrels are also available. Most barrels have a cutting shoe that is either flush with the outer tube cutting bit or, it is made to extend past the outer tube core bit. Sample barrels may be of either the solid- or split-tube configuration. Some barrels may also be equipped with either a split tube or solid tube inner liner to minimize exposure of the core to fluids or other materials. Important considerations for optimum sampling results and maximum core recovery are: use of the "correct" lead distance of the inner barrel cutting shoe, using the "optimum" clearance ratio or head space of the cutting shoe, and prevention of inner-barrel rotation. (For optimum core recovery and minimum core damage the user is referred to 3.2.7 and 3.2.13 for making proper lead-distance or head-space adjustments of the inner-barrel cutting shoe.) The lead distance of the cutting shoe ahead of the cutting bit depends on the stiffness of the formations to be sampled. Stiffer materials require less lead distance.

6.2.6.2 The clearance ratio or head space of the cutting shoe with respect to inner barrel should be selected to result in core that fills the barrel without excessive compression of the core due to friction. If the clearance ratio is too great and the core is over cut, core-erosion damage may occur. If core is over cut and air is present in the barrel, alteration of the soil may occur during subsequent packaging, sealing, and storage of the core(s). Use of single-tube split barrels below the water table may expose soil cores to fluids present in the drill rod.

6.2.7 *Pressure Hose*, conducting the drilling fluid from the circulation pump to the swivel.

6.2.8 *Swivel*, directing the drilling fluid to the rotating kelly or drill-rod column.

6.3 *Rotary Wireline Drilling*, with water-based drilling fluids.

6.3.1 *Mud Pit*, a reservoir for the drilling fluid and, if properly designed and utilized, provides sufficient flow velocity reduction to allow separation of drill cuttings from the fluid before recirculation. The mud pit is usually a shallow, open metal tank with baffles; however, for some circumstances, an excavated pit with some type of liner, designed to prevent loss of drilling fluid and to contain potential contaminants that may be present in the cuttings, may be used. The mud pit can be used as a mixing reservoir for the initial quantity of drilling fluid and, in some circumstances, for adding water and additives to the drilling fluid as drilling progresses.

NOTE 8—Some drilling-fluid components must be added to the composite mixture before other components; consequently, an auxiliary mixing reservoir may be required to premix these components with water before adding to the mud pit. All quantities and types of drilling-fluid components and additives used in the composite drilling-fluid mixture should be documented.

6.3.2 *Suction Hose*, sometimes equipped with a foot valve or strainer, or both, conducts the drilling fluid from the mud pit to the drilling-fluid circulation pump.

6.3.3 *Drilling-Fluid Circulation Pump*, having the capability to lift the drilling fluid from the mud pit and move it through the system against variable pumping heads at a flow rate to provide an annular velocity that is adequate to transport drill cuttings out of the drill hole.

6.3.3.1 Fluid pressures at the bit should be as low as necessary to maintain circulation in order to minimize hydraulic fracturing or excessive erosion of the surrounding materials. Fluid pressures should be monitored during drilling. Normally, injection fluid pressures are readily monitored. Changes in fluid return and circulation pressures may indicate occurrence of excessive erosion, formation fluid loss, or formation fracturing. Any abrupt changes or anomalies in the fluid pressures should be duly noted and documented including the depth(s) of occurrence(s).

6.3.4 *Drilling Fluid*, usually consisting of a water-based circulation media and one or more additives that increase viscosity or provide other desirable physical or chemical properties. Principal functions of drilling fluid include: sealing the drill hole wall to minimize loss of drilling fluid, providing a hydraulic pressure against the drill-hole wall to support the open drill hole, removing cuttings generated at the bit, and lubricating and cooling of the bit. Drilling-fluid management requires considerable experience for successful use. Drilling-fluid program design can be aided by review of literature (**1, 3, 4, 5, 6**) and consultation with manufacturers. If changes to the circulating medium are made, the depth(s) or interval(s) of these changes should be documented. Samples of cuttings can be collected for analysis of materials being penetrated. If samples are taken, the depth(s) and interval(s) should be documented.

NOTE 9—Particular attention should be given to the drilling-fluid makeup-water source and the means used to transport the makeup water to the drilling site as potential sources of contamination in the drilling fluid. If the chemical makeup of the water is determined the test results should be documented.

6.3.4.1 Some commonly used additives for water-based drilling fluids are listed in 6.3.4.2-6.3.4.10.

6.3.4.2 Beneficiated bentonite, a primary viscosifier and borehole sealer, consists of montmorillonite with other naturally occurring minerals and various additives such as sodium carbonate or polyacrylates, or both.

6.3.4.3 Unbeneficiated bentonite, a primary viscosifier and drill hole sealer, consists of montmorillonite with other naturally occurring minerals but without additives such as sodium carbonate or polyacrylates.

6.3.4.4 Sodium carbonate powder (soda ash) is used to precipitate calcium carbonate hardness from the drilling fluid water-base before adding other components. An increase in pH will occur with the addition of sodium carbonate. Sodium hydroxide (caustic soda) generally should not be used in this application.

6.3.4.5 Carboxylmethylcellulose powder (CMC) is sometimes used in a water-based fluid as a viscosifier and as an inhibitor to clay hydration.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:35:11 MDT

NOTE 10—Some additives to water-based drilling-fluid systems retard clay hydration, thus inhibiting swelling of clays on the drill-hole wall and inhibiting "balling" or "smearing" of the bit.

6.3.4.6 Potassium chloride (muriated potash) or diammonium phosphate can be used as an inhibitor to clay hydration.

6.3.4.7 Polyacrylamide, a primary viscosifier and clay-hydration inhibitor, is a polymer that is mixed with water to create a drilling fluid.

6.3.4.8 Barium sulfate increases the density of water-based drilling fluids. It is a naturally occurring high specific gravity mineral processed to a powder for rotary drilling-fluid applications.

6.3.4.9 Lost-circulation materials are used to seal the borehole wall when fluids are being lost through large pores, cracks, or joints. These additives usually consist of various coarse-textured materials such as shredded paper or plastic, bentonite chips, wood fibers, or mica.

6.3.4.10 Attapulgite, a primary viscosifier for rotary drilling in high-salinity environments, is a clay mineral drilling-fluid additive.

NOTE 11—The listing and discussion of the above drilling-fluid additives does not imply general acceptance for geoenvironmental exploration. Some of the additives listed above may impact water-quality analyses. Some readily available, but not as common, drilling-fluid additives, not listed above, could cause significant contamination in a borehole or hydrologic unit. Each additive should be evaluated for each specific application. The types, amounts, and chemical compositions of all additives used should be documented. In addition, a hole log should document the depths where any new additives were introduced. Methods to break revertible fluids should be documented.

If drilling-fluid additives, such as any of those previously mentioned, cannot be tolerated it is recommended that other drilling method(s) be considered by the user.

6.4 *Rotary Wireline Drilling*, using air as the circulation medium:

6.4.1 *Air Compressor*, providing an adequate volume of air, without significant contamination, for removal of cuttings. Air requirements will depend upon the drill rod and bit configuration, the character of the material penetrated, the depth of drilling below groundwater level, and the total depth of drilling. The airflow rate requirements are usually based on an annulus upflow air velocity of about 1000 to 1300 m/min (about 3000 to 4000 ft/min) even though air-upflow rates of less than 1000 m/min are often adequate for cuttings transport. In order to maintain air circulation between the annulus of the hole wall and large-diameter drill rods, special reaming shells may be required (2). For some geologic conditions, air-blast erosion may increase the borehole diameter in easily eroded materials such that 1000 m/min may not be appropriate for cuttings transport. Should air-blast erosion occur, the depth(s) of the occurrence(s) should be noted and documented so that subsequent monitoring equipment installation quality may be evaluated accordingly.

NOTE 12—The quality of compressed air entering the borehole and the quality of air discharged from the borehole and the cyclone separator must be considered. If not adequately filtered, the air produced by most oil-lubricated air compressors inherently introduces a significant quantity of oil into the circulation system. High-efficiency, in-line, air filters are usually required to prevent significant contamination of the borehole.

6.4.2 *Pressure Hose*, conducting the air from the air compressor to the swivel.

6.4.3 *Swivel*, directing the air to the rotating kelly or drill-rod column.

6.4.4 *Dust Collector*, conducting air and cuttings from the borehole annulus past the drill-rod column to an air cleaning device (cyclone separator).

6.4.5 *Air-Cleaning Device*, (cyclone separator) separates cuttings from the air returning from the borehole by means of the dust collector.

NOTE 13—A properly sized cyclone separator can remove practically all of the cuttings from the return air. A small quantity of fine particles, however, are usually discharged to the atmosphere with the "cleaned" air. Some air-cleaning devices consist of a cyclone separator alone; whereas, some utilize a cyclone separator combined with a power blower and sample-collection filters. It is virtually impossible to direct the return "dry" air past the drill rods without some leakage of air and return cuttings. Samples of drill cuttings can be collected for analysis of materials penetrated. If samples are obtained, the depth(s) and interval(s) should be documented.

NOTE 14—Zones of low air return also zones of no air return should be documented. Likewise, the depth(s) of sampled interval(s) and quality of samples obtained should be documented.

NOTE 15—Compressed air alone can often transport cuttings from the borehole and cool the bit. For some geologic conditions, injection of water into the air stream will help control dust or break down "mud rings" that tend to form on the drill rods. If water is injected the depth(s) of water injection should be documented. Under other circumstances, for example, if the borehole starts to produce water, the injection of a foaming agent may be required. The depth when a foaming agent is added should also be recorded. When foaming agents are used, a cyclone-type cuttings separator is not used and foam discharge accumulates near the top of the borehole. When contaminants are encountered during drilling and returning from the borehole at geoenvironmental-exploration sites, special measures should be taken to contain the foam and protect personnel and the environment. Therefore, added water and some available foaming agents could affect water-quality analyses. The need for chemical analysis of added water or foaming agents should be considered and documented.

7. Drilling Procedures

7.1 As a prelude to and throughout the drilling process, stabilize the drill rig and raise the drill-rig mast. Surface casings can be installed using a variety of drilling methods. The surface casing is normally backfilled, pressed, or sealed in place with bentonite or cement, or both. Establish and document a datum for measuring hole depth. This datum normally consists of a stake driven into the stable ground surface, the top of the surface casing, or the drilling deck. If there is a possibility for movement of the surface casing it should not be used as a datum. If the hole is to be later surveyed for elevation, record and report the height of the datum to the ground surface.

7.1.1 For water-based fluid drilling operation, position a mud pit to collect and filter fluid return flow. Mix an initial quantity of drilling fluid, usually using the mud pit as the primary mixing reservoir.

NOTE 16—The need for chemical analysis of samples of each drilling-fluid component and the final mixture should be documented.

7.1.2 For air-based circulation systems, position the dust collector or cyclone separator and "seal" to the ground surface. If air-monitoring operations are performed, consider the prevalent wind direction relative to the exhaust from the drill rig.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:35:11 MDT

Also, consider the location of the cyclone relative to the rig exhaust since air-quality monitoring will be performed at the cyclone separator discharge point.

NOTE 17—Deeper casing(s) or nested casing(s) may be required to facilitate adequate downhole fluid circulation and hole control. Records of casing(s) lengths and depth intervals installed should be maintained and documented if required. All casing used should be decontaminated according to Practice D5088 prior to use.

NOTE 18—Check above the drilling rig for overhead obstructions or hazards, such as power lines prior to raising the mast. In most cases, it is required to perform a survey of underground and all other utilities prior to drilling to evaluate possible hazards.

7.2 Drilling usually progresses as follows:

7.2.1 Attach an initial assembly of lead drill rod and a bit or core barrel below the top-head drive unit with the bit or drill head placed within the top of the surface casing. Record hole depth by knowing the length of the rod-bit assemblies and comparing its position relative to the established surface datum.

NOTE 19—The drill rig, drilling, hoisting and sampling tools, the rotary gear or chain case, the spindle and all components of the rotary drive above the drilling axis should be cleaned and decontaminated according to Practice D5088 prior to commencing drilling and sampling operations.

7.2.2 Activate the drilling-fluid circulation pump, causing drilling fluid to circulate through the system including the mud pit.

7.2.3 Initiate rotation of the bit and apply axial force to the bit.

7.2.4 Initiate drilling fluid or air circulation and apply rotation and axial force to the bit until drilling progresses to a depth where sampling or in-situ testing will be performed, the length of the drill-rod column limits further penetration, or (when core drilling) the core specimen has fully entered the core barrel or blockage is apparent. Monitor downfeed pressures, fluid/gas pressure, and circulations return during drilling. Observe the ease of drilling during drilling, and drill cuttings as they relate to the geologic strata being penetrated. Document occurrences of any significant abrupt changes and anomalies that occur during drilling.

7.2.4.1 During air drilling operations air-quality monitoring may be required. If air-quality monitoring is performed, document the sampled intervals and air-quality data.

NOTE 20—If circulation is lost and input fluid pressure is allowed to increase, the possibility of fracturing materials and excessive drill hole erosion exists.

NOTE 21—The time required to remove the cuttings from the drill hole will depend mainly upon the pumping rate, the cross-sectional area of the borehole annulus, the borehole depth, the viscosity of the drilling fluid, and the size of the cuttings.

7.2.5 Stop rotation, lift the bit slightly off the bottom of the hole to facilitate drill-cuttings removal, and continue circulation for a short time until the drill cuttings are removed from the borehole annulus. If sampling is to be done, stop circulation and rest the bit on the hole bottom to determine hole depth. Document the hole depth and amount of any caving that occurred.

7.2.6 Increase drilling depth by attaching an additional drill-rod section to the top of the previously advanced drill-rod column and resuming drilling operations according to 7.2.2-7.2.4.

NOTE 22—Drilling rates depend on many factors such as the density or stiffness of unconsolidated material and the existence of cobbles or boulders, the hardness or durability of the rock, or both, the swelling activity of clays or shales encountered in the borehole, and the erosiveness of the wall. Drilling rates can vary from a few mm/min (less than an in./min) to about a m/min (3 ft/min), depending on subsurface conditions. Other factors influencing drilling rates include the weight of the drill string, collar(s) weight and size of drill pipe, and the rig pulldown or holdback pressure. These data as well as any other drilling-rate information should be recorded.

7.2.7 In some cases it may be necessary to remove the pilot bit or core barrel during the drilling process. Remove core barrels when full or there is evidence of core blockage and loss of circulation. Remove pilot bits for sampling events, when worn, or when there is evidence of circulation blockage. When replacing the pilot bit or core barrel note the condition of the base of the boring and any difficulty latching the assembly. If heave is present it may be necessary to lift the drill rod to engage the locking mechanism. When drilling in unconsolidated materials it is recommended to use a vented hoisting plug when removing the inner bit or continued circulation to avoid "heaving," "piping," or "sanding in" problems.

7.2.8 In some cases it may be necessary to remove the drill-rod string to change the lead-rod bit. If the string is removed and replaced, check the depth to the base of the boring where the end of the string rests and compare to the cleanout depth to evaluate hole quality. If excessive hole cave or erosion is suspected, casing may be required to protect the borehole wall. As the drilling progresses, note and document drilling procedures such as circulation rates or losses, intervals where equipment is changed, intervals where casing is installed, or drilling method is changed.

7.2.9 *Continuous Sampling with Rock- or Soil-Core Barrels*—Continuous coring can be performed by using core barrels designed for rock or soil coring. Rock coring is performed in accordance with Practice D2113. Soil coring follows similar procedures. When replacing the core barrel, note the elevation of the base of the boring. Record the sampling length and measure core recovery. If blockage of core and circulation loss is noted during the sampling process, the sample should be recovered.

7.2.10 Sampling or in-situ testing can be performed at any depth by interrupting the advance of the bit, stopping the fluid circulation (after cleaning the annulus of cuttings) and removing pilot bit from the drill-rod column. If there are apparent cuttings or evidence of caving, these should be noted as part of the sample taken. Drill-rod removal is not necessary when a sample can be obtained or an in-situ test can be performed through the hollow axis of the drill rods and bit.

7.2.10.1 Some sampling method may be performed through the drill-rod column (see Test Method D1586, Practice D1587, Practice D2113, and Practice D3550). Compare sampling depth to the cleanout depth. Verify the depth-comparison data by first resting the sampler on the bottom of the hole and

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:35:11 MDT

comparing that measurement with the cleanout-depth measurement. This should be done before every sampling or in-situ testing is performed in the hole. If bottom-hole sloughing is apparent from a depth measurement made prior to sampling (determined by comparing the hole-cleanout depth with the sampling depth) it may be necessary to reclean the hole by rotary recirculation. Record the depth of in-situ testing or sampling as well as the depth below the sampler/bit for evaluation of data quality. Decontaminate sampling and testing devices according to Practice D5088 prior to testing.

7.2.10.2 Perform in-situ testing at the base of the drill hole or at shallower depths if the hole is uncased. If in-situ testing is performed at the base of the borehole similar depth, perform checks to accurately document the location of the test.

7.2.10.3 Water testing is frequently performed in consolidated deposits by pulling back on the drill rods and passing inflatable packer(s) with pressure fittings for water injection through the drill rod to test the open borehole wall (see Test Methods D4630 and D4631). Record the depths or intervals of water tests performed.

7.2.10.4 Air drilling may not be satisfactory in unconsolidated or cohesionless soils, or both, under the groundwater table. One problem is "heaving" or "sanding in" of sands during pilot-bit removal. Another problem with wet clays is bit plugging and formation of mud rings which may cause loss of circulation. If the purpose of the drilling is to perform in-situ tests or undisturbed sampling, and soil instability is evident from unbalanced hydrostatic pressure or pilot-bit disturbance, give consideration to using fluids or other casing-advancement methods where disturbance may be more readily controlled.

7.3 When drilling must progress through material suspected of being contaminated, installation of single or multiple (nested) casings may be required to isolate zones of suspected contamination. Isolation casings are usually installed in a predrilled borehole or by using a casing-advancement method. A grout seal is then installed, usually by applying the grout at the bottom of the annulus with the aid of a grout shoe or a grout packer and a tremie pipe. Allow the grout to set before drilling activities are continued. Document complete casing and grouting records, including location(s) of nested casings for the hole.

8. Installation of Monitoring Devices

8.1 Subsurface water-quality monitoring devices are generally installed in boreholes drilled by wireline casing-advancement methods using a three-step procedure. The three steps are: (*1*) drilling, with or without sampling, (*2*) removal of the pilot bit or core barrel and placement of the monitoring device, and (*3*) addition of other materials such as filter packs, seals, and grouts as the drill rods are removed. During placement of filter packs and seals the drill rods may be lifted in increments to be backfilled. Place tremie rods between riser and drill-rod wall to extend past the end of the drill rods for placement increments. The retraction increments depend on the anticipated borehole wall stability. Alternately, if borehole conditions are stable the complete drill-rod column may be removed prior to placement of the monitoring device.

NOTE 23—Document the volumes of emplaced filter packs and seals and compare the results to actual volumes of cuttings return based on hole diameter for evaluation of hole quality.

8.2 Assemble water-quality monitoring devices, with attached fluid conductors (risers), and insert into the borehole with the least possible addition of contaminants. The user is referred to Practice D5092 for monitoring-well installation methods and Practice D5088 for suggested methods of field equipment decontamination.

NOTE 24—If the integrity of the borehole wall will not be compromised by removing the filter cake from the borehole in the vicinity of the screened-intake device the drilling mud should be removed using a well-development procedure as suggested in Practice D5099 prior to inserting a monitoring well or a water-quality monitoring device in the borehole.

8.2.1 Some materials, such as screens and risers, require cleaning or decontamination, or both, at the job site (see Practice D5088).

8.2.2 Prior to installation, store all monitoring-device materials undercover and place upwind and well away from the drill rig and any other sources of contamination such as electrical generators, air compressors, or industrial machinery.

8.2.3 Clean and decontaminate hoisting tools, particularly wire rope and hoisting swivels, according to Practice D5088 before using.

8.3 Select and install filter materials, bentonite pellets, granules and chips and grouts according to specific subsurface-monitoring requirements. Document this information.

NOTE 25—Filter packs for monitoring devices are usually installed in wireline casing-advancement drill holes using a tremie pipe inserted in the annulus formed between the drill rod or borehole wall (when drill rods are removed) and the monitoring device. The minimum annulus between riser pipe and drill rod or borehole wall should be about 1-in. (25.4 mm) completely around the riser pipe. The sizes of wireline drill rod are limited and the riser size may have to be reduced to accommodate placement clearances (see 6.2.2).

8.3.1 Monitoring devices installed in a saturated zone typically have sand-sized filter packs selected on the basis of the grain-size characteristics of the hydrologic unit adjacent to the screened intake. Filter-pack materials are usually selected with a coefficient of uniformity of less than 2.5. Filter packs for vadose-zone monitoring devices may be predominantly slit sized (however, soil-gas monitoring devices should not use silt-sized filter packs but typically use coarse sand or gravel filter packs). These filter materials are often mixed with water of known quality and then inserted through a tremie pipe and tamped into place around the device. The type(s) and volumes of filter materials used and the quality and quantities of mixing water should be documented. In most cases, a centralizer should be used to position the monitoring device in the borehole. The intake device and riser(s) should be suspended above the bottom of the borehole during installation of the filter pack(s), seal(s), and backfill to keep the riser(s) as straight as possible. Care should be taken when adding backfill or filter material(s), or both, so that the materials do not bridge. However, if bridging does occur during the installation procedure, tamping rods or other tamping devices may be used to dislodge the "bridge".

8.4 Place sealing materials, consisting of either bentonite pellets, chips, or granules, directly above the filter pack.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:35:11 MDT

NOTE 26—It may be effective, when granular filter packs are used, to install a thin, fine sand, secondary filter either below the annular seal or both above and below the seal. These secondary filters protect both the monitoring-device filter and the seal from intrusion of grout installed above the seal.

8.5 The backfill that is placed above the annular seal is usually a bentonite or cement-base grout.

NOTE 27—Grouts should be designed and installed in consideration of the ambient hydrogeologic conditions. The constituents should be selected according to specific performance requirements and these data documented. Typical grout mixtures are given in Practice D5092 and Test Method D4630.

8.5.1 In most cases, the grout should be pumped into the annulus formed between the borehole wall and the monitoring device(s) riser(s) using a tremie pipe.

NOTE 28—Grouting equipment should be cleaned and decontaminated prior to use according to Practice D5088. Also, the equipment used for grouting should be constructed from materials that do not "leach" significant amounts of contaminants to the grout.

8.5.2 The initial position of the tremie pipe and grouting pressures should be controlled to prevent materials from being jetted into underlying seal(s) and filter(s) (use of a tremie pipe having a plugged bottom and side-discharge ports should be considered to minimize bottom-jetting problems).

8.5.3 In most cases, the grout should be discharged at a depth of approximately 1.5 to 3 m (5 to 10 ft) below the grout surface within the annulus (after the placement of the initial 1.5 to 3 m (5 to 10 ft) of grout above the underlying filter or seal). Additional grout should be discharged at a depth of approximately 1.5 to 3 m (5 to 10 ft) below the grout surface within the annulus. The tremie pipe should be periodically raised as grout is discharged to maintain the appropriate depth below the grout surface.

NOTE 29—The need for chemical analysis of samples of each grout component and chemical analysis of the final mixture should be documented. Also, it should be noted that if cements are used for grouting, they generate hydroxides and thereby, can cause a localized increase in the alkalinity and pH of the surrounding groundwater.

8.5.4 Install the grout from the bottom of the borehole to the top of the borehole so as to displace fluids in the borehole.

9. Development

9.1 Most monitoring-device installations should be developed to remove suspended solids from drilling fluids and disturbance of geologic materials during installation and to improve the hydraulic characteristics of the filter pack and the geologic unit adjacent to the monitoring-device intake. The method(s) selected and time expended to develop the installation and the changes in water quality discharged at the surface should be carefully observed and documented. For suggested well-development methods and techniques the user is referred to Practice D5099.

NOTE 30—Under most circumstances, development should be initiated as soon as possible following completion, however, time should be allowed for setting of grout.

10. Field Report and Project Control

10.1 The field report should include all information identified as necessary and pertinent to the needs of the exploration program. Information normally required for the project include exploration type and execution, drilling equipment and methods used, and subsurface conditions encountered including: groundwater conditions, sampling events, and installations.

10.2 Additional information should be considered as follows:

10.2.1 *Drilling Methods*:

10.2.1.1 Description of the wireline casing-advancer system including type, sizes, pilot bits, core barrels, fluid pump, fluid circulation, and discharge systems. Note and document intervals of equipment change or drilling method changes and reasons for change.

10.2.1.2 Type, quantities, and locations in the borehole of use of additives such as water or foaming agent(s) added to the circulation media.

10.2.1.3 Descriptions of circulation rates and cuttings return, including quantities, over intervals used. Locations and probable cause of loss of circulation in the borehole.

10.2.1.4 Descriptions of drilling conditions related to drilling pressures, rotation rates, and general ease of drilling related to subsurface materials encountered.

10.2.2 *Sampling*—Document conditions of the bottom of the borehole at the base prior to sampling, and report any slough or cuttings present in the recovered sample.

10.2.2.1 Samples of fluid circulation cuttings can be collected for analysis of materials being penetrated. If samples are taken, the depth(s) and interval(s) should be documented.

10.2.3 *In-Situ Testing*:

10.2.3.1 For devices inserted below the bottom of the borehole, document the depths below the bottom of the hole and any unusual conditions during testing.

10.2.3.2 For devices testing or seating at the borehole wall, report any unusual conditions of the borehole wall such as inability to seat borehole packers.

10.2.4 *Installations*—A description of well-completion materials and placement methods, approximate volumes placed, depth-intervals of placement, methods of confirming placement, and areas of difficulty of material placement or unusual occurrences.

11. Keywords

11.1 drilling; geoenvironmental exploration; groundwater; vadose zone; wireline drilling method

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:35:11 MDT

REFERENCES

(1) Shuter, E., and Teasdale, W. E., "Application of Drilling, Coring, and Sampling Techniques to Test Holes and Wells," *U.S. Geological Survey Techniques of Water-Resource Investigations, TWRI 2-F1*, 1989.

(2) Teasdale, W. E., and Pemberton, R. R.," Wireline-Rotary Air Coring of Bandeiler Tuff, Los Alamos, New Mexico," *U.S. Geological Survey, Water-Resources Investigations Report 84-4176*, 1984.

(3) *Diamond Drilling Handbook*, Heinz, W. F., First Edition, South African Drilling Association, Johannesburg, Republic of South Africa, 1985.

(4) *DCDMA Technical Manual*, Drilling Equipment Manufacturers Association, 3008 Millwood Ave., Columbia, SC 29205, 1991.

(5) O'Rourke, J. E., Gibbs, H. J., and O'Connor, K. O., "Core Recovery Techniques for Soft or Poorly Consolidated Materials," *Final Report, Contract No. J0275003*, U.S. Bureau of Mines, Department of Interior, Washington, DC, April 28, 1978.

(6) *Drillers Handbook*, Ruda, T. C., and Bosscher, P. J., editors, National Drilling Contractors Association, 3008 Millwood Ave., Columbia, SC 29205, June 1990.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the ASTM website (www.astm.org/COPYRIGHT/).

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:35:11 MDT

Designation: D 5079 – 08

Standard Practices for Preserving and Transporting Rock Core Samples[1]

This standard is issued under the fixed designation D 5079; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 These practices cover the preservation, transportation, storage, cataloging, retrieval, and post-test disposition of rock core samples obtained for testing purposes and geologic study.

1.2 These practices apply to both hard and soft rock, but exclude ice and permafrost.

1.3 These practices do not apply to those situations in which changes in volatile gas components, contamination of the pore fluids, or mechanical stress relaxation affect the intended use for the core.

1.4 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.5 *This practice offers a set of instruction for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgement. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

1.6 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 420 Guide to Site Characterization for Engineering Design and Construction Purposes

D 653 Terminology Relating to Soil, Rock, and Contained Fluids

D 2113 Practice for Rock Core Drilling and Sampling of Rock for Site Investigation

D 4220 Practices for Preserving and Transporting Soil Samples

2.2 *API Standard:*

API RP-40 Recommended Practice for Core Analysis Procedure[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:* See Terminology D 653 for general definitions.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *critical care*—samples which are fragile or fluid or temperature sensitive. This protection level includes the requirements prescribed for routine and special care.

3.2.2 *routine care*—non-sensitive, non-fragile samples for which only general visual identification is necessary, and samples which will not change or deteriorate before laboratory testing.

3.2.3 *soil-like care*—materials which are so poorly consolidated that soil sampling procedures must be employed to obtain intact pieces of core.

3.2.4 *special care*—fluid sensitive samples and those which must later be subjected to testing. Requirements for this level of protection include those prescribed for routine care.

4. Significance and Use

4.1 The geologic characteristics and the intended use of the rock core samples determine the extent and type of preservation required. If engineering properties are to be determined for the core, it must be handled and preserved in such a way that the measured properties are not significantly influenced by mechanical damage, changes in chemistry, and environmental conditions of moisture and temperature, from the time that the core is recovered from the core drill until testing is performed. Drill core is also the sample record for the subsurface geology at the borehole location, and as such must be preserved for some period of time, in some cases indefinitely, for future geologic study.

[1] These practices are under the jurisdiction of ASTM Committee D18 on Soil and Rock and are the direct responsibility of Subcommittee D18.12 on Rock Mechanics.

Current edition approved July 1, 2008. Published July 2008. Originally approved in 1990. Last previous edition approved in 2006 as D 5079 – 02 (2006).

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, http://www.api.org.

***A Summary of Changes section appears at the end of this standard.**

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:39:00 MDT

4.2 These practices present a selection of curatorial requirements which apply to the majority of projects. The requirements are given for a variety of rock types and project types ranging from small to large and from noncritical to critical. Noncritical projects are those in which failure of an element or the structure would result in negligible risk of injury and property loss, while there is great risk to property and life after failure of critical structures and projects. Guidance is given for the selection of those specific requirements which should be followed for a given project.

5. Guide for Implementation

5.1 A qualified person shall be assigned to have curatorial management responsibility for a given project. This person shall be technically competent in the management of rock core samples and shall have a knowledge of the various end uses for the cores and their associated preservation requirements. This responsible person shall have the authority to implement the requirements selected from these practices. In some cases, he or she may also have to decide between competing uses for the same core.

5.2 The responsible person shall select from Sections 6-11 those requirements and procedures that should be applied for the core from a particular project. The curatorial manager shall then see that these procedures are implemented, and also see that the records specified in Section 12 are kept.

5.3 The following factors should be considered when selecting the curatorial requirements from Sections 6-11:

5.3.1 Project requirements for use of the core range from simple ones, in which the only need is to identify and locate the various lithologic units, to complex and critical ones in which detailed property testing of the core is required for engineering design. Priorities for multiple uses or different types of tests must sometimes be established when available core lengths are limited and when one use or test precludes another. For example, splitting a core for detailed geologic study prevents later strength testing, which requires an intact core.

5.3.2 Mechanical property tests for structural design purposes should be performed on a core in its natural moisture state, particularly if the rocks are argillaceous. Irreversible changes occur when such rocks are allowed to dry out, often resulting in invalid design data. The initial moisture content of such a core should therefore be preserved.

5.3.3 Freezing of pore water in the core may reduce the strength of the rock. The high temperature associated with unventilated storage sheds in summer, and temperatures alternating between hot and cold, may cause moisture migration from the core and weakening of the rock due to differential thermal expansion and contraction between grains. Such temperature extremes should therefore be avoided, particularly for weak sedimentary rock types.

5.3.4 A weak rock core may be broken or further weakened by careless handling, such as dropping a core box, or by mechanical vibration and shock during transportation. Breaking of the core reduces sample lengths available for testing. Weakening caused by such mechanical stressing may lower measured strength parameters and may affect other properties.

5.3.5 The required preservation time may vary from as short as three months to several years, and sometimes core may need to be stored indefinitely. A core taken simply to identify the bedrock lithology beneath a small structure may be needed for a few months only. For large and critical structures, it may be necessary to retain the core for many years as re-examination and testing may be required at some later time for additional geologic study or re-evaluation of property data. Some states have regulations governing the disposition and storage of core obtained within the state.

5.4 Fig. 1 is a flow chart that shows the various core handling, use, and storage activities and the corresponding section numbers in these practices. Note that four care or protection levels are defined in Section 3 to account for the great variety of rock sensitivities and core uses encountered in practice.

5.5 The person assigned curatorial management responsibility should study the flow chart in Fig. 1 as it relates to the designated Sections 6-11 in these practices. Note in particular, that a selection of the required protection must be made in 7.5, where four levels of protection are specified, namely routine care, special care, critical care, and soil-like care.

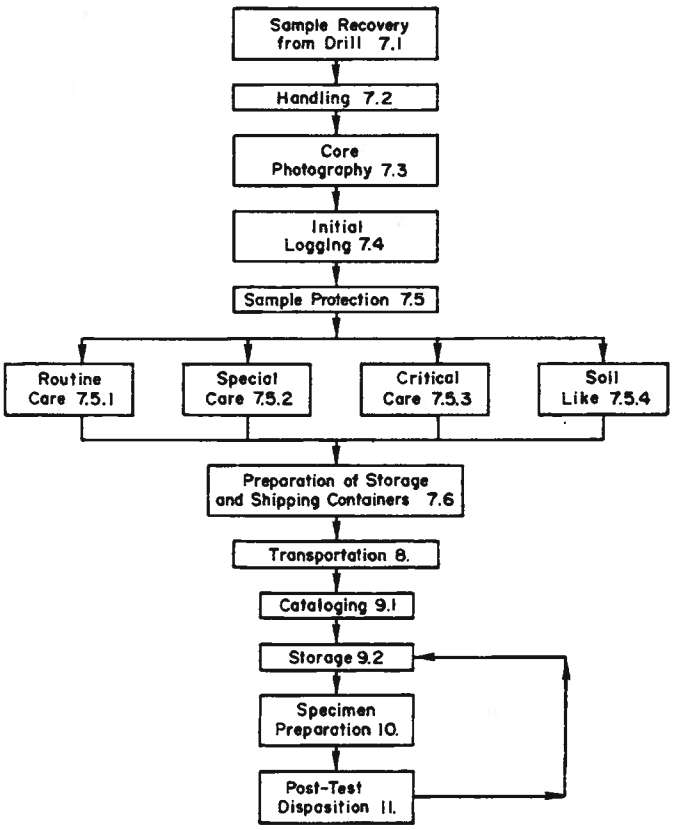
5.6 Special attention is also directed to records requirements in Section 12, that document the history of the core handling, preservation, and storage.

6. Apparatus

6.1 *Camera*, for taking photographs of cores for logging.

6.2 *Controlled Humidity Room.*

6.3 *Core Boxes*—See 7.6.1.



NOTE 1—Numbers refer to corresponding sections of this practice.

FIG. 1 Flow Chart for Core Handling, Use, and Storage Activities

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:39:00 MDT

6.4 *Vinylidene Chloride Plastic Film, Aluminum Foil, Plastic Microcrystalline Wax*, for sealing in moisture content of cores.

6.5 *Polyethylene Layflat Plastic Tubing.*

6.6 *Poly(vinyl chloride) Tubing.*

6.7 *Sawdust, Rubber, Polystyrene*, or material of similar resiliency to cushion the core.

6.8 *Miscellaneous Equipment*, such as adhesive tape and waterproof felt-tip markers.

7. Requirements and Procedures at the Drilling Site

7.1 *Sample Recovery*:

7.1.1 Accomplish sample recovery in accordance with Practice D 2113 or API RP-40.

7.1.2 Whichever approved drilling method is used, remove the samples from the core barrel with a minimum of disturbance.

7.2 *Handling*:

7.2.1 Each borehole shall be given full-time attention by a qualified inspector constantly available for observing, directing, photographing, and field logging. The inspector shall not perform simultaneously the same duties for more than one boring unless the borings are close enough to each other so that the entire inspection process can be done for each boring.

7.2.2 For relatively solid pieces of core that will not be adversely affected, the inspector shall use a marker, such as a felt-tip, to orient each piece so that later users will always be able to distinguish top from bottom. Acceptable formats are a continuous line with arrows or parallel solid and dashed lines with the dashed line always on the same side of the solid line. The direction convention shall be recorded in the log book. Locations of known depths should be marked directly on the core when the orientation marks are drawn.

7.3 *Core Photography*:

7.3.1 Perform core photography on all core samples with a camera of 35 mm (minimum) format using color film to record permanently the unaltered appearance of the rock. The film selected should be color balanced for the available lighting (daylight, flash, incandescent, or florescent), or an appropriate filter should be placed on the camera to compensate for the difference. The core should be cleaned prior to any photography.

7.3.2 A commercially available color strip chart should be included in the photo frame to serve as a reference to check the accuracy of the photographic reproduction of the rock core colors.

7.3.3 For rock placed in core boxes, take one photo of each box once it is filled to capacity. Include the inside of the box lid with appropriate identification data and a clearly visible length scale laid along one edge of the box so that it also shows in the photo.

7.3.4 Where very long, intact cores are being preserved in single plastic tubes, make detail-revealing close-ups of each core interval in addition to a single photo showing the complete core.

7.3.5 Take photographs before the core is obscured by protective sealants and wraps, and before any deterioration begins in particularly fragile or sensitive rock types.

7.3.6 For a boxed core that is not particularly sensitive and for which maintenance of in situ moisture content is not important, two photos should be made: one with the core in a surface dry condition and one with the core in a wet condition to bring out optical properties that would not otherwise be apparent.

7.3.7 This procedure may require photography both in the field and then later in the storage facility, but it must be completed before any test core removal and before damage from mishandling has a chance to occur.

7.3.8 Where it is impossible for a photo to show identification data marked directly on the sample or its container, then mount appropriately marked placards so as to be included in the frame.

7.3.9 Organize the photographs and mount in a folder for easy access and preservation.

7.4 *Initial Logging*:

7.4.1 The boring inspector must complete at least a preliminary field log of the core before it is packed away to be transported. Suggested procedures for logging are given in the literature.[4, 5, 6, 7] The preliminary log must include all identification data for the borehole and personnel and equipment involved, notations of coring run depths, recovery percentages, lithologic contact depths, types and locations of protection applied to samples, and any facts that would otherwise be unknown to whomever may complete a more detailed log at a later time. It is desirable that detailed logs be completed by the same inspector who does the field logging. It is advisable for the inspector immediately to make notations on the depths at which, in his judgment, any core losses occurred. Sometimes it is possible later to fill in gaps in the initial log by interpretations from wireline logs.

7.4.2 The inspector is to complete a detailed log on the drill site (see the literature[4, 5, 6, 7]) in cases where the core is likely to deteriorate or otherwise change before being examined again.

7.4.3 For fragile core that must be immediately protected by wrapping and sealing, preliminary logging should take place in the field, but application of protective measures are to take precedence over time-consuming detailed logging.

NOTE 1—It is permissible later to make changes in detailed logs when laboratory analysis indicates original misidentification of rock type or other geologic features.

7.5 *Sample Protection*—Four levels of sample protection are covered (see Section 3): routine care, special care, critical

[4] Association of Engineering Geologist, Core Logging Committee, South Africa Section, "A Guide to Core Logging for Rock Engineering," *Bulletin of the Association of Engineering Geologist*, Vol 15, No. 3, 1978, pp. 295–328.

[5] Deere, D. U., Dunn, J. R., Fickies, R. H., and Proctor, R. J., "Geologic Logging and Sampling of Rock Core for Engineering Purposes (Tentative)," Association of Professional Geological Scientists, 1977.

[6] The Geological Society, Engineering Group Working Party, "The Logging of Rock Core for Engineering Purposes," *Quarterly Journal of Engineering Geology*, Vol 3, 1970, pp. 1–24.

[7] International Society for Rock Mechanics, "Basic Geotechnical Description of Rock Masses," *International Journal of Rock Mechanics and Mining Sciences and Geomechanics Abstracts*, Vol 18, 1980, pp. 85–110.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:39:00 MDT

care, and soil-like care. The level of protection chosen will depend on the geologic character of the rock and the intended use for the core.

7.5.1 *Routine care (see* Fig. 2*)*:

7.5.1.1 For rock cored in 5 to 10-ft. (1.5 to 3.0 m) runs, samples are sufficiently protected if placed in structurally sound core boxes. Enclosing the core in a loose-fitting polyethylene sleeve (layflat tubing) prior to placing the core in the core box is recommended.

7.5.1.2 Where very long solid cores have been recovered and need to be preserved intact, place each core in a reasonably stiff tube (poly(vinyl chloride) (PVC) tubing is recommended) of equal or slightly greater length and secure both ends to prevent slippage. The inside diameter of the tube should be slightly greater than the core diameter; the wall thickness of the tube must be sufficient to provide the rigidity to prevent core breakage due to bending.

7.5.2 *Special Care*:

7.5.2.1 The moisture state of some rocks, and even the moisture-state history of rocks such as shales, affects their properties. If tests are to be performed on the core, and if it is possible that a change in the moisture state may influence the test results, then the core must be sealed to prevent changes in the moisture state until the time of testing. This same procedure also applies to other samples where it is important to maintain fluids other than water (for example, hydrocarbons).

7.5.2.2 Seal samples requiring special care. Such sealing shall consist of a tightly fitting wrapping of a plastic film, such as vinylidene chloride (for example, saran or similar material). Over this, place another tight wrapping of aluminum foil (Note 2). Apply both of these wrappings so that as little air as possible is trapped beneath the wrappings. Overlap the ends of the wrappings over the ends of the sample and fold over so as to seal the ends of the sample. Finally, apply a few coats of a plastic microcrystalline wax (Note 3), preferably with a paint brush, although rapid dipping in molten wax is also acceptable. Apply a minimum of ⅛ in. (3 mm) of this plastic microcrystalline wax over the entire surface of the sample. This thickness of wax shall consist of at least two coatings of wax and preferably more. For long periods of storage, apply a minimum of ¼ in. (6 mm) of wax.

NOTE 2—In some instances where the aluminum foil wrapping chemically reacts with either the rock or its fluids, it should be replaced with some other metal foil which is nonreactive. A less preferable method is to delete the metal foil and increase the thickness of the sealing wax. If the metal foil is not used, the thickness of the sealing wax should be increased to ¼ in. (6 mm) when the required storage time is short (one week to a few months). For longer period, a thickness of ⅜ in. (9 mm) may be adequate.

NOTE 3—It is important that plastic microcrystalline wax be utilized, as it does not become brittle. Regular microcrystalline waxes are brittle and susceptible to cracking, which then severely limits their use as sealants.

7.5.2.3 Polyethylene layflat tubing is a much less effective moisture barrier than the wax-metal foil seal described in 7.5.2.2, and particular attention must be paid to the manner in which the ends of the tubing are sealed. This method of sealing may be used only if the person having testing management responsibility can prove that moisture loss can be tolerated without significantly affecting the pertinent mechanical and physical properties of the samples. The thickness of the polyethylene plastic tubing shall be at least 0.005 in. (0.13 mm). Tightly seal the ends of the tubing, for example, by doubling the ends over at least twice and securing with adhesive tape. Make a check for seal quality; an acceptable method is to squeeze the sealed tube and observe if the air escapes easily. The effectiveness of this barrier can be improved by tightly wrapping the core with plastic film, such as vinylidene chloride, before it is placed in the polyethylene tubing. Even with the addition of this extra plastic film, the

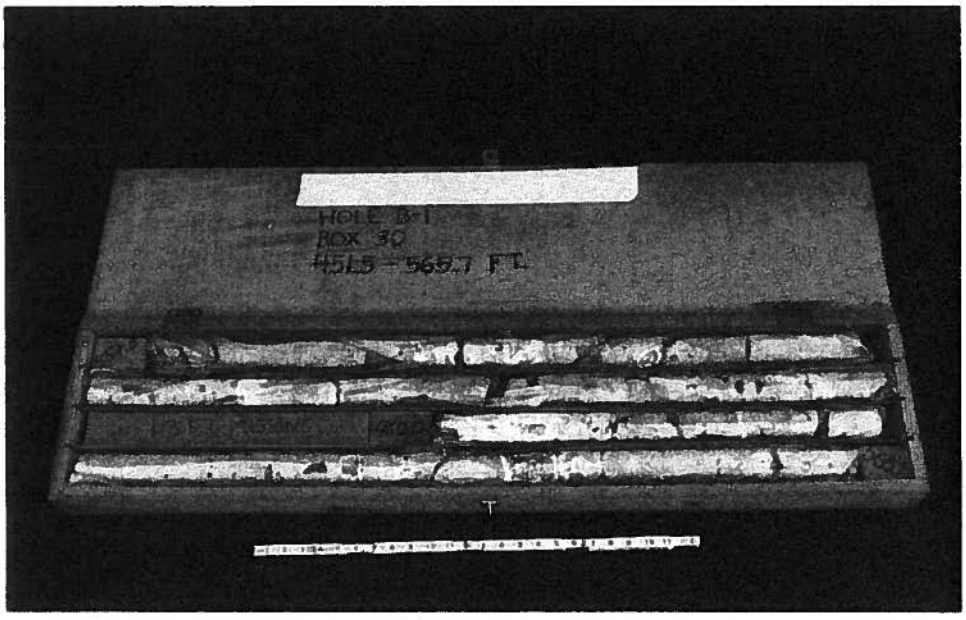


FIG. 2 Typical Wooden Core Box Showing Core Placement, Labeling, and Polyethylene Layflat Tubing Placed Over Core as Suggested for Routine Care (as in 7.5.1)

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5805905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:39:00 MDT

moisture barrier is still not nearly as effective as the wax-metal foil seal described in 7.5.2.2.

7.5.3 *Critical Care*:

7.5.3.1 If shock and vibration or variations in temperature, or both, may subject samples to unacceptable conditions during transport, place samples in suitable core boxes that provide cushioning or thermal insulation, or both.

7.5.3.2 Protect fragile samples from mechanical disturbance, such as vibration and shock. Completely encase each sample in the cushioning material (sawdust, rubber, polystyrene, urethane foam, or material of similar resiliency). The cushioning between the samples and walls of the core boxes should have a minimum thickness of 1 in. (25 mm). Provide a minimum thickness of 2 in. (50 mm) on the core box floor and lid. Samples must fit snugly into their assigned space.

7.5.3.3 Thermally insulate samples that are temperature sensitive. Thermal insulation is best provided by placing the core container (box or tube) inside another container that is designed specifically to provide thermal insulation. Such insulating containers are generally constructed of double or triple layers of an insulating material. Additionally, such containers are usually relatively airtight.

7.5.3.4 Seal samples that are sensitive to mechanical disturbance, fluid content, and temperature in accordance with 7.5.2.2.

7.5.4 *Soil-Like Care*—Such materials should be treated as indicated in Practices D 4220.

7.5.5 Handling of sensitive samples between borehole and transport vehicle should take place within a covered space that either provides shade against intense sunlight or can be heated to keep temperatures above freezing.

7.6 *Preparation of Storage and Shipping Containers*:

7.6.1 Core boxes must be constructed rigidly enough to prevent flexing of the core when the box is picked up by its ends. Wood is the desired construction material, and it should be ½ to ¾ in. (13 to 19 mm) thick. Partitions between core lengths shall be firmly nailed in place to increase the stiffness of the box. The lid should have sturdy hinges and a strong hasp or screw closure. Do not drive nails in the lid.

7.6.2 Packing material should be placed in the core box to support the core and prevent it from rolling around in the box.

7.6.3 Gently place the core in the core box starting with the shallowest depth at the upper left corner (nearest the hinge, if the lid is hinged) and progressing downward, as in reading a book, to the deepest depth at the lower right corner as shown in Fig. 2.

7.6.4 Core blocks should be placed at the ends of each core run.

7.6.5 Where a run of less than 100 % recovery yields a core that is too short to fill its assigned trough, the recovered core should be held secure and prevented from scattering by placement of spacer block such as a piece of wood or cardboard tube cut to the length of the missing core.

7.6.6 Unnecessary breaking of the core to fit the core box should be avoided as it may reduce the number of available test specimens. Any intentional breaks shall be recorded on the log.

7.6.7 Mark depths of the top and bottom of the core length in the box with a waterproof marker near the core ends and corresponding box corners. Intermediate depths that are accurately known should also be similarly marked.

7.6.8 Mark both the top and one edge of the core box with the following information before transportation:

7.6.8.1 Company or project name, or both,

7.6.8.2 Drill hole number or location,

7.6.8.3 Core box number in sequence down the hole, and

7.6.8.4 Depths from a specified hole datum to top and bottom of core length in box marked on appropriate corners of box.

7.6.9 The following additional information may be required to be written on the core box for specific projects:

7.6.9.1 Percent core recovery, and

7.6.9.2 Rock quality designation,

7.6.10 Mark or label tubes with the same information as core boxes. Such tubes shall have sufficient flexural rigidity to prevent core breakage due to bending. A core that requires protection from mechanical shock should have shock absorbing material packed concentrically around the core.

8. Transportation Requirements and Procedures

8.1 Damage may occur to the core if certain precautions are not taken during transportation. The mode of transportation, distance, terrain, and handling at each end are important factors. The following requirements and procedures should be considered when transporting a core from one location to another:

8.1.1 Remove the core from the drill site before it has a chance to freeze or heat up or be damaged by activities at the drill site.

8.1.2 Handling during loading and unloading should be done gently. Never drop core boxes or tubes, but rather slide gently into position. If a box is accidentally dropped, record this fact.

8.1.2.1 A record of mishandling by commercial carriers is often difficult, if not impossible, to obtain. The use of company or agency vehicles is recommended, with someone assigned to supervise handling and storage along the transport route.

8.1.3 Provide transportation by a suitable vehicle to prevent damage by mechanical vibration, shock, freezing, and high temperatures along the entire transport route.

8.1.3.1 Rough terrain near the drill site may require the use of four-wheel drive vehicles for transport. In such cases, protect a core in the critical care category (7.5.3) by vibration insulators around the sides, bottom, and top of the core containers.

8.1.3.2 Highway transportation by passenger car, rather than van or truck, may be required for a fragile core.

8.1.4 Thermally insulate temperature sensitive cores. Thermally insulated containers are the preferred method (7.5.3.3). Such containers will maintain a thermal environment suitable for core preservation during the period of transport.

9. Cataloging and Storage Requirements and Procedures

9.1 *Cataloging*—Assign unique identification numbers to each core sample in the inventory. The identification numbers shall be easily traceable to the borehole number and depth interval from which the core was recovered. An acceptable method in many cases is to use the borehole number and depth

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:39:00 MDT

interval as the identification numbers. It may be helpful to include the run numbers and core box numbers in the cataloging system, as well as other important subgroupings on large projects.

9.1.1 *Specimen Identification*—Core samples may be removed from inventory for a variety of tests and analyses. Preparation of specimens for these tests typically requires that the original sample be sawed and sometimes recorded. Assign core material removed for testing and analysis unique identification numbers which are easily traceable to the borehole number and depth, or which may actually be the borehole number and depth. The depth shall be taken as the depth to the midpoint of the specimen, or the depths to the top and bottom of the specimen, as required for the test program. It is often convenient to further identify the specimen with letters or numbers signifying the type of test or analysis to be performed. End pieces remaining after cutting shall be returned to their respective locations in the original core box. To hold the end pieces in their proper locations in the core box, a spacer should be placed in the location from which a test specimen has been removed.

9.2 *Storage*—Store the core so that it can be easily retrieved and in an environment that does not alter properties that are of interest.

9.2.1 *Storage Racks*—Place core boxes on racks that provide support over the entire length of the box. To reduce damage due to handling of core in the critical-care and soil-like categories, do not place more that a single layer of core boxes on a shelf. The lowest shelf in the rack should be raised slightly above floor level to allow air circulation for temperature control and as a precaution against water damage.

9.2.2 *Temperature Control*—Prevent from freezing core samples that contain significant amounts of water. Also, temperature extremes may need to be limited to prevent undesirable chemical changes, such as dewatering of certain minerals. More stringent temperature control is required to prevent large temperature variations which could pump fluid from the sample.

10. Laboratory Specimen Preparation Requirements and Procedures

10.1 Samples removed from the inventory for testing and analysis in the laboratory must be handled carefully to preserve fluid content and integrity.

10.1.1 *Specimen Preparation*—Preparation techniques must be selected carefully to prevent sample alteration. Give special attention to the choice of cutting fluids that are used in machining operations such as sawing, coring, and grinding. For example, water will dissolve halite and will cause some shales to swell or slake. Specimens of water-sensitive material may be prepared with air cooling if the material is soft enough that there are no hot spots during the operation. Cutting oils, if used, should be removed from the sample using a solvent because the oil may affect certain properties of some rock types. Specimens that are moisture sensitive should be sealed between various steps of preparation if the time between steps is more than 30 min. A controlled-humidity room or chamber may be necessary for preparation of some rock types. Samples that were sealed in the inventory should be sealed after preparation until tested, unless testing occurs within 30 min after preparation.

11. Post-Test Disposition Requirements and Procedures

11.1 The requirements and procedures for disposition of test specimens vary with the nature of the project and the length of time for which there is a need to preserve the test material. The need for future re-examination may arise in the verification of engineering design or litigation.

11.2 The tests performed may be destructive or nondestructive. Reassemble and tape major fragments from destructive tests and place in a plastic bag with the fine material. Identify the bag with the specimen identification (borehole number and depth). Also place specimens from nondestructive tests in plastic bags and label.

11.3 If the specimen material is not moisture sensitive, and if easy access to the test specimens is desired, the specimens in the labeled bags may be placed in a box labeled as follows:

11.3.1 Company or project name, or both,

11.3.2 Borehole number or location,

11.3.3 Types of tests performed on specimens,

11.3.4 Name of person responsible for disposition, and

11.3.5 Date of disposition.

11.4 For moisture sensitive test specimens for which easy access is desired, the requirements of 11.3 apply except that the specimen shall be suitably sealed from the atmosphere. The sealing procedure shall be as stringent for post-test disposition as it was for the initial core preservation, as given in 7.5.

11.5 Storage requirements for boxes in 11.3 and 11.4 are the same as for the original core as given in 9.2.

11.6 If easy access to all of the test specimens is not required, return the tested specimens to their original locations in the core boxes or PVC tubes, and reseal as appropriate.

12. Records

12.1 A permanent, legible record shall be maintained by a technically qualified person for the core from each borehole. The following information shall be recorded:

12.1.1 Company and project names,

12.1.2 Drill hole number and location,

12.1.3 Orientation of borehole,

12.1.4 Elevation of hole collar, Kelly bushing, or some other datum,

12.1.5 Date(s) of coring,

12.1.6 Core box or PVC tube numbers and core depth interval contained in each,

12.1.7 Date and name of person doing initial logging,

12.1.8 Geologic log or reference to its location, and

12.1.9 Photographs of core or reference to their location.

NOTE 4—A permanent historical record needs to be maintained so that it is accessible for later use in identifying the core, ascertaining what geologic study and testing has been performed on the core, and its storage location of other disposition.

12.2 Desirable records which may also be required for some projects are as follows:

12.2.1 Each date of transportation, from where to where, mode of transportation, and name of responsible person,

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS
Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:39:00 MDT

12.2.2 Storage locations and source of temperature and relative humidity data,

12.2.3 Date and person removing test samples from storage, including a list of sample lengths removed and their depths,

12.2.4 Name and location of testing laboratory, nature of testing, if known, and name of responsible person,

12.2.5 Post-test disposition including date, name of responsible person, and location of tested specimens if not returned to original core boxes or PVC tubes, and

12.2.6 Date and nature of any other activity involving the core such as handling, and additional inspection and testing.

13. Keywords

13.1 identification; microcrystalline wax; sampling; transportation

SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (D 5079 – 02 (2006)) that may impact the use of this standard. (Approved July 1, 2008.)

(*1*) Inserted Section 1.4 on units.
(*2*) Edited 7.5.1.1.
(*3*) Edited 9.2.1.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:39:00 MDT

Designation: D 425 – 88 (Reapproved 2008)

Standard Test Method for Centrifuge Moisture Equivalent of Soils[1]

This standard is issued under the fixed designation D 425; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the moisture equivalent of soil in the laboratory by means of a centrifuge technique.

1.2 This test method is limited to disturbed specimens of coarse-grained soils having fines of low plasticity such as SP, SW, SC-SM, or SM soils. The test is limited to soils passing the 2.00-mm sieve or that fraction of a soil passing a 2.00-mm sieve.

NOTE 1—Test Method D 3152 or Test Method D 2325 should be used to evaluate the capillary-moisture relations of fine-grained soils and coarse-grained soils having fines of medium to high plasticity, undisturbed soils, and soils at specific desired units weights.

1.3 The test method is temperature-dependent, and consistent comparable results can be obtained only if the tests are performed under a constant-temperature condition.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 653 Terminology Relating to Soil, Rock, and Contained Fluids

D 2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D 2325 Test Method for Capillary-Moisture Relationships for Coarse- and Medium-Textured Soils by Porous-Plate Apparatus[3]

D 3152 Test Method for Capillary-Moisture Relationships for Fine-Textured Soils by Pressure-Membrane Apparatus[3]

D 4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

E 11 Specification for Wire Cloth and Sieves for Testing Purposes

3. Terminology

3.1 All definitions are in accordance with Terminology D 653. Terms of particular significance are as follows:

3.2 *capillary fringe zone*—the zone above the free water elevation in which water is held by capillary action.

3.3 *centrifuge moisture equivalent*—the water content of a soil after it has been saturated with water and then subjected for one hour to a centrifugal force equal to 1000 times that of gravity.

3.4 *specific retention*—the ratio of the volume of water that cannot be drained from a saturated soil under the action of force of gravity to the total volume of voids.

3.5 *water-holding capacity*—the smallest value to which the water content of soil or rock can be reduced by gravity drainage.

4. Summary of Test Method

4.1 The centrifuge moisture equivalent of soils is determined by initially air-drying the soil, selecting two 5-g test specimens, thoroughly soaking each test specimen, and then determining the water content of each specimen after it has been centrifuged for 1 h at a force equal to 1000 times that of gravity at a controlled temperature of 20 ± 1°C.

5. Significance and Use

5.1 Not all water contained in a saturated soil can be removed by gravity drainage. The amount of water retained after gravity drainage is usually expressed as water holding capacity or specific retention. It varies with time, and with the particle-size distribution and plasticity of the soil (in general, increasing in value with increasing plasticity index).

5.2 In general, the centrifuge moisture equivalent is based on the theory of applying a centrifugal force great enough to reduce the capillary fringe zone enough that it can be ignored without introducing much error, even in small specimens, and yet not so great as to withdraw a large proportion of the water

[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity and Density Characteristics of Soils.

Current edition approved Feb. 1, 2008. Published March 2008. Originally approved in 1935. Last previous edition approved in 2001 as D 425 – 88 (2001).

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Withdrawn.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/28/2012 13:23:17 MDT

that is held securely above the capillary fringe. For example, if a soil will hold water 100 mm by capillarity acting against gravity, the soil will theoretically be able to hold the water only 0.1 mm against a centrifugal force that is 1000 times greater than the force of gravity. It has been determined that for at least medium-textured soils (sandy to silty particle-size distribution) the centrifuge moisture equivalent approximates the water holding capacity and when combined with the bulk density can be used to calculate an approximate specific retention and specific yield. These properties when combined with porosity can be used to estimate aquifer storage coefficient.

6. Apparatus

6.1 *Centrifuge*—A centrifuge of such a size and so driven that a force equal to 1000 times the force of gravity may be exerted on the center of gravity of the soil specimen for 1 h. The centrifuge chamber shall be capable of maintaining a controlled temperature of 20 ± 1°C. The revolutions per minute, N, required to provide a centrifugal force of 1000 times gravity is determined from the equation:

$$N = \sqrt{\frac{RCF}{0.0000111\,rm}} \quad (1)$$

where:

N = revolutions per minute,
RCF = relative centrifugal force (1000),
r = radius of rotation to center of gravity of the test specimen, cm, and
m = mass of the body, taken as unity.

For normal equipment installation, N will equal approximately 2300 rpm.

6.2 *Gooch Crucible*—A procelain Gooch crucible having a perforated bottom, a capacity of approximately 25 mL, and a diameter at bottom of about 20 mm (Fig. 1). Crucibles should be numbered and paired in such a way that their masses meet the requirements of the manufacturer of the centrifuge.

6.3 *Babcock Trunnion Cups*—At least one pair of centrifuge cups with caps and with a crucible holder for supporting the Gooch crucible above the bottom of the cup (Fig. 1). The holder shall have sufficient clearance to fit fully within the cup and short support the cup in such a manner that the water ejected during the centrifuging operation does not come in contact with the crucible and soil. Cups and crucible holders should be balanced in pairs opposite each other in the centrifuge and should be numbered in pairs (for example, 1, 1A, 2, 2A, etc.).

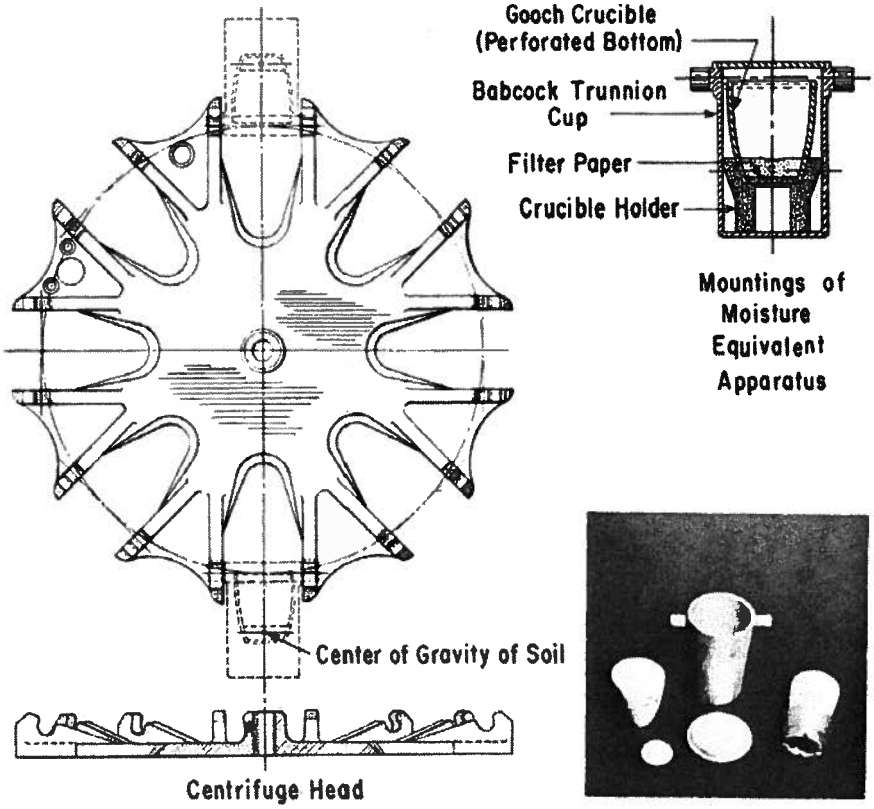


FIG. 1 Crucible, Trunnion Cup, and Cup Holder

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS
Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/28/2012 13:23:17 MDT

6.4 *Filter Paper*—A circular piece of filter paper just large enough to cover the inside bottom of the Gooch crucible.

NOTE 2—Filter papers may be purchased already cut to size from a scientific supply company. A medium speed, high wet strength (hardened) paper is recommended.

6.5 *Balance*—A balance having a readability of 0.01 g, and accurate to ±0.03 g.

NOTE 3—For additional information on balances refer to Specification D 4753.

6.6 *Humidifier*—A desiccator cabinet or large desiccator jar with water in the lower half works satisfactorily. The desiccator plate should be covered with hardware cloth on which to set the crucibles and soil specimens.

6.7 *Oven*—A thermostatically controlled drying oven, preferably of the forced-draft type, capable of maintaining a uniform temperature of 110 ± 5°C throughout.

6.8 *Water Content Containers*—Suitable containers made of material resistant to corrosion and change in mass upon repeated heating, cooling, and cleaning. Containers shall have close-fitting lids to prevent loss of moisture from the soil before initial weighing and to prevent absorption of moisture from the atmosphere following oven drying and before final weighing. One container is needed for each water content determination. Containers should be 60 to 90-mL capacity.

NOTE 4—It is recommended that the containers be numbered in pairs to coincide with the crucible numbers.

6.9 *Mortar and Pestle*—A mortar and rubber-covered pestle suitable for breaking up the aggregations of soil particles.

6.10 *Sieve*—2.00-mm (No. 10) sieve conforming to Specification E 11. A sieve bottom pan and lid are also highly desirable for facilitating specimen preparation.

7. Test Specimen

7.1 Expose the soil sample, as received, to the air at room temperature until dried thoroughly. Break up the aggregations thoroughly in the mortar with a rubber-covered pestle. Dry sieve this material on a 2.00-mm sieve.

NOTE 5—If the sample, as received, has a mass much greater than required to perform this test, the sample can be thoroughly mixed and a sample weighing about 50 g split out for air drying.

7.2 Take two 5-g specimens from the material passing the 2.00-mm sieve after it has been thoroughly mixed. The crucibles should be numbered and paired in such a way that the masses of the crucibles and contents meet the requirements of the manufacturer of the centrifuge.

8. Procedure

8.1 Place the specimens loosely but evenly in the paired Gooch crucibles, in which previously has been placed a piece of wet filter paper that just covers the bottom of each crucible. Place the crucible in a pan of distilled or demineralized water of a depth at least 5 mm greater than the height of soil in the cup and allow the soil to take up water until "saturated." Eight hours or overnight usually is satisfactory, as indicated by the presence of free water covering the surface of the soil. Place the crucible and specimen in the humidifier to drain for at least 12 h to ensure uniform distribution of moisture throughout the soil mass. Pour or siphon off any water standing above the surface of the specimens and place the crucibles in the centrifuge cups fitted as described in 6.2. Place the paired crucibles in the centrifuge cups opposite each other in the centrifuge.

8.2 Control the centrifuge at a temperature 20 ± 1°C. Bring the centrifuge to the required speed within 5 min by five successive equal steps of the rheostat, with 1 min at each step. Maintain the centrifuge for 60 min at a constant speed which, for the diameter head used, will exert a centrifugal force 1000 times the force of gravity upon the center of gravity of the test specimen. Following this period of centrifuging at required speed, allow the centrifuge to come to rest with as little braking as possible, but not to exceed a 5-min time interval.

8.3 When water is observed on the top of the soil after centrifuging, the soil is said to have water-logged. The centrifuge moisture equivalent is not considered to be valid for this material and the report should be so noted.

8.4 Immediately after centrifuging, transfer soil from the crucibles to the water content containers as quickly as possible to minimize moisture loss; it is not necessary to remove all of the soil from the crucibles. Then determine the water content of each specimen in accordance with Method D 2216.

8.5 A copy of a sample data sheet is shown in Fig. 2. Any data sheet can be used, provided the form contains all the required data.

9. Calculation

9.1 Calculate the centrifuge moisture equivalent as the average of the water contents of the two specimens.

10. Report

10.1 The report shall include the following:

10.1.1 Identification of sample (material) being tested, by boring number, sample number, test number, etc.

10.1.2 Average centrifuge moisture equivalent of the specimen to the nearest 1 %. If the specimen was water-logged, the report should state that the centrifuge moisture equivalent was not valid for the soil tested.

10.1.3 Indication of which test specimen, if any, was water-logged.

11. Precision and Bias

11.1 *Precision*—Information on the precision of this test method is being gathered.[4]

12. Keywords

12.1 centrifuge moisture equivalent; specific retention; specific yield, storage capacity; water holding capacity

[4] For a discussion of variables which affect the results of this test, see Johnson, A. I., Prill, R. C., and Morris, D. A., "Specific Yield–Column Drainage and Centrifuge Moisture Content," U.S. Geological Survey Water Supply Paper 1662-A, 1963.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/28/2012 13:23:17 MDT

(1) Laboratory Sample No.								
(2) Container No.								
(3) Weight of container + wet sample (W_{cws}), g								
(4) Weight of container + dry sample (W_{cs}), g								
(5) Weight of moisture (W_w), g (3 - 4)								
(6) Weight of container (W_c), g								
(7) Weight of dry sample, (W_s), g (4 - 6)								
(8) Moisture Equivalent (w_{cme}), % (5 ÷ 7) x 100								
(9) Average Moisture Equivalent (w_{cme}), %								

Date and Name: Analyzed_____ Computed_____ Checked_____
Remarks: _____

(1) Laboratory Sample No.								
(2) Container No.								
(3) Weight of container + wet sample (W_{cws}), g								
(4) Weight of container + dry sample (W_{cs}), g								
(5) Weight of moisture (W_w), g (3 - 4)								
(6) Weight of container (W_c), g								
(7) Weight of dry sample, (W_s), g (4 - 6)								
(8) Moisture Equivalent (w_{cme}), % (5 ÷ 7) x 100								
(9) Average Moisture Equivalent (w_{cme}), %								

Date and Name: Analyzed_____ Computed_____ Checked_____
Remarks: _____

FIG. 2 Centrifuge Moisture Equivalent Sample Data Sheet

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/28/2012 13:23:17 MDT

Designation: D445 – 11a

British Standard 2000: Part 71:1990

Designation: 71/1/97

Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)[1]

This standard is issued under the fixed designation D445; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method specifies a procedure for the determination of the kinematic viscosity, ν, of liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity, η, can be obtained by multiplying the kinematic viscosity, ν, by the density, ρ, of the liquid.

NOTE 1—For the measurement of the kinematic viscosity and viscosity of bitumens, see also Test Methods D2170 and D2171.

NOTE 2—ISO 3104 corresponds to Test Method D445.

1.2 The result obtained from this test method is dependent upon the behavior of the sample and is intended for application to liquids for which primarily the shear stress and shear rates are proportional (Newtonian flow behavior). If, however, the viscosity varies significantly with the rate of shear, different results may be obtained from viscometers of different capillary diameters. The procedure and precision values for residual fuel oils, which under some conditions exhibit non-Newtonian behavior, have been included.

1.3 The range of kinematic viscosities covered by this test method is from 0.2 to 300 000 mm^2/s (see Table A1.1) at all temperatures (see 6.3 and 6.4). The precision has only been determined for those materials, kinematic viscosity ranges and temperatures as shown in the footnotes to the precision section.

1.4 The values stated in SI units are to be regarded as standard. The SI unit used in this test method for kinematic viscosity is mm^2/s, and the SI unit used in this test method for dynamic viscosity is mPa·s. For user reference, 1 $mm^2/s = 10^{-6}$ m^2/s = 1 cSt and 1 mPa·s = 1 cP = 0.001 Pa·s.

1.5 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers

D1193 Specification for Reagent Water

D1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer

D1480 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer

D1481 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer

D2162 Practice for Basic Calibration of Master Viscometers and Viscosity Oil Standards

D2170 Test Method for Kinematic Viscosity of Asphalts (Bitumens)

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved May 1, 2011. Published May 2011. Originally approved in 1937. Last previous edition approved in 2011 as D445–11. DOI: 10.1520/D0445-11A.

In the IP, this test method is under the jurisdiction of the Standardization Committee.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard.**

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:10:10 MDT

D2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer
D6071 Test Method for Low Level Sodium in High Purity Water by Graphite Furnace Atomic Absorption Spectroscopy
D6074 Guide for Characterizing Hydrocarbon Lubricant Base Oils
D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
D6617 Practice for Laboratory Bias Detection Using Single Test Result from Standard Material
E1 Specification for ASTM Liquid-in-Glass Thermometers
E77 Test Method for Inspection and Verification of Thermometers
E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

2.2 *ISO Standards:*[3]

ISO 3104 Petroleum Products—Transparent and Opaque Liquids—Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity
ISO 3105 Glass Capillary Kinematic Viscometers—Specification and Operating Instructions
ISO 3696 Water for Analytical Laboratory Use—Specification and Test Methods
ISO 5725 Accuracy (trueness and precision) of measurement methods and results.
ISO 9000 Quality Management and Quality Assurance Standards—Guidelines for Selection and Use
ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories

2.3 *NIST Standards:*[4]

NIST Technical Note 1297 Guideline for Evaluating and Expressing the Uncertainty of NIST Measurement Results[5]
NIST GMP 11 Good Measurement Practice for Assignment and Adjustment of Calibration Intervals for Laboratory Standards[6]
NIST Special Publication 819 Guide for the Use of the International System of Units (SI) [7]

3. Terminology

3.1 See also *International Vocabulary of Metrology.*[8]

3.2 *Definitions:*

3.2.1 *digital contact thermometer (DCT), n*—an electronic device consisting of a digital display and associated temperature sensing probe.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *automated viscometer, n*—apparatus which, in part or in whole, has mechanized one or more of the procedural steps indicated in Section 11 or 12 without changing the principle or technique of the basic manual apparatus. The essential elements of the apparatus in respect to dimensions, design, and operational characteristics are the same as those of the manual method.

3.3.1.1 *Discussion*—Automated viscometers have the capability to mimic some operation of the test method while reducing or removing the need for manual intervention or interpretation. Apparatus which determine kinematic viscosity by physical techniques that are different than those used in this test method are not considered to be Automated Viscometers.

3.3.2 *density, n*—the mass per unit volume of a substance at a given temperature.

3.3.3 *dynamic viscosity, η, n*—the ratio between the applied shear stress and rate of shear of a material.

3.3.3.1 *Discussion*—It is sometimes called the coefficient of dynamic viscosity or absolute viscosity. Dynamic viscosity is a measure of resistance to flow or deformation which constitutes a material's ability to transfer momentum in response to steady or time-dependent external shear forces. Dynamic viscosity has the dimension of mass divided by length and time and its SI unit is pascal times second (Pa·s). Among the transport properties for heat, mass, and momentum transfer, dynamic viscosity is the momentum conductivity.

3.3.4 *kinematic viscosity, ν, n*—the ratio of the dynamic viscosity (η) to the density (ρ) of a material at the same temperature and pressure.

3.3.4.1 *Discussion*—Kinematic viscosity is the ratio between momentum transport and momentum storage. Such ratios are called diffusivities with dimensions of length squared divided by time and the SI unit is metre squared divided by second (m^2/s). Among the transport properties for heat, mass, and momentum transfer, kinematic viscosity is the momentum diffusivity.

3.3.4.2 *Discussion*—Formerly, kinematic viscosity was defined specifically for viscometers covered by this test method as the resistance to flow under gravity. More generally, it is the ratio between momentum transport and momentum storage.

3.3.4.3 *Discussion*—For gravity-driven flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density, ρ, if the density of air is negligible compared to that of the liquid. For any particular viscometer covered by this test method, the time of flow of a fixed volume of liquid is directly proportional to its kinematic viscosity, ν, where $\nu = \eta/\rho$, and η is the dynamic viscosity.

4. Summary of Test Method

4.1 The time is measured for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled and known temperature. The kinematic viscosity (determined value) is the product of the measured flow time and the calibration constant of the viscometer. Two such determinations are needed from which to calculate a kinematic viscosity result that is the average of two acceptable determined values.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[4] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.

[5] http://physics.nist.gov/cuu/Uncertainty/bibliography.html

[6] http://ts.nist.gov/WeightsAndMeasures/upload/GMP_11_Mar_2003.pdf

[7] http://www.nist.gov/pml/pubs/sp811/index.cfm

[8] *International Vocabulary of Metrology — Basic and General Concepts and Associated Terms (VIM)*, 3rd ed., 2008, http://www.bipm.org/en/publications/guides/vim.html.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:10:10 MDT

5. Significance and Use

5.1 Many petroleum products, and some non-petroleum materials, are used as lubricants, and the correct operation of the equipment depends upon the appropriate viscosity of the liquid being used. In addition, the viscosity of many petroleum fuels is important for the estimation of optimum storage, handling, and operational conditions. Thus, the accurate determination of viscosity is essential to many product specifications.

6. Apparatus

6.1 *Viscometers*—Use only calibrated viscometers of the glass capillary type, capable of being used to determine kinematic viscosity within the limits of the precision given in the precision section.

6.1.1 Viscometers listed in Table A1.1, whose specifications meet those given in Specifications D446 and in ISO 3105 meet these requirements. It is not intended to restrict this test method to the use of only those viscometers listed in Table A1.1. Annex A1 gives further guidance.

6.1.2 *Automated Viscometers*—Automated apparatus may be used as long as they mimic the physical conditions, operations or processes of the manual apparatus. Any viscometer, temperature measuring device, temperature control, temperature controlled bath or timing device incorporated in the automated apparatus shall conform to the specification for these components as stated in Section 6 of this test method. Flow times of less than 200 s are permitted, however, a kinetic energy correction shall be applied in accordance with Section 7 on Kinematic Viscosity Calculation of Specifications D446. The kinetic energy correction shall not exceed 3.0 % of the measured viscosity. The automated apparatus shall be capable of determining kinematic viscosity of a certified viscosity reference standard within the limits stated in 9.2.1 and Section 17. The precision shall be of statistical equivalence to, or better (has less variability) than the manual apparatus.

NOTE 3—Precision and bias of kinematic viscosity measurements for flow times of less than 200 s has not been determined. The precision stated in Section 17 is not know to be valid for kinematic viscosity measurements with flow times less than 200 s.

6.2 *Viscometer Holders*—Use viscometer holders to enable all viscometers which have the upper meniscus directly above the lower meniscus to be suspended vertically within 1° in all directions. Those viscometers whose upper meniscus is offset from directly above the lower meniscus shall be suspended vertically within 0.3° in all directions (see Specifications D446 and ISO 3105).

6.2.1 Viscometers shall be mounted in the constant temperature bath in the same manner as when calibrated and stated on the certificate of calibration. See Specifications D446, see Operating Instructions in Annexes A1–A3. For those viscometers which have Tube L (see Specifications D446) held vertical, vertical alignment shall be confirmed by using (*1*) a holder ensured to hold Tube L vertical, or (*2*) a bubble level mounted on a rod designed to fit into Tube L, or (*3*) a plumb line suspended from the center of Tube L, or (*4*) other internal means of support provided in the constant temperature bath.

6.3 *Temperature-Controlled Bath*—Use a transparent liquid bath of sufficient depth such, that at no time during the measurement of flow time, any portion of the sample in the viscometer is less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

6.3.1 *Temperature Control*—For each series of flow time measurements, the temperature control of the bath liquid shall be such that within the range from 15 to 100°C, the temperature of the bath medium does not vary by more than ±0.02°C of the selected temperature over the length of the viscometer, or between the position of each viscometer, or at the location of the thermometer. For temperatures outside this range, the deviation from the desired temperature must not exceed ±0.05°C.

6.4 *Temperature Measuring Device in the Range from 0 to 100°C*—Use either calibrated liquid-in-glass thermometers (Annex A2) with an accuracy after correction of ±0.02°C or better, or a digital contact thermometer as described in 6.4.2 with equal or better accuracy.

6.4.1 If calibrated liquid-in-glass thermometers are used, the use of two thermometers is recommended. The two thermometers shall agree within 0.04°C.

6.4.2 When using a digital contact thermometer (DCT) the following shall apply:

(1) The only acceptable sensors are a resistance temperature device (RTD), such as a platinum resistance thermometer (PRT), or a thermistor.

(2) A minimum display resolution of 0.01°C.

(3) A combined (display and probe) minimum accuracy of ±0.02°C.

(4) A response time of less than 6 s as defined in Specification E1137/E1137M.

(5) A drift of less than 10 mK (0.01°C) per year.

(6) Linearity of 10 mK over the range of intended use.

(7) The DCT shall have a report of temperature calibration traceable to a national calibration or metrology standards body issued by a competent calibration laboratory with demonstrated competency in temperature calibration.

(8) The calibration report shall include data for the series of test points which are appropriate for its intended use.

6.4.2.1 For a constant temperature bath employed in manual viscosity measurements, the probe shall be immersed at least 100 mm below the liquid level of the bath but no less than 3 times the sensor element length. See Fig. 1. The end of the probe sheath shall not extend past the bottom of the viscometer. It is preferable for the center of the sensing element to be located at the same level as the lower half of the working capillary as long as the minimum immersion requirements are met.

6.4.2.2 In the case of constant temperature baths used in instruments for automatic viscosity determinations, the user is to contact the instrument manufacturer for the correct DCT that has performance equivalence to that described here.

6.4.3 Outside the range from 0 to 100°C, use either calibrated liquid-in-glass thermometers of an accuracy after correction of ±0.05°C or better, or any other thermometric device

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:10:10 MDT

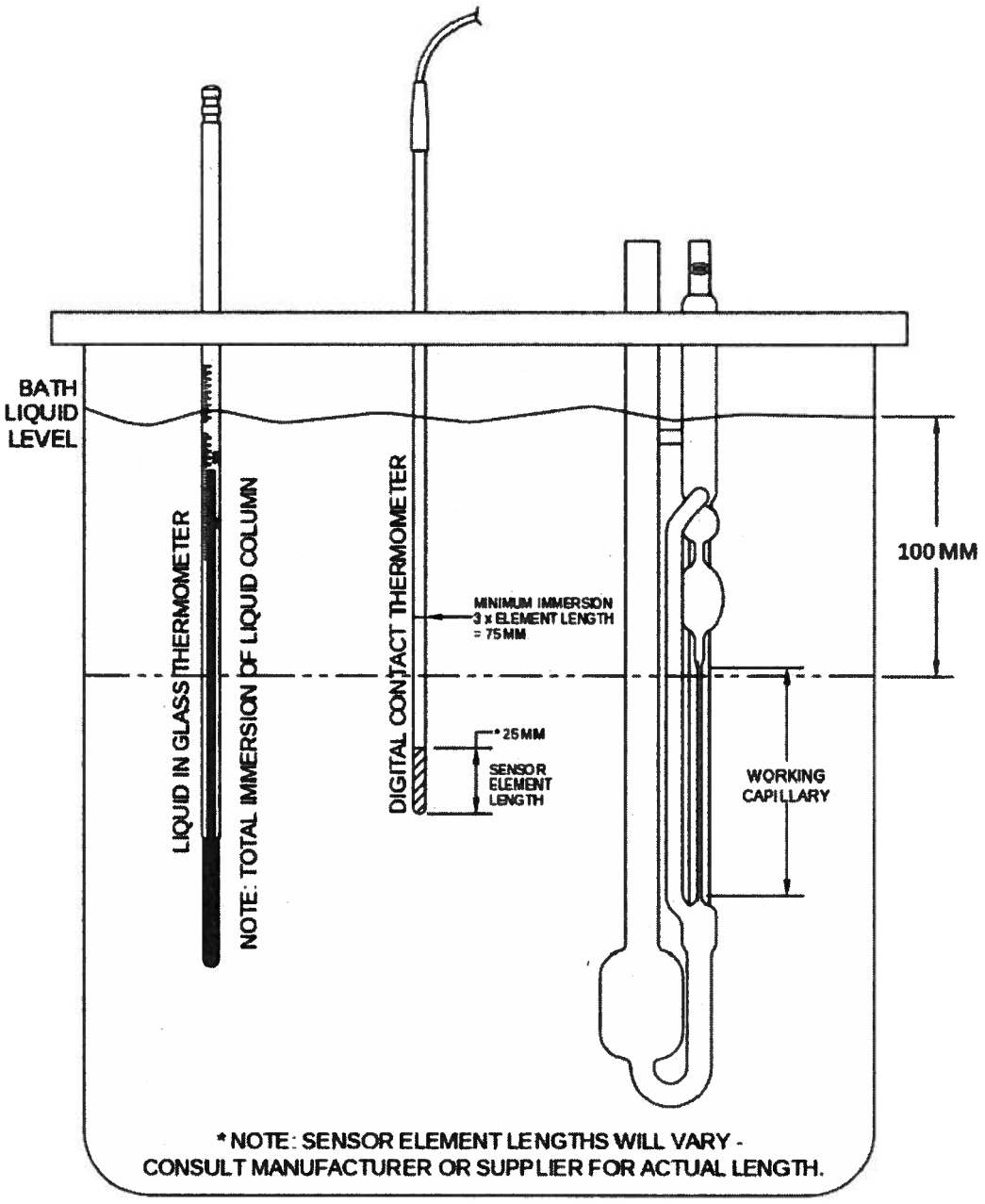


FIG. 1 Temperature Probe Immersion in Constant Temperature Bath

of equal or better accuracy. When two temperature measuring devices are used in the same bath, they shall agree within ±0.1°C.

6.4.4 When using liquid-in-glass thermometers, such as those in Table A2.1, use a magnifying device to read the thermometer to the nearest ⅕ division (for example, 0.01°C or 0.02°F) to ensure that the required test temperature and temperature control capabilities are met (see 10.1). It is recommended that thermometer readings (and any corrections supplied on the certificates of calibrations for the thermometers) be recorded on a periodic basis to demonstrate compliance with the test method requirements. This information can be quite useful, especially when investigating issues or causes relating to testing accuracy and precision.

6.5 *Timing Device*—Use any timing device that is capable of taking readings with a discrimination of 0.1 s or better and has an accuracy within ±0.07 % (see Annex A3) of the reading when tested over the minimum and maximum intervals of expected flow times.

6.5.1 Electrical timing devices may be used if the current frequency is controlled to an accuracy of 0.05 % or better. Alternating currents, as provided by some public power systems, are intermittently rather than continuously controlled.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:10:10 MDT

When used to actuate electrical timing devices, such control can cause large errors in kinematic viscosity flow time measurements.

7. Reagents and Materials

7.1 *Chromic Acid Cleaning Solution*, or a nonchromium-containing, strongly oxidizing acid cleaning solution. (**Warning**—Chromic acid is a health hazard. It is toxic, a recognized carcinogen, highly corrosive, and potentially hazardous in contact with organic materials. If used, wear a full face-shield and full-length protective clothing including suitable gloves. Avoid breathing vapor. Dispose of used chromic acid carefully as it remains hazardous. Nonchromium-containing, strongly oxidizing acid cleaning solutions are also highly corrosive and potentially hazardous in contact with organic materials, but do not contain chromium which has special disposal problems.)

7.2 *Sample Solvent*, completely miscible with the sample. Filter before use.

7.2.1 For most samples a volatile petroleum spirit or naphtha is suitable. For residual fuels, a prewash with an aromatic solvent such as toluene or xylene may be necessary to remove asphaltenic material.

7.3 *Drying Solvent*, a volatile solvent miscible with the sample solvent (see 7.2) and water (see 7.4). Filter before use.

7.3.1 Acetone is suitable. (**Warning**—Extremely flammable.)

7.4 *Water*, deionized or distilled and conforming to Specification D1193 or Grade 3 of ISO 3696. Filter before use.

8. Certified Viscosity Reference Standards

8.1 Certified viscosity reference standards shall be certified by a laboratory that has been shown to meet the requirements of ISO 17025 by independent assessment. Viscosity standards shall be traceable to master viscometer procedures described in Test Method D2162.

8.2 The uncertainty of the certified viscosity reference standard shall be stated for each certified value (k = 2, 95% confidence). See ISO 5725 or NIST 1297.

9. Calibration and Verification

9.1 *Viscometers*—Use only calibrated viscometers, thermometers, and timers as described in Section 6.

9.2 *Certified Viscosity Reference Standards* (Table A1.2)—These are for use as confirmatory checks on the procedure in the laboratory.

9.2.1 If the determined kinematic viscosity does not agree within the acceptable tolerance band, as calculated from Annex A4, of the certified value, recheck each step in the procedure, including thermometer and viscometer calibration, to locate the source of error. Annex A1 gives details of standards available.

NOTE 4—In previous issues of Test Method D445, limits of ±0.35% of the certified value have been used. The data to support the limit of ±0.35% cannot be verified. Annex A4 provides instructions on how to determine the tolerance band. The tolerance band combines both the uncertainty of the certified viscosity reference standard as well as the uncertainty of the laboratory using the certified viscosity reference standard.

9.2.1.1 As an alternative to the calculation in Annex A4, the approximate tolerance bands in Table 1 may be used.

9.2.2 The most common sources of error are caused by particles of dust lodged in the capillary bore and temperature measurement errors. It must be appreciated that a correct result obtained on a standard oil does not preclude the possibility of a counterbalancing combination of the possible sources of error.

9.3 The calibration constant, C, is dependent upon the gravitational acceleration at the place of calibration and this must, therefore, be supplied by the standardization laboratory together with the instrument constant. Where the acceleration of gravity, g, differs by more that 0.1 %, correct the calibration constant as follows:

$$C_2 = (g_2/g_1) \times C_1 \quad (1)$$

where the subscripts 1 and 2 indicate, respectively, the standardization laboratory and the testing laboratory.

10. General Procedure for Kinematic Viscosity

10.1 Adjust and maintain the viscometer bath at the required test temperature within the limits given in 6.3.1 taking account of the conditions given in Annex A2 and of the corrections supplied on the certificates of calibration for the thermometers.

10.1.1 Thermometers shall be held in an upright position under the same conditions of immersion as when calibrated.

10.1.2 In order to obtain the most reliable temperature measurement, it is recommended that two thermometers with valid calibration certificates be used (see 6.4).

10.1.3 They should be viewed with a lens assembly giving approximately five times magnification and be arranged to eliminate parallax errors.

10.2 Select a clean, dry, calibrated viscometer having a range covering the estimated kinematic viscosity (that is, a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). The flow time for manual viscometers shall not be less than 200 s or the longer time noted in Specifications D446. Flow times of less than 200 s are permitted for automated viscometers, provided they meet the requirements of 6.1.2.

10.2.1 The specific details of operation vary for the different types of viscometers listed in Table A1.1. The operating instructions for the different types of viscometers are given in Specifications D446.

10.2.2 When the test temperature is below the dew point, fill the viscometer in the normal manner as required in 11.1. To ensure that moisture does not condense or freeze on the walls

TABLE 1 Approximate Tolerance Bands

NOTE—The tolerance bands were determined using Practice D6617. The calculation is documented in Research Report RR:D02-1498.[A]

Viscosity of Reference Material, mm²/s	Tolerance Band
< 10	±0.30%
10 to 100	±0.32%
100 to 1000	±0.36%
1000 to 10 000	±0.42%
10 000 to 100 000	±0.54%
> 100 000	±0.73%

[A]Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1498.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:10:10 MDT

of the capillary, draw the test portion into the working capillary and timing bulb, place rubber stoppers into the tubes to hold the test portion in place, and insert the viscometer into the bath. After insertion, allow the viscometer to reach bath temperature, and the remove the stoppers. When performing manual viscosity determinations, do not use those viscometers which cannot be removed from the constant temperature bath for charging the sample portion.

10.2.2.1 The use of loosely packed drying tubes affixed to the open ends of the viscometer is permitted, but not required. If used, the drying tubes shall fit the design of the viscometer and not restrict the flow of the sample by pressures created in the instrument.

10.2.3 Viscometers used for silicone fluids, fluorocarbons, and other liquids which are difficult to remove by the use of a cleaning agent, shall be reserved for the exclusive use of those fluids except during their calibration. Subject such viscometers to calibration checks at frequent intervals. The solvent washings from these viscometers shall not be used for the cleaning of other viscometers.

11. Procedure for Transparent Liquids

11.1 Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity with that employed when the instrument was calibrated. If the sample is thought or known to contain fibers or solid particles, filter through a 75 μm screen, either prior to or during charging (see Specifications D446).

NOTE 5—To minimize the potential of particles passing through the filter from aggregating, it is recommended that the time lapse between filtering and charging be kept to a minimum.

11.1.1 In general, the viscometers used for transparent liquids are of the type listed in Table A1.1, A and B.

11.1.2 With certain products which exhibit *gel-like* behavior, exercise care that flow time measurements are made at sufficiently high temperatures for such materials to flow freely, so that similar kinematic viscosity results are obtained in viscometers of different capillary diameters.

11.1.3 Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Where one bath is used to accommodate several viscometers, never add or withdraw, or clean a viscometer while any other viscometer is in use for measuring a flow time.

11.1.4 Because this time will vary for different instruments, for different temperatures, and for different kinematic viscosities, establish a safe equilibrium time by trial.

11.1.4.1 Thirty minutes should be sufficient except for the highest kinematic viscosities.

11.1.5 Where the design of the viscometer requires it, adjust the volume of the sample to the mark after the sample has reached temperature equilibrium.

11.2 Use suction (if the sample contains no volatile constituents) or pressure to adjust the head level of the test sample to a position in the capillary arm of the instrument about 7 mm above the first timing mark, unless any other value is stated in the operating instructions for the viscometer. With the sample flowing freely, measure, in seconds to within 0.1 s, the time required for the meniscus to pass from the first to the second timing mark. If this flow time is less than the specified minimum (see 10.2), select a viscometer with a capillary of smaller diameter and repeat the operation.

11.2.1 Repeat the procedure described in 11.2 to make a second measurement of flow time. Record both measurements.

11.2.2 From the two measurements of flow time, calculate two determined values of kinematic viscosity.

11.2.3 If the two determined values of kinematic viscosity calculated from the flow time measurements agree within the stated determinability figure (see 17.1.1) for the product, use the average of these determined values to calculate the kinematic viscosity result to be reported. Record the result. If not, repeat the measurements of flow times after a thorough cleaning and drying of the viscometers and filtering (where required, see 11.1) of the sample until the calculated kinematic viscosity determinations agree with the stated determinability.

11.2.4 If the material or temperature, or both, is not listed in 17.1.1, use 1.5% as an estimate of the determinability.

12. Procedure for Opaque Liquids

12.1 For steam-refined cylinder oils and black lubricating oils, proceed to 12.3 ensuring a thoroughly representative sample is used. The kinematic viscosity of residual fuel oils and similar waxy products can be affected by the previous thermal history and the following procedure described in 12.1.1-12.2.2 shall be followed to minimize this.

12.1.1 In general, the viscometers used for opaque liquids are of the reverse-flow type listed in Table A1.1, C.

12.1.2 Heat in the original container, in an oven, at 60 ± 2°C for 1 h.

12.1.3 Thoroughly stir the sample with a suitable rod of sufficient length to reach the bottom of the container. Continue stirring until there is no sludge or wax adhering to the rod.

12.1.4 Recap the container tightly and shake vigorously for 1 min to complete the mixing.

12.1.4.1 With samples of a very waxy nature or oils of high kinematic viscosity, it may be necessary to increase the heating temperature above 60°C to achieve proper mixing. The sample should be sufficiently fluid for ease of stirring and shaking.

12.2 Immediately after completing 12.1.4, pour sufficient sample to fill two viscometers into a 100-mL glass flask and loosely stopper.

12.2.1 Immerse the flask in a bath of boiling water for 30 min. (**Warning**—Exercise care as vigorous boil-over can occur when opaque liquids which contain high levels of water are heated to high temperatures.)

12.2.2 Remove the flask from the bath, stopper tightly, and shake for 60 s.

12.3 Two determinations of the kinematic viscosity of the test material are required. For those viscometers that require a complete cleaning after each flow time measurement, two viscometers may be used. A single viscometer in which an immediate, repeat flow time measurement can be made without cleaning may also be used for the two measurements of flow time and calculation of kinematic viscosity. Charge two viscometers in the manner dictated by the design of the instrument. For example, for the cross-arm or the BS U-tube viscometers for opaque liquids, filter the sample through a 75-μm filter into two viscometers previously placed in the bath.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:10:10 MDT

For samples subjected to heat treatment, use a preheated filter to prevent the sample coagulating during the filtration.

12.3.1 Viscometers which are charged before being inserted into the bath may need to be preheated in an oven prior to charging the sample. This is to ensure that the sample will not be cooled below test temperature.

12.3.2 After 10 min, adjust the volume of the sample (where the design of the viscometer requires) to coincide with the filling marks as in the viscometer specifications (see Specifications D446).

12.3.3 Allow the charged viscometers enough time to reach the test temperature (see 12.3.1). Where one bath is used to accommodate several viscometers, never add or withdraw, or clean a viscometer while any other viscometer is in use for measuring flow time.

12.4 With the sample flowing freely, measure in seconds to within 0.1 s, the time required for the advancing ring of contact to pass from the first timing mark to the second. Record the measurement.

12.4.1 In the case of samples requiring heat treatment described in 12.1 through 12.2.1, complete the measurements of flow time within 1 h of completing 12.2.2. Record the measured flow times.

12.5 Calculate kinematic viscosity, ν, in mm^2/s, from each measured flow time. Regard these as two determined values of kinematic viscosity.

12.5.1 For residual fuel oils, if the two determined values of kinematic viscosity agree within the stated determinability figure (see 17.1.1), use the average of these determined values to calculate the kinematic viscosity result to be reported. Record the result. If the calculated kinematic viscosities do not agree, repeat the measurements of flow times after thorough cleaning and drying of the viscometers and filtering of the sample. If the material or temperature, or both, is not listed in 17.1.1, for temperatures between 15 and 100°C use as an estimate of the determinability 1.0 %, and 1.5 % for temperatures outside this range; it must be realized that these materials can be non-Newtonian, and can contain solids which can come out of solution as the flow time is being measured.

13. Cleaning of Viscometer

13.1 Between successive determinations of kinematic viscosity, clean the viscometer thoroughly by several rinsings with the sample solvent, followed by the drying solvent (see 7.3). Dry the tube by passing a slow stream of filtered dry air through the viscometer for 2 min or until the last trace of solvent is removed.

13.2 Periodically clean the viscometer with the cleaning solution (**Warning**—see 7.1), for several hours to remove residual traces of organic deposits, rinse thoroughly with water (7.4) and drying solvent (see 7.3), and dry with filtered dry air or a vacuum line. Remove any inorganic deposits by hydrochloric acid treatment before the use of cleaning acid, particularly if the presence of barium salts is suspected. (**Warning**—It is essential that alkaline cleaning solutions are not used as changes in the viscometer calibration can occur.)

14. Calculation

14.1 Calculate each of the determined kinematic viscosity values, ν_1 and ν_2, from the measured flow times, t_1 and t_2, and the viscometer constant, C, by means of the following equation:

$$\nu_{1,2} = C \cdot t_{1,2} \quad (2)$$

where:

$\nu_{1,2}$ = determined kinematic viscosity values for ν_1 and ν_2, respectively, mm^2/s,
C = calibration constant of the viscometer, mm^2/s^2, and
$t_{1,2}$ = measured flow times for t_1 and t_2, respectively, s.

Calculate the kinematic viscosity result, ν, as an average of ν_1 and ν_2 (see 11.2.3 and 12.5.1).

14.2 Calculate the dynamic viscosity, η, from the calculated kinematic viscosity, ν, and the density, ρ, by means of the following equation:

$$\eta = \nu \times \rho \times 10^{-3} \quad (3)$$

where:

η = dynamic viscosity, mPa·s,
ρ = density, kg/m^3, at the same temperature used for the determination of the kinematic viscosity, and
ν = kinematic viscosity, mm^2/s.

14.2.1 The density of the sample can be determined at the test temperature of the kinematic viscosity determination by an appropriate method such as Test Methods D1217, D1480, or D1481.

15. Expression of Results

15.1 Report the test results for the kinematic or dynamic viscosity, or both, to four significant figures, together with the test temperature.

16. Report

16.1 Report the following information:

16.1.1 Type and identification of the product tested,

16.1.2 Reference to this test method or a corresponding international standard,

16.1.3 Result of the test (see Section 15),

16.1.4 Any deviation, by agreement or otherwise, from the procedure specified,

16.1.5 Date of the test, and

16.1.6 Name and address of the test laboratory.

17. Precision and Bias

17.1 *Comparison of Determined Values*:

17.1.1 *Determinability (d)*—The difference between successive determined values obtained by the same operator in the same laboratory using the same apparatus for a series of operations leading to a single result, would in the long run, in the normal and correct operation of this test method, exceed the values indicated only in one case in twenty:

Base oils at 40 and 100°C[9]	0.0020 y	(0.20 %)
Formulated oils at 40 and 100°C[10]	0.0013 y	(0.13 %)
Formulated oils at 150°C[11]	0.015 y	(1.5 %)
Petroleum wax at 100°C[12]	0.0080 y	(0.80 %)
Residual fuel oils at 80 and 100°C[13]	0.011 (y + 8)	
Residual fuel oils at 50°C[13]	0.017 y	(1.7 %)
Additives at 100°C[14]	$0.00106\ y^{1.1}$	

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:10:10 MDT

Gas oils at 40°C[15]	0.0013 (y+1)	
Jet fuels at –20°C[16]	0.0018 y	(0.18 %)

where: y is the average of determined values being compared.

17.2 *Comparison of Results*:

17.2.1 *Repeatability (r)*—The difference between successive results obtained by the same operator in the same laboratory with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values indicated only in one case in twenty:

Base oils at 40 and 100°C[9]	0.0011 x	(0.11 %)
Formulated oils at 40 and 100°C[10]	0.0026 x	(0.26 %)
Formulated oils at 150°C[11]	0.0056 x	(0.56 %)
Petroleum wax at 100°C[12]	0.0141 $x^{1.2}$	
Residual fuel oils at 80 and 100°C[13]	0.013 (x + 8)	
Residual oils at 50°C[13]	0.015 x	(1.5 %)
Additives at 100°C[14]	0.00192 $x^{1.1}$	
Gas oils at 40°C[15]	0.0043 (x+1)	
Jet fuels at –20°C[16]	0.007 x	(0.7 %)

where: x is the average of results being compared.

17.2.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators working in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values indicated below only in one case in twenty.

Base oils at 40 and 100°C[9]	0.0065 x	(0.65 %)
Formulated oils at 40 and 100°C[10]	0.0076 x	(0.76 %)
Formulated oils at 150°C[11]	0.018 x	(1.8 %)
Petroleum wax at 100°C[12]	0.0366 $x^{1.2}$	
Residual fuel oils at 80 and 100°C[13]	0.04 (x + 8)	
Residual oils at 50°C[13]	0.074 x	(7.4 %)
Additives at 100°C[14]	0.00862 $x^{1.1}$	
Gas oils at 40°C[15]	0.0082 (x+1)	
Jet fuels at –20°C[16]	0.019 x	(1.9 %)

where: x is the average of results being compared.

17.3 The precision for used oils has not been determined but is expected to be poorer than that for formulated oils. Because of the extreme variability of such used oils, it is not anticipated that the precision of used oils will be determined.

17.4 The precision for specific automated viscometers has not been determined. However, an analysis has been made of a large data set including both automated and manual viscometers over the temperature range of 40 to 100°C. The reproducibility of automated viscometer data is not statistically significantly different than the reproducibility of manual viscometer data. It is also shown that there is no bias of the automated data in comparison to the manual data.[17]

18. Keywords

18.1 dynamic viscosity; kinematic viscosity; viscometer; viscosity

[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR:D02-1331 and RR:D02-1132. These precision values were obtained by statistical examination of interlaboratory results from six mineral oils (base oils without additive package) in the range from 8 to 1005 mm^2/s at 40°C and from 2 to 43 mm^2/s at 100°C, and were first published in 1989. Request See Guide D6074.

[10] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1332. These precision values were obtained by statistical examination of interlaboratory results from seven fully formulated engine oils in the range from 36 to 340 mm^2/s at 40°C and from 6 to 25 mm^2/s at 100°C, and were first published in 1991. See Guide D6071.

[11] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1333. These precision values were obtained by statistical examination of interlaboratory results for eight fully formulated engine oils in the range from 7 to 19 mm^2/s at 150°C, and first published in 1991. See Guide D6074.

[12] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1334. These precision values were obtained by statistical examination of interlaboratory results from five petroleum waxes in the range from 3 to 16 mm^2/s at 100°C, and were first published in 1988.

[13] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1198. These precision values were obtained by statistical examination of interlaboratory results from fourteen residual fuel oils in the range from 30 to 1300 mm^2/s at 50°C and from 5 to 170 mm^2/s at 80 and 100°C, and were first published in 1984.

[14] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1421. These precision values were obtained by statistical examination of interlaboratory results from eight additives in the range from 145 to 1500 mm^2/s at 100°C and were first available in 1997.

[15] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1422. These precision values were obtained by statistical examination of interlaboratory results from eight gas oils in the range from 1 to 13 mm^2/s at 40°C and were first available in 1997.

[16] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1420. These precision values were obtained by statistical examination of interlaboratory results from nine jet fuels in the range from 4.3 to 5.6 mm^2/s at– 20°C and were first available in 1997.

[17] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1498.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:10:10 MDT

ANNEXES

(Mandatory Information)

A1. VISCOMETER TYPES AND CERTIFIED VISCOSITY REFERENCE STANDARDS

A1.1 Viscometer Types

A1.1.1 Table A1.1 lists capillary viscometers commonly in use for viscosity determinations on petroleum products. For specifications, operating instructions, and calibration, refer to specifications in Specifications D446.

A1.1.2 Table A1.2 lists certified viscosity reference standards.

TABLE A1.1 Viscometer Types

Viscometer Identification	Kinematic Viscosity Range,[A] mm^2/s
A. Ostwald Types for Transparent Liquids	
Cannon-Fenske routine[B]	0.5 to 20 000
Zeitfuchs	0.6 to 3 000
BS/U-tube[B]	0.9 to 10 000
BS/U/M miniature	0.2 to 100
SIL[B]	0.6 to 10 000
Cannon-Manning semi-micro	0.4 to 20 000
Pinkevitch[B]	0.6 to 17 000
B. Suspended-level Types for Transparent Liquids	
BS/IP/SL[B]	3.5 to 100 000
BS/IP/SL(S)[B]	1.05 to 10 000
BS/IP/MSL	0.6 to 3 000
Ubbelohde[B]	0.3 to 100 000
FitzSimons	0.6 to 1 200
Atlantic[B]	0.75 to 5 000
Cannon-Ubbelohde(A), Cannon Ubbelohde dilution[B](B)	0.5 to 100 000
Cannon-Ubbelohde semi-micro	0.4 to 20 000
C. Reverse-flow Types for Transparent and Opaque Liquids	
Cannon-Fenske opaque	0.4 to 20 000
Zeitfuchs cross-arm	0.6 to 100 000
BS/IP/RF U-tube reverse-flow	0.6 to 300 000
Lantz-Zeitfuchs type reverse-flow	60 to 100 000

[A] Each range quoted requires a series of viscometers. To avoid the necessity of making a kinetic energy correction, these viscometers are designed for a flow time in excess of 200 s except where noted in Specifications D446.

[B] In each of these series, the minimum flow time for the viscometers with lowest constants exceeds 200 s.

TABLE A1.2 Certified Viscosity Reference Standards

Designation	Approximate Kinematic Viscosity, mm^2/s					
	20°C	25°C	40°C	50°C	80 °C	100°C
S3	4.6	4.0	2.9	...	...	1.2
S6	11	8.9	5.7	...	...	1.8
S20	44	34	18	...	...	3.9
S60	170	120	54	...	...	7.2
S200	640	450	180	...	...	17
S600	2400	1600	520	280	67	32
S2000	8700	5600	1700	...	...	75
S8000	37 000	23 000	6700	...	...	...
S30 000	...	81 000	23 000	11 000	...	...

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:10:10 MDT

A2. KINEMATIC VISCOSITY TEST THERMOMETERS

A2.1 Short-Range Specialized Thermometer

A2.1.1 Use a short-range specialized thermometer conforming to the generic specification given in Table A2.1 and Table A2.2 and to one of the designs shown in Fig. A2.1.

A2.1.2 The difference in the designs rests mainly in the position of the ice point scale. In Design A, the ice point is within the scale range, in Design B, the ice point is below the scale range, and in Design C, the ice point is above the scale range.

A2.2 Calibration

A2.2.1 Use liquid-in-glass thermometers with an accuracy after correction of 0.02°C or better, calibrated by a laboratory meeting the requirements of ISO 9000 or ISO 17025, and carrying certificates confirming that the calibration is traceable to a national standard. As an alternative, use thermometric devices such as platinum resistance thermometers, of equal or better accuracy, with the same certification requirements.

A2.2.2 The scale correction of liquid-in-glass thermometers can change during storage and use, and therefore regular re-calibration is required. This is most conveniently achieved in a working laboratory by means of a re-calibration of the ice point, and all of the main scale corrections altered for the change seen in the ice point.

A2.2.2.1 The interval for ice-point recalibration shall be no longer than six months (see NIST GMP 11). For new thermometers, monthly checking for the first six months is recommended. A change of one or more scale divisions in the ice point means that the thermometer may have been overheated or damaged, and it may be out of calibration. Such thermometers shall be removed from service until inspected, or recalibrated, or both. A complete recalibration of the thermometer, while permitted, is not necessary in order to meet the accuracy ascribed to this design thermometer (see NIST Special Publication 819). Any change in ice-point correction shall be added to the other corrections of the original Report of Calibration.

A2.2.2.2 Other thermometric devices, if used, will also require periodic recalibration. Keep records of all recalibration.

A2.2.3 *Procedure for Ice-point Recalibration of Liquid-in-glass Thermometers.*

A2.2.3.1 Unless otherwise listed on the certificate of calibration, the recalibration of calibrated kinematic viscosity thermometers requires that the ice-point reading shall be taken within 60 min after being at test temperature for not less than 3 min.

A2.2.3.2 Select clear pieces of ice, preferably made from distilled or pure water. Discard any cloudy or unsound portions. Rinse the ice with distilled water and shave or crush into small pieces, avoiding direct contact with the hands or any chemically unclean objects. Fill the Dewar vessel with the crushed ice and add sufficient water to form a slush, but not enough to float the ice. As the ice melts, drain off some of the water and add more crushed ice. Insert the thermometer, and pack the ice gently about the stem, to a depth approximately one scale division below the 0°C graduation.

A2.2.3.3 After at least 3 min have elapsed, tap the thermometer gently and repeatedly at right angles to its axis while making observations. Successive readings taken at least 1 min apart shall agree within 0.005°C.

A2.2.3.4 Record the ice-point readings and determine the thermometer correction at this temperature from the mean reading. If the correction is found to be higher or lower than that corresponding to a previous calibration, change the correction at all other temperatures by the same value.

A2.2.3.5 During the procedure, apply the following conditions:

(*1*) The thermometer shall be supported vertically.

(*2*) View the thermometer with an optical aid that gives a magnification of approximately five and also eliminates parallax.

(*3*) Express the ice-point reading to the nearest 0.005°C.

TABLE A2.1 General Specification for Thermometers

NOTE—Table A2.2 gives a range of ASTM, IP, and ASTM/IP thermometers that comply with the specification in Table A2.1, together with their designated test temperatures. See Specification E1 and Test Method E77.

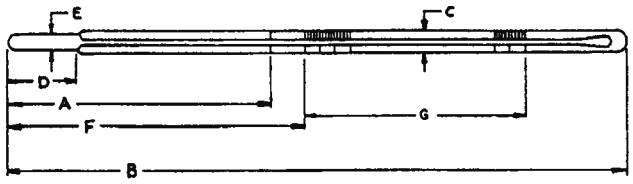
Immersion		Total
Scale marks:		
Subdivisions	°C	0.05
Long lines at each	°C	0.1 and 0.5
Numbers at each	°C	1
Maximum line width	mm	0.10
Scale error at test temperature, max	°C	0.1
Expansion chamber:		
Permit heating to	°C	105 up to 90, 120 between 90 and 95 130 between 95 and 105, 170 above 105
Total length	mm	300 to 310
Stem outside diameter	mm	6.0 to 8.0
Bulb length	mm	45 to 55
Bulb outside diameter	mm	no greater than stem
Length of scale range	mm	40 to 90

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

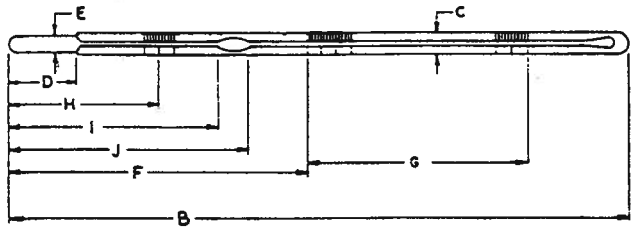
Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:10:10 MDT

TABLE A2.2 Complying Thermometers

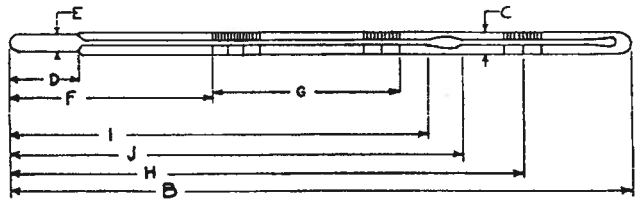
Thermometer No.	Test Temperature		Thermometer No.	Test Temperature	
	°C	°F		°C	°F
ASTM 132C, IP 102C	150		ASTM 128C, F/IP 33C	0	32
ASTM 110C, F/IP 93C	135	275	ASTM 72C, F/IP 67C	−17.8	0
ASTM 121C/IP 32C	98.9, 100	210, 212	ASTM 127C/IP 99C	−20	−4
			ASTM 126C, F/IP 71C	−26.1	−20
ASTM 129C, F/IP 36C	93.3	200	ASTM 73C, F/IP 68C	−40	−40
ASTM 48C, F/IP 90C	82.2	180	ASTM 74C, F/IP 69C	−53.9	−65
IP 100C	80				
ASTM 47C, F/IP 35C	60	140			
ASTM 29C, F/IP 34C	54.4	130			
ASTM 46C F/IP 66C	50	122			
ASTM 120C/IP 92C	40				
ASTM 28C, F/IP 31C	37.8	100			
ASTM 118C, F	30	86			
ASTM 45C, F/IP 30C	25	77			
ASTM 44C, F/IP 29C	20	68			



(a)



(b)



(c)

FIG. A2.1 Thermometer Designs

A2.2.4 When in use, immerse the thermometric device to the same depth as when it was fully calibrated. For example, if a liquid-in-glass thermometer was calibrated at the normal total immersion condition, it shall be immersed to the top of the mercury column with the remainder of the stem and the expansion volume at the uppermost end exposed to room

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:10:10 MDT

temperature and pressure. In practice, this means that the top of the mercury column shall be within a length equivalent to four scale divisions of the surface of the medium whose temperature is being measured.

A2.2.4.1 If this condition cannot be met, then an extra correction may be necessary.

A3. TIMER ACCURACY

A3.1 Regularly check timers for accuracy and maintain records of such checks.

A3.1.1 Time signals as broadcast by the National Institute of Standards and Technology are a convenient and primary standard reference for calibrating timing devices. The following can be used to an accuracy of 0.1 s:

WWV	Fort Collins, CO	2.5, 5, 10, 15, 20 MHz
WWVH	Kauai, HI	2.5, 5, 10, 15, MHz
CHU	Ottawa, Canada	3.33, 7.335, 14.67 MHz

A3.1.2 Radio broadcast of voice and audio on a telephone line at phone 303-499-7111. Additional time services are available from the National Institute of Standards and Technology.

A4. CALCULATION OF ACCEPTABLE TOLERANCE ZONE (BAND) TO DETERMINE CONFORMANCE WITH A CERTIFIED REFERENCE MATERIAL

NOTE A4.1—These calculations are based on Practice D6617.

A4.1 Determine the standard deviation for site uncertainty, σ_{site}, from a laboratory quality control program. (See Practice D6299.)

A4.1.1 If the standard deviation for site uncertainty, σ_{site}, is not known, use the value 0.19%.

A4.2 Determine the combined extended uncertainty (CEU) of the accepted reference value (ARV) of the certified reference material (CRM) from the supplier's label or included documentation. In this test method, the CRM is the Certified Viscosity Reference Standard (CVRS) as defined in 9.2.

NOTE A4.2—Combined Extended Uncertainty (CEU) is equivalent to Expanded Uncertainty (U). See NIST Technical Note 1297.

A4.3 Calculate the standard error of the accepted reference value (SE_{ARV}) by dividing the CEU by the coverage factor, k, listed on the supplier's label or included documentation.

$$SE_{ARV} = \frac{CEU_{ARV}}{k} \quad \text{(A4.1)}$$

NOTE A4.3—Standard Error (SE_{ARV}) is equivalent to Combined Standard Uncertainty (U_C). See NIST Technical Note 1297.

A4.3.1 If the coverage factor, k, is not known, use the value 2.

A4.4 Construct the acceptable tolerance zone:

$$TZ = \pm 1.44\sqrt{\sigma_{site}^2 + SE_{ARV}^2} \quad \text{(A4.2)}$$

A4.5 Worked out example for kinematic viscosity zone (band):

$$\sigma_{site} = 0.19\ \% \text{ (default value from A4.1.1)} \quad \text{(A4.3)}$$

$$CEU_{ARV} = 0.22\ \% \text{ (from the CVRS label or documentation)} \quad \text{(A4.3)}$$

$$k = 2 \text{ (from the CVRS label or documentation)} \quad \text{(A4.3)}$$

$$SE_{ARV} = \frac{0.22}{2} = 0.11 \quad \text{(A4.3)}$$

$$TZ = \pm 1.44\sqrt{0.19^2 + 0.11^2} = \pm 1.44\sqrt{0.0361 + 0.0121} = \pm 0.32\ \% \quad \text{(A4.3)}$$

A4.5.1 In this example, the tolerance zone will be ±0.32 % of the certified viscosity reference standard value on the report of test or bottle label. If this site uses a CVRS (for example) with a kinematic viscosity of 33.98 mm^2/s, the TZ = 33.87 to 34.09 mm^2/s, with 95 % certainty. Viscosity measurements made with this CVRS at that site should fall within that tolerance zone (band) 19 out of 20 times.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:10:10 MDT

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D445–11) that may impact the use of this standard. (Approved May 1, 2011.)

(*1*) Revised 6.4 to define requirements for electronic temperature measurement.

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D445–10) that may impact the use of this standard. (Approved Jan. 1, 2011.)

(*1*) Revised 3.3.3 and 3.3.4 to update definitions.
(*2*) Added discussions under 3.3.4 to provide continuity between the old and the new definitions.
(*3*) Added reference to the International Vocabulary of Metrology.

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D445–09) that may impact the use of this standard. (Approved Oct. 1, 2010.)

(*1*) Revised Annex A4 to clarify terms and add a worked example.
(*2*) Revised 1.4 to clarify SI units used in the test method and add units reference for users.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the ASTM website (www.astm.org/COPYRIGHT/).

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS
Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:10:10 MDT

Designation: D854 – 10

Standard Test Methods for Specific Gravity of Soil Solids by Water Pycnometer[1]

This standard is issued under the fixed designation D854; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 These test methods cover the determination of the specific gravity of soil solids that pass the 4.75-mm (No. 4) sieve, by means of a water pycnometer. When the soil contains particles larger than the 4.75-mm sieve, Test Method C127 shall be used for the soil solids retained on the 4.75-mm sieve and these test methods shall be used for the soil solids passing the 4.75-mm sieve.

1.1.1 Soil solids for these test methods do not include solids which can be altered by these methods, contaminated with a substance that prohibits the use of these methods, or are highly organic soil solids, such as fibrous matter which floats in water.

NOTE 1—The use of Test Method D5550 may be used to determine the specific gravity of soil solids having solids which readily dissolve in water or float in water, or where it is impracticable to use water.

1.2 Two methods for performing the specific gravity are provided. The method to be used shall be specified by the requesting authority, except when testing the types of soils listed in 1.2.1

1.2.1 *Method A*—Procedure for Moist Specimens, described in 9.2. This procedure is the preferred method. For organic soils; highly plastic, fine grained soils; tropical soils; and soils containing halloysite, Method A shall be used.

1.2.2 *Method B*—Procedure for Oven-Dry Specimens, described in 9.3.

1.3 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.3.1 The procedures used to specify how data are collected/recorded and calculated in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of these test methods to consider significant digits used in analysis methods for engineering design.

1.4 The values stated in SI units are to be regarded as standard. The inch-pound units given in parentheses are mathematical conversions which are provided for information purposes only and are not considered standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C127 Test Method for Density, Relative Density (Specific Gravity), and Absorption of Coarse Aggregate

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D2487 Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D5550 Test Method for Specific Gravity of Soil Solids by Gas Pycnometer

D6026 Practice for Using Significant Digits in Geotechnical Data

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to

[1] This standard is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity and Density Characteristics of Soils.

Current edition approved Jan. 1, 2010. Published March 2010. Originally approved in 1945. Last previous edition approved in 2006 as D854 – 06 ε1. DOI: 10.1520/D0854-10.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard.**

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/28/2012 13:24:19 MDT

Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions*—For definitions of technical terms used in these test methods, refer to Terminology D653.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *specific gravity of soil solids, G_s, n*—the ratio of the mass of a unit volume of a soil solids to the mass of the same volume of gas-free distilled water at 20°C.

4. Significance and Use

4.1 The specific gravity of a soil solids is used in calculating the phase relationships of soils, such as void ratio and degree of saturation.

4.1.1 The specific gravity of soil solids is used to calculate the density of the soil solids. This is done by multiplying its specific gravity by the density of water (at proper temperature).

4.2 The term soil solids is typically assumed to mean naturally occurring mineral particles or soil like particles that are not readily soluble in water. Therefore, the specific gravity of soil solids containing extraneous matter, such as cement, lime, and the like, water-soluble matter, such as sodium chloride, and soils containing matter with a specific gravity less than one, typically require special treatment (see Note 1) or a qualified definition of their specific gravity.

4.3 The balances, pycnometer sizes, and specimen masses are established to obtain test results with three significant digits.

NOTE 2—The quality of the result produced by these test methods is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of these test methods are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

5. Apparatus

5.1 *Pycnometer*—The water pycnometer shall be either a stoppered flask, stoppered iodine flask, or volumetric flask with a minimum capacity of 250 mL. The volume of the pycnometer must be 2 to 3 times greater than the volume of the soil-water mixture used during the deairing portion of the test.

5.1.1 The stoppered flask mechanically sets the volume. The stoppered iodine flask has a flared collar that allows the stopper to be placed at an angle during thermal equilibration and prevents water from spilling down the sides of the flask when the stopper is installed. The wetting the outside of the flask is undesirable because it creates changes in the thermal equilibrium. When using a stopper flask, make sure that the stopper is properly labeled to correspond to the flask.

5.2 *Balance*—A balance meeting the requirements of Guide D4753 for a balance of 0.01 g readability. When using the 250–mL pycnometers, the balance capacity shall be at least 500 g and when using the 500–mL pycnometers, the balance capacity shall be at least 1000 g.

5.3 *Drying Oven*—Thermostatically controlled oven, capable of maintaining a uniform temperature of 110 ± 5°C throughout the drying chamber. These requirements usually require the use of a forced-draft oven.

5.4 *Thermometric Device*, capable of measuring the temperature range within which the test is being performed, having a readability of 0.1°C and a maximum permissible error of 0.5°C. The device must be capable of being immersed in the sample and calibration solutions to a depth ranging between 25 and 80 mm. Full immersion thermometers shall not be used. To ensure the accuracy of the thermometric device, the thermometric device shall be standardized by comparison to a NIST traceable thermometric device. The standardization shall include at least one temperature reading within the range of testing. The thermometric device shall be standardized at least once every twelve months.

5.5 *Desiccator*—A desiccator cabinet or large desiccator jar of suitable size containing silica gel or anhydrous calcium sulfate.

NOTE 3—It is preferable to use a desiccant that changes color to indicate when it needs reconstitution.

5.6 *Entrapped Air Removal Apparatus*—To remove entrapped air (deairing process), use one of the following:

5.6.1 *Hot Plate or Bunsen Burner*, capable of maintaining a temperature adequate to boil water.

5.6.2 *Vacuum System*, a vacuum pump or water aspirator, capable of producing a partial vacuum of 100 mm of mercury (Hg) or less absolute pressure. **Warning**—Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website – http://www.epa.gov/mercury/faq.htm - for additional information. Users should be aware that selling mercury and/or mercury containing products into your state may be prohibited by state law.

NOTE 4—A partial vacuum of 100 mm Hg absolute pressure is approximately equivalent to a 660 mm (26 in.) Hg reading on vacuum gauge at sea level.

5.7 *Insulated Container*—A Styrofoam cooler and cover or equivalent container that can hold between three and six pycnometers plus a beaker (or bottle) of deaired water, and a thermometer. This is required to maintain a controlled temperature environment where changes will be uniform and gradual.

5.8 *Funnel*—A non-corrosive smooth surface funnel with a stem that extends past the calibration mark on the volumetric flask or stoppered seal on the stoppered flasks. The diameter of the stem of the funnel must be large enough that soil solids will easily pass through.

5.9 *Pycnometer Filling Tube with Lateral Vents (optional)*—A device to assist in adding deaired water to the pycnometer without disturbing the soil-water mixture. The device may be fabricated as follows. Plug a 6 to 10-mm (¼ to ⅜ in.) diameter plastic tube at one end and cut two small vents (notches) just above the plug. The vents should be perpendicular to the axis of the tube and diametrically opposed. Connect a valve to the other end of the tube and run a line to the valve from a supply of deaired water.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905805100, User=Isaac, Rick
Not for Resale, 03/28/2012 13:24:19 MDT

5.10 *Sieve*— 4.75 mm (No. 4) conforming to the requirements of Specification E11.

5.11 *Blender (optional)*—A blender with mixing blades built into the base of the mixing container.

5.12 *Miscellaneous Equipment*, such as a computer or calculator (optional), specimen dishes, and insulated gloves.

6. Reagents

6.1 *Purity of Water*—Distilled water is used in this test method. This water may be purchased and is readily available at most grocery stores; hereafter, distilled water will be referred to as water.

7. Test Specimen

7.1 The test specimen may be moist or oven-dry soil and shall be representative of the soil solids that pass the 4.75-mm (No. 4) sieve in the total sample. Table 1 gives guidelines on recommended dry soil mass versus soil type and pycnometer size.

7.1.1 Two important factors concerning the amount of soil solids being tested are as follows. First, the mass of the soil solids divided by its specific gravity will yield four-significant digits. Secondly, the mixture of soil solids and water is a slurry not a highly viscous fluid (thick paint) during the deairing process.

8. Calibration of Pycnometer

8.1 Determine the mass of the clean and dry pycnometer to the nearest 0.01 g (typically five significant digits). Repeat this determination five times. One balance should be used for all of the mass measurements. Determine and record the average and standard deviation. The standard deviation shall be less than or equal to 0.02 g. If it is greater, attempt additional measurements or use a more stable or precise balance.

8.2 Fill the pycnometer with deaired water to above or below the calibration mark depending on the type of pycnometer and laboratory preference to add or remove water.

8.2.1 It is recommended that water be removed to bring the water level to the calibration mark. The removal method reduces the chances of altering the thermal equilibrium by reducing the number of times the insulated container is opened.

8.2.2 The water must be deaired to ensure that there are no air bubbles in the water. The water may be deaired using either boiling, vacuum, combination of vacuum and heat, or a deairing device. This deaired water should not be used until it has equilibrated to room temperature. Also, this water shall be added to the pycnometer following the guidance given in 9.6.

8.3 Up to six pycnometers can be calibrated concurrently in each insulated container. Put the pycnometer(s) into a covered insulated container along with the thermometric device (or the temperature sensing portion of the thermometric device), a beaker (or bottle) of deaired water, stopper(s) (if a stoppered pycnometer is being used), and either an eyedropper or pipette. Let the pycnometer(s) come to thermal equilibrium (for at least 3 h). The equilibrium temperature should be within 4°C of room temperature and between 15 and 30°C.

TABLE 1 Recommended Mass for Test Specimen

Soil Type	Specimen Dry Mass (g) When Using 250 mL Pycnometer	Specimen Dry Mass (g) When Using 500 mL Pycnometer
SP, SP-SM	60 ± 10	100 ± 10
SP-SC, SM, SC	45 ± 10	75 ± 10
Silt or Clay	35 ± 5	50 ± 10

8.4 Move the insulated container near the balance or vice versa. Open the container and remove one pycnometer. Only the rim of the pycnometer shall be touched as to prevent the heat from handling changing the thermal equilibrium. Either work in the container or place the pycnometer on an insulated block (Styrofoam) while making water level adjustments.

8.4.1 If using a volumetric flask as a pycnometer, adjust the water to the calibration mark, with the bottom of the meniscus level with the mark. If water has to be added, use the thermally equilibrated water from the insulated container. If water has to be removed, use a small suction tube or paper towel. Check for and remove any water beads on the pycnometer stem or on the exterior of the flask. Measure and record the mass of pycnometer and water to the nearest 0.01 g.

8.4.2 If a stoppered flask is used, adjust the water to prevent entrapment of any air bubbles below the stopper during its placement. If water has to be added, use the thermally equilibrated water from the insulated container. Then, place the stopper in the bottle. If water has to be removed, before or after inserting the stopper, use an eyedropper. Dry the rim using a paper towel. Be sure the entire exterior of the flask is dry. Measure and record the mass of pycnometer and water to the nearest 0.01 g.

8.5 Measure and record the temperature of the water to the nearest 0.1°C using the thermometric device that has been thermally equilibrated in the insulated container. Insert the thermometric device (or the temperature sensing portion of the thermometric device) to the appropriate depth of immersion (see 5.4). Return the pycnometer to the insulated container. Repeat the measurements for all pycnometers in the container.

8.6 Readjust the water level in each pycnometer to above or below the calibration line or empty the pycnometer and fill to the above or below the calibration line. Allow the pycnometers to thermally equilibrate (for at least 3 h) in the covered insulated container. Adjust the water level to the calibration line by removing water from the pycnometer or by filling the pycnometer to the calibration mark with the thermally equilibrated deaired water from the insulated container. Measure and record the mass and temperature of the filled pycnometer.

8.6.1 Repeat the procedure in 8.6 until a total of five independent measurements of the mass of the filled pycnometer and temperature readings are obtained. The temperatures do not need to bracket any particular temperature range.

8.7 Using each of these five data points, compute the calibrated volume of each pycnometer, V_p, using the following equation:

$$V_p = \frac{(M_{pw,c} - M_p)}{\rho_{w,c}} \quad (1)$$

where:

$M_{pw,c}$ = the mass of the pycnometer and water at the calibration temperature, g,

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/28/2012 13:24:19 MDT

M_p = the average mass of the dry pycnometer at calibration, g, and

$\rho_{w,c}$ = the mass density of water at the calibration temperature g/mL, (Table 2).

8.8 Calculate the average and the standard deviation of the five volume determinations. The standard deviation shall be less than or equal to 0.05 mL (rounded to two decimal places). If the standard deviation is greater than 0.05 mL, the calibration procedure has too much variability and will not yield accurate specific gravity determinations. Evaluate areas of possible refinement (adjusting the volume to the calibration mark, achieving temperature equilibrium, measuring temperature, deairing method or changing to the stoppered flasks) and revise the procedure until the standard deviation is less than or equal to 0.05 mL.

9. Procedure

9.1 *Pycnometer Mass*—Using the same balance used to calibrate the pycnometer, verify that the mass of the pycnometer is within 0.06 g of the average calibrated mass. If it is not, re-calibrate the dry mass of the pycnometer.

9.2 *Method A—Procedure for Moist Specimens:*

9.2.1 Determine the water content of a portion of the sample in accordance with Test Method D2216. Using this water

TABLE 2 Density of Water and Temperature Coefficient (*K*) for Various Temperatures[A,B]

Temperature (°C)	Density (g/mL)[C]	Temperature Coefficient (*K*)	Temperature (°C)	Density (g/mL)[C]	Temperature Coefficient (*K*)	Temperature (°C)	Density (g/mL)[C]	Temperature Coefficient (*K*)	Temperature (°C)	Density (g/mL)[C]	Temperature Coefficient (*K*)
15.0	0.99910	1.00090	16.0	0.99895	1.00074	17.0	0.99878	1.00057	18.0	0.99860	1.00039
.1	0.99909	1.00088	.1	0.99893	1.00072	.1	0.99876	1.00055	.1	0.99858	1.00037
.2	0.99907	1.00087	.2	0.99891	1.00071	.2	0.99874	1.00054	.2	0.99856	1.00035
.3	0.99906	1.00085	.3	0.99890	1.00069	.3	0.99872	1.00052	.3	0.99854	1.00034
.4	0.99904	1.00084	.4	0.99888	1.00067	.4	0.99871	1.00050	.4	0.99852	1.00032
.5	0.99902	1.00082	.5	0.99886	1.00066	.5	0.99869	1.00048	.5	0.99850	1.00030
.6	0.99901	1.00080	.6	0.99885	1.00064	.6	0.99867	1.00047	.6	0.99848	1.00028
.7	0.99899	1.00079	.7	0.99883	1.00062	.7	0.99865	1.00045	.7	0.99847	1.00026
.8	0.99898	1.00077	.8	0.99881	1.00061	.8	0.99863	1.00043	.8	0.99845	1.00024
.9	0.99896	1.00076	.9	0.99879	1.00059	.9	0.99862	1.00041	.9	0.99843	1.00022
19.0	0.99841	1.00020	20.0	0.99821	1.00000	21.0	0.99799	0.99979	22.0	0.99777	0.99957
.1	0.99839	1.00018	.1	0.99819	0.99998	.1	0.99797	0.99977	.1	0.99775	0.99954
.2	0.99837	1.00016	.2	0.99816	0.99996	.2	0.99795	0.99974	.2	0.99773	0.99952
.3	0.99835	1.00014	.3	0.99814	0.99994	.3	0.99793	0.99972	.3	0.99770	0.99950
.4	0.99833	1.00012	.4	0.99812	0.99992	.4	0.99791	0.99970	.4	0.99768	0.99947
.5	0.99831	1.00010	.5	0.99810	0.99990	.5	0.99789	0.99968	.5	0.99766	0.99945
.6	0.99829	1.00008	.6	0.99808	0.99987	.6	0.99786	0.99966	.6	0.99764	0.99943
.7	0.99827	1.00006	.7	0.99806	0.99985	.7	0.99784	0.99963	.7	0.99761	0.99940
.8	0.99825	1.00004	.8	0.99804	0.99983	.8	0.99782	0.99961	.8	0.99759	0.99938
.9	0.99823	1.00002	.9	0.99802	0.99981	.9	0.99780	0.99959	.9	0.99756	0.99936
23.0	0.99754	0.99933	24.0	0.99730	0.99909	25.0	0.99705	0.99884	26.0	0.99679	0.99858
.1	0.99752	0.99931	.1	0.99727	0.99907	.1	0.99702	0.99881	.1	0.99676	0.99855
.2	0.99749	0.99929	.2	0.99725	0.99904	.2	0.99700	0.99879	.2	0.99673	0.99852
.3	0.99747	0.99926	.3	0.99723	0.99902	.3	0.99697	0.99876	.3	0.99671	0.99850
.4	0.99745	0.99924	.4	0.99720	0.99899	.4	0.99694	0.99874	.4	0.99668	0.99847
.5	0.99742	0.99921	.5	0.99717	0.99897	.5	0.99692	0.99871	.5	0.99665	0.99844
.6	0.99740	0.99919	.6	0.99715	0.99894	.6	0.99689	0.99868	.6	0.99663	0.99842
.7	0.99737	0.99917	.7	0.99712	0.99892	.7	0.99687	0.99866	.7	0.99660	0.99839
.8	0.99735	0.99914	.8	0.99710	0.99889	.8	0.99684	0.99863	.8	0.99657	0.99836
.9	0.99732	0.99912	.9	0.99707	0.99887	.9	0.99681	0.99860	.9	0.99654	0.99833
27.0	0.99652	0.99831	28.0	0.99624	0.99803	29.0	0.99595	0.99774	30.0	0.99565	0.99744
.1	0.99649	0.99828	.1	0.99621	0.99800	.1	0.99592	0.99771	.1	0.99562	0.99741
.2	0.99646	0.99825	.2	0.99618	0.99797	.2	0.99589	0.99768	.2	0.99559	0.99738
.3	0.99643	0.99822	.3	0.99615	0.99794	.3	0.99586	0.99765	.3	0.99556	0.99735
.4	0.99641	0.99820	.4	0.99612	0.99791	.4	0.99583	0.99762	.4	0.99553	0.99732
.5	0.99638	0.99817	.5	0.99609	0.99788	.5	0.99580	0.99759	.5	0.99550	0.99729
.6	0.99635	0.99814	.6	0.99607	0.99785	.6	0.99577	0.99756	.6	0.99547	0.99726
.7	0.99632	0.99811	.7	0.99604	0.99783	.7	0.99574	0.99753	.7	0.99544	0.99723
.8	0.99629	0.99808	.8	0.99601	0.99780	.8	0.99571	0.99750	.8	0.99541	0.99720
.9	0.99627	0.99806	.9	0.99598	0.99777	.9	0.99568	0.99747	.9	0.99538	0.99716

[A] Reference: CRC Handbook of Chemistry and Physics, David R. Lide, Editor-in-Chief, 74th Edition, 1993–1994.

[B] $\rho_w = 1.00034038 - (7.77 \times 10^{-6}) \times T - (4.95 \times 10^{-8}) \times T^2$

where:

ρ_w = Density of water in g/mL,

T = the test temperature in °C, and

K = $\rho_w/0.9982063$

[C] mL = cm^3.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/28/2012 13:24:19 MDT

content, calculate the range of wet masses for the specific gravity specimen in accordance with 7.1. From the sample, obtain a specimen within this range. Do not sample to obtain an exact predetermined mass.

9.2.2 To disperse the soil put about 100 mL of water into the mixing container of a blender or equivalent device. Add the soil and blend. The minimum volume of slurry that can be prepared by this equipment will typically require using a 500-mL pycnometer.

9.2.3 Using the funnel, pour the slurry into the pycnometer. Rinse any soil particles remaining on the funnel into the pycnometer using a wash/spray squirt bottle.

9.2.4 Proceed as described in 9.4.

9.3 *Method B—Procedure for Oven-Dried Specimens:*

9.3.1 Dry the specimen to a constant mass in an oven maintained at 110 ± 5°C. Break up any clods of soil using a mortar and pestle. If the soil will not easily disperse after drying or has changed composition, use Test Method A. Refer to 1.2.1 for soils that require use of Test Method A.

9.3.2 Place the funnel into the pycnometer. The stem of the funnel must extend past the calibration mark or stopper seal. Spoon the soil solids directly into the funnel. Rinse any soil particles remaining on the funnel into the pycnometer using a wash/spray squirt bottle.

9.4 *Preparing the Soil Slurry*—Add water until the water level is between ⅓ and ½ of the depth of the main body of the pycnometer. Agitate the water until slurry is formed. Rinse any soil adhering to the pycnometer into the slurry.

9.4.1 If slurry is not formed, but a viscous paste, use a pycnometer having a larger volume. See 7.1.1.

NOTE 5—For some soils containing a significant fraction of organic matter, kerosene is a better wetting agent than water and may be used in place of distilled water for oven-dried specimens. If kerosene is used, the entrapped air should only be removed by use of an aspirator. Kerosene is a flammable liquid that must be used with extreme caution.

9.5 *Deairing the Soil Slurry*—Entrapped air in the soil slurry can be removed using either heat (boiling), vacuum or combining heat and vacuum.

9.5.1 When using the heat-only method (boiling), use a duration of at least 2 h after the soil-water mixture comes to a full boil. Use only enough heat to keep the slurry boiling. Agitate the slurry as necessary to prevent any soil from sticking to or drying onto the glass above the slurry surface.

9.5.2 If only a vacuum is used, the pycnometer must be continually agitated under vacuum for at least 2 h. Continually agitated means the silt/clay soil solids will remain in suspension, and the slurry is in constant motion. The vacuum must remain relatively constant and be sufficient to cause bubbling at the beginning of the deairing process.

9.5.3 If a combination of heat and vacuum are used, the pycnometers can be placed in a warm water bath (not more than 40°C) while applying the vacuum. The water level in the bath should be slightly below the water level in the pycnometer, if the pycnometer glass becomes hot, the soil will typically stick to or dry onto the glass. The duration of vacuum and heat must be at least 1 h after the initiation of boiling. During the process, the slurry should be agitated as necessary to maintain boiling and prevent soil from drying onto the pycnometer.

9.6 *Filling the Pycnometer with Water*—Fill the pycnometer with deaired water (see 8.2.2) by introducing the water through a piece of small-diameter flexible tubing with its outlet end kept just below the surface of the slurry in the pycnometer or by using the pycnometer filling tube. If the pycnometer filling tube is used, fill the tube with water, and close the valve. Place the tube such that the drainage holes are just at the surface of the slurry. Open the valve slightly to allow the water to flow over the top of the slurry. As the clear water layer develops, raise the tube and increase the flow rate. If the added water becomes cloudy, do not add water above the calibration mark or into the stopper seal area. Add the remaining water the next day.

9.6.1 If using the stoppered iodine flask, fill the flask, such that the base of the stopper will be submerged in water. Then rest the stopper at an angle on the flared neck to prevent air entrapment under the stopper. If using a volumetric or stoppered flask, fill the flask to above or below the calibration mark depending on preference.

9.7 If heat has been used, allow the specimen to cool to approximately room temperature.

9.8 *Thermal Equilibrium*—Put the pycnometer(s) into a covered insulated container along with the thermometric device (or the temperature sensing portion of the thermometric device), a beaker (or bottle) of deaired water, stopper(s) (if a stoppered pycnometer is being used), and either an eyedropper or pipette. Keep these items in the closed container overnight to achieve thermal equilibrium.

9.9 *Pycnometer Mass Determination*—If the insulated container is not positioned near a balance, move the insulated container near the balance or vice versa. Open the container and remove the pycnometer. Only touch the rim of the pycnometer because the heat from hands can change the thermal equilibrium. Place the pycnometer on an insulated block (Styrofoam or equivalent).

9.9.1 If using a volumetric flask, adjust the water to the calibration mark following the procedure in 8.4.1.

9.9.2 If a stoppered flask is used, adjust the water to prevent entrapment of any air bubbles below the stopper during its placement. If water has to be added, use the thermally equilibrated water from the insulated container. Then, place the stopper in the bottle. If water has to be removed, before or after inserting the stopper, use an eyedropper. Dry the rim using a paper towel. Be sure the entire exterior of the flask is dry.

9.10 Measure and record the mass of pycnometer, soil, and water to the nearest 0.01 g using the same balance used for pycnometer calibration.

9.11 *Pycnometer Temperature Determination*—Measure and record the temperature of the slurry/soil-water mixture to the nearest 0.1°C using the thermometric device and method used during calibration in 8.5. This is the test temperature, T_t.

9.12 *Mass of Dry Soil*—Determine the mass of a tare or pan to the nearest 0.01 g. Transfer the soil slurry to the tare or pan. It is imperative that all of the soil be transferred. Water can be added. Dry the specimen to a constant mass in an oven

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/28/2012 13:24:19 MDT

maintained at 110 ± 5°C and cool it in a desiccator. If the tare can be sealed so that the soil can not absorb moisture during cooling, a desiccator is not required. Measure the dry mass of soil solids plus tare to the nearest 0.01 g using the designated balance. Calculate and record the mass of dry soil solids to the nearest 0.01 g.

NOTE 6—This method has been proven to provide more consistent, repeatable results than determining the dry mass prior to testing. This is most probably due to the loss of soil solids during the de-airing phase of testing.

10. Calculation

10.1 Calculate the mass of the pycnometer and water at the test temperature as follows:

$$M_{pw,t} = M_p + (V_p \cdot \rho_{w,t}) \quad (2)$$

where:

$M_{\rho w,t}$ = mass of the pycnometer and water at the test temperature (T_t), g,
M_p = the average calibrated mass of the dry pycnometer, g,
V_p = the average calibrated volume of the pycnometer, mL, and
$\rho_{w,t}$ = the density of water at the test temperature (T_t), g/mL from Table 2.

10.2 Calculate the specific gravity at soil solids the test temperature, G_t as follows:

$$G_t = \frac{\rho_s}{\rho_{w,t}} = \frac{M_s}{(M_{\rho_{w,t}} - (M_{pws,t} - M_s))} \quad (3)$$

where:

ρ_s = the density of the soil solids Mg/m^3 or g/cm^3,
$\rho_{w,t}$ = the density of water at the test temperature (T_t), from Table 2, g/mL or g/cm^3.
M_s = the mass of the oven dry soil solids (g), and
$M_{\rho ws,t}$ = the mass of pycnometer, water, and soil solids at the test temperature, (T_t), g.

10.3 Calculate the specific gravity of soil solids at 20°C as follows:

$$G_{20°C} = K \cdot G_t \quad (4)$$

where:

K = the temperature coefficient given in Table 2.

10.4 For soil solids containing particles greater than the 4.75-mm (No. 4) sieve for which Test Method C127 was used to determine the specific gravity of these particles, calculate an average specific gravity. Test Method C127 requires the test be performed at 23 ± 1.7°C and does not require the specific gravity data to be corrected to 20°C. Use 10.3 to correct this measurement to 20°C. Use the following equation to calculate the average specific gravity:

$$G_{avg@20°C} = \frac{1}{\frac{R}{100 \cdot G_{1@20°C}} + \frac{P}{100 \cdot G_{2@20°C}}} \quad (5)$$

where:

R = the percent of soil retained on the 4.75-mm sieve,
P = the percent of soil passing the 4.75-mm sieve,
$G_{1@20°C}$ = the apparent specific gravity of soils retained on the 4.75-mm sieve as determined by Test Method C127, corrected to 20°C
$G_{2@20°C}$ = the specific gravity of soil solids passing the 4.75-mm sieve as determined by these test methods (Equation 4).

11. Report: Test Data Sheets(s)/Form(s)

11.1 The method used to specify how data are recorded on the test data sheets or forms, as given below, is the industry standard, and are representative of the significant digits that should be retained. These requirements do not consider in situ material variation, use of the data, special purpose studies, or any considerations for the user's objectives. It is common practice to increase or reduce significant digits of reported data commensurate with these considerations. It is beyond the scope of the standard to consider significant digits used in analysis methods for engineering design.

11.2 Record as a minimum the following information (data):

11.2.1 Identification of the soil (material) being tested, such as boring number, sample number, depth, and test number.

11.2.2 Visual classification of the soil being tested (group name and symbol in accordance with Practice D2487).

11.2.3 Percent of soil particles passing the 4.75-mm (No. 4) sieve.

11.2.4 If any soil or material was excluded from the test specimen, describe the excluded material.

11.2.5 Method used (Method A or Method B).

11.2.6 All mass measurements (to the nearest 0.01 g).

11.2.7 Test temperature (to the nearest 0.1°C).

11.2.8 Specific gravity at 20°C (G, G_s, $G_{20°C}$) to the nearest 0.01. If desired, values to the nearest 0.001 may be recorded.

11.2.9 Average specific gravity at 20°C (G_{ave} or $G_{avg@20°C}$) to the nearest 0.01, if applicable. (See 10.4).

12. Precision and Bias

12.1 *Precision*—Criteria for judging the acceptability of test results obtained by these test methods on a range of soil types using Method A (except the soil was air dried) is given in Tables 3 and 4. These estimates of precision are based on the results of the interlaboratory program conducted by the ASTM Reference Soils and Testing Program.[3] In this program, some laboratories performed three replicate tests per soil type (triplicate test laboratory), while other laboratories performed a single test per soil type (single test laboratory). A description of the soils tested is given in 12.1.4. The precision estimates may vary with soil type and method used (Method A or B). Judgement is required when applying these estimates to another soil or method.

12.1.1 The data in Table 3 are based on three replicate tests performed by each triplicate test laboratory on each soil type. The single operator and multilaboratory standard deviation shown in Table 3, Column 4 were obtained in accordance with Practice E691, which recommends each testing laboratory perform a minimum of three replicate tests. Results of two

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: RR:D18-1009.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/28/2012 13:24:19 MDT

TABLE 3 Summary of Test Results from Triplicate Test Laboratories (Specific Gravity)

(1) Soil Type	(2) Number of Triplicate Test Labs	(3) Average Value[A]	(4) Standard Deviation[B]	(5) Acceptable Range of Two Results[C]
Single-Operator Results (Within- Laboratory Repeatability):				
CH	14	2.717	0.009	0.03
CL	13	2.670	0.006	0.02
ML	14	2.725	0.006	0.02
SP	14	2.658	0.006	0.02
Multilaboratory Results (Between- Laboratory Reproducibility):				
CH	14	2.717	0.028	0.08
CL	13	2.670	0.022	0.06
ML	14	2.725	0.022	0.06
SP	14	2.658	0.008	0.02

[A] The number of significant digits and decimal places presented are representative of the input data. In accordance with Practice D6026, the standard deviation and acceptable range of results cannot have more decimal places than the input data.

[B] Standard deviation is calculated in accordance with Practice E691 and is referred to as the 1*s* limit.

[C] Acceptable range of two results is referred to as the d2*s* limit. It is calculated as $1.960\sqrt{2}\cdot 1s$, as defined by Practice E177. The difference between two properly conducted tests should not exceed this limit. The number of significant digits/decimal places presented is equal to that prescribed by these test methods or Practice D6026. In addition, the value presented can have the same number of decimal places as the standard deviation, even if that result has more significant digits than the standard deviation.

TABLE 4 Summary of Single Test Result from Each Laboratory (Specific Gravity)[A]

(1) Soil Type	(2) Number of Test Laboratories	(3) Average Value	(4) Standard Deviation	(5) Acceptable Range of Two Results
Multilaboratory Results (Single-Test Performed by Each Laboratory):				
CH	18	2.715	0.027	0.08
CL	18	2.673	0.018	0.05
ML	18	2.726	0.022	0.06
SP	18	2.660	0.007	0.02

[A] See footnotes in Table 3.

properly conducted tests performed by the same operator on the same material, using the same equipment, and in the shortest practical period of time should not differ by more than the single-operator d2*s* limits shown in Table 3, Column 5. For definition of d2*s* see Footnote C in Table 3. Results of two properly conducted tests performed by different operators and on different days should not differ by more than the multilaboratory d2*s* limits shown in Table 3, Column 5.

12.1.2 In the ASTM Reference Soils and Testing Program, many of the laboratories performed only a single test. This is common practice in the design and construction industry. The data in Table 4 are based upon the first test result from the triplicate test laboratories and the single test results from the other laboratories. Results of two properly conducted tests performed by two different laboratories with different operators using different equipment and on different days should not vary by more than the d2*s* limits shown in Table 4, Column 5. The results in Tables 3 and 4 are dissimilar because the data sets are different.

12.1.3 Table 3 presents a rigorous interpretation of triplicate test data in accordance with Practice E691 from prequalified laboratories. Table 4 is derived from test data that represents common practice.

12.1.4 *Soil Type*—Based on the multilaboratory test results, the soil used in the program is described below in accordance with Practice D2487. In addition, the local name of the soil is given.

CH—Fat clay, CH, 99 % fines, LL=60, PI=39, grayish brown, soil had been air dried and pulverized. Local name—Vicksburg Buckshot Clay

CL—Lean clay, CL, 89 % fines, LL=33, PI=13, gray, soil had been air dried and pulverized. Local name—Annapolis Clay

ML—Silt, ML, 99 % fines, LL=27, PI=4, light brown, soil had been air dried and pulverized. Local name—Vicksburg Silt

SP—Poorly graded sand; SP, 20 % coarse sand, 48 % medium sand, 30 % fine sand, 2 % fines, yellowish brown. Local name—Frederick sand

12.2 *Bias*—There is no acceptable reference value for this test method, therefore, bias cannot be determined.

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition ($06^{\varepsilon 1}$) that may impact the use of this standard.

(*1*) Subsections 5.4, 8.3, 8.5, 8.6, 9.8, and 9.11 were changed to permit the use of standardized non-mercury thermometers.

(*2*) Subsection 8.6 was changed and subsection 8.6.1 was added to clarify the pycnometer calibration procedure.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the ASTM website (www.astm.org/COPYRIGHT/).

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/28/2012 13:24:19 MDT

Designation: D 1481 – 02 (Reapproved 2007)

An American National Standard

Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer[1]

This standard is issued under the fixed designation D 1481; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the density of oils more viscous than 15 cSt at 20°C (mm^2/s), and of viscous oils and melted waxes at elevated temperatures, but not at temperatures at which the sample would have a vapor pressure of 100 mm Hg (13 kPa) or above.

NOTE 1—To determine the densities of less viscous liquids at 20 or 25°C use Test Method D 1217.

1.2 This test method provides a calculation procedure for converting density to relative density (specific gravity).

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer

D 1250 Guide for Use of the Petroleum Measurement Tables

3. Terminology

3.1 *Definitions:*

3.1.1 *density*—the weight in a vacuum (that is, the mass) of a unit volume of the material at any given temperature.

3.1.2 *relative density (specific gravity)*—the ratio of the mass (weight in a vacuum) of a given volume of material at a temperature, t_1, to the mass of an equal volume of water at a reference temperature, t_2; or the ratio of the density of the material at t_1 to the density of water at t_2.

4. Summary of Test Method [3]

4.1 The liquid is drawn into the bicapillary pycnometer through the removable siphon arm and adjusted to volume at the temperature of test, in such a manner that there is practically no drainage in the unfilled tubing. After equilibration at the test temperature, liquid levels are read, and the pycnometer is removed from the thermostated bath, cooled to room temperature, and weighed.

4.2 Density or relative density (specific gravity), as desired, is then calculated from the volume at the test temperature and the weight of the sample. The effect of air buoyancy is included in the calculations.

5. Significance and Use

5.1 Density is a fundamental physical property that can be used in conjunction with other properties to characterize both the light and heavy fractions of petroleum and to access the quality of crude oils.

5.2 Determination of the density or relative density of petroleum and its products is necessary for the conversion of measured volumes to volumes at the standard temperatures of 15°C.

5.3 The determination of densities at the elevated temperatures of 40 and 100°C is particularly useful in providing the data needed for the conversion of kinematic viscosities in centistokes (mm^2/s) to the corresponding dynamic viscosities in centipoises (mPa·s).

6. Apparatus

6.1 *Pycnometer*[4]—A side-arm type of pycnometer conforming to the dimensions given in Fig. 1 and made of borosilicate glass. The weight shall not exceed 35 g without the side arm.

---

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04.0D on Physical and Chemical Methods.

Current edition approved Nov. 1, 2007. Published January 2008. Originally approved in 1957. Last previous edition approved in 2002 as D 1481–02.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] For a more complete discussion of this procedure, see Lipkin, M. R., Mills, I. W., Martin, C. C., and Harvey, W. T., *Analytical Chemistry*, ANCHA, Vol 21, 1949, p. 504.

[4] The sole source of supply of the pycnometers known to the committee at this time is Reliance Glass Co., 220 Gateway Rd., Bensenville, IL 60106-0825 have been found satisfactory. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:07:35 MDT

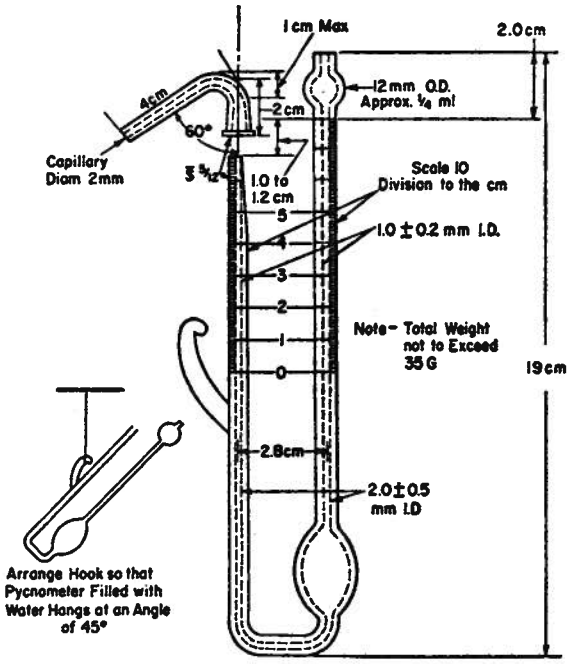


FIG. 1 Pycnometer

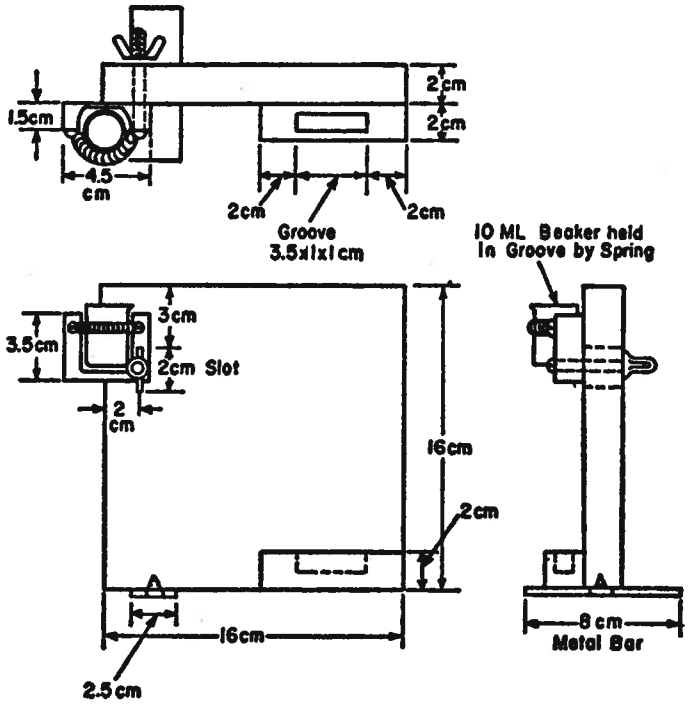


FIG. 2 Rack for Filling Pycnometer

6.2 *Rack*—A rack to use in filling the pycnometer (see Fig. 2).

6.3 *Constant-Temperature Oven*—An oven for use in filling the pycnometer. Any oven capable of holding the filling rack, and of maintaining a temperature of approximately 100°C, can be used.

6.4 *Constant-Temperature Bath*—A mixture of water and glycerin, or oil bath having a depth of at least 305 mm (12 in.) and provided with heating, stirring, and thermostating devices adequate to maintain desired temperatures in the range from 20 to 100°C with an accuracy of ±0.01°C.

6.5 *Bath Thermometers*—Thermometers graduated in 0.1°C subdivisions and standardized for the range of use to the nearest 0.01°C (ASTM Saybolt Viscosity Thermometers 17C to 22C are recommended). For most hydrocarbons, the density coefficient is about 0.0008 units/°C, and therefore a temperature error of ±0.013°C would cause an error of ±0.000 01 in density.

6.6 *Pycnometer Holder*—A holder, as shown in Fig. 3, is recommended for supporting the pycnometer in the bath. A single clamp device may be used.

6.7 *Balance*—A balance able to reproduce weighings within 0.1 mg when carrying a load of 35 g or less on each pan. The balance shall be located in a room shielded from drafts and fumes and in which the temperature changes between related weighings (empty and filled pycnometer) do not cause a significant change in the ratio of the balance arms. Otherwise, weighings shall be made by the substitution method in which the calibrated weights and pycnometer are alternatively weighed on the same balance pan. The same balance shall be used for all related weighings.

6.8 *Weights*—Weights shall be used whose relative values are known to the nearest 0.05 mg or better. The same set of weights shall be used for the calibration of the pycnometer and the determination of the densities, or the sets of weights shall be calibrated relative to each other.

7. Reagents and Materials

7.1 *Acetone*—(**Warning**—Extremely flammable. Use adequate ventilation.)

7.2 *Isopentane*—(**Warning**—Extremely flammable. Avoid buildup of vapors and remove all sources of ignition, especially nonexplosion-proof electrical apparatus.)

7.3 *Chromic Acid (Potassium Dichromate/Conc. Sulfuric Acid)*—(**Warning**—Causes severe burns. A recognized carcinogen. Do not get in eyes, on skin or clothing.)

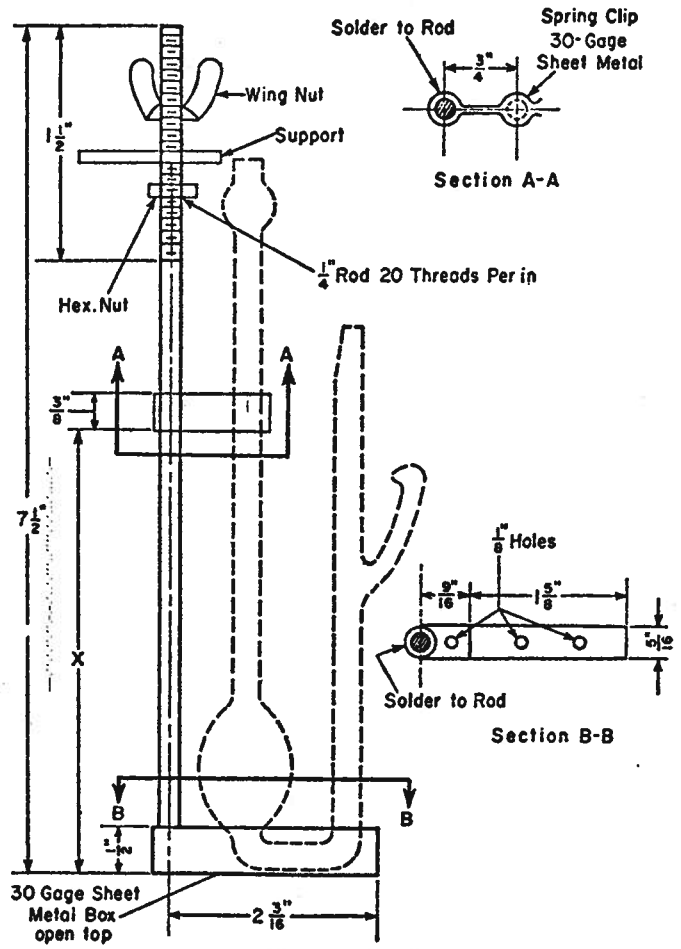
7.4 *Xylenes*—(**Warning**—Flammable liquid. Aspiration hazard. May irritate skin, eyes, respiratory tract or digestive tract, or both. May cause central nervous system depression, liver and kidney damage, or exhibit reproductive and fetal effects, or both.)

8. Preparation of Apparatus

8.1 Thoroughly clean the pycnometer and side arm with hot chromic acid cleaning solution (**Warning**—See 7.4). Chromic acid solution is the most effective cleaning agent. However, surfactant cleaning fluids have also been used successfully. Rinse well with distilled water; and dry at 105 to 110°C for at least 1 h, preferably with a slow current of filtered air passing through the pycnometer. Cleaning shall be done in this manner whenever the pycnometer is to be calibrated or whenever liquid fails to drain cleanly from the walls of the pycnometer or its capillary. Ordinarily, the pycnometer may be cleaned between determinations by washing with a suitable solvent, such as isopentane or xylenes, and vacuum drying. If acetone is used as the wash liquid, the pycnometer should then be rinsed with isopentane or xylenes.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:07:35 MDT



Metric Equivalents

in.	mm	in.	mm	in.	mm
½	3.2	½	12.7	1⅝	41.3
¼	6.4	9/16	14.3	2 3/16	55.7
5/16	7.9	¾	19.1	7½	191
⅜	9.5	1½	38.1		

FIG. 3 Pycnometer Holder

9. Calibration of Pycnometer

9.1 Weigh the clean, dry pycnometer (without the side arm) to the nearest 0.1 mg, and record the weight.

9.2 Fill the pycnometer with freshly boiled distilled water. This may be conveniently done by placing the pycnometer in the holder with the side arm dipping into a sample cup containing water. Allow the pycnometer to fill by siphoning. Break the siphon by removing the side arm when the liquid level in the bulb arm of the pycnometer reaches 6 on the scale.

9.3 Remove the side arm which was used to fill the pycnometer and remove excess liquid from the capillary tip by wiping with a small piece of absorbent paper.

9.4 Place the pycnometer in the holder in the constant-temperature bath at temperature t with the liquid level in the capillaries below the liquid level in the bath. When the liquid level has reached equilibrium (not less than 15 min), read the scale to the nearest 0.2 small division at the liquid level in each arm. After 5 min, read the liquid level again. If the sum of the scale readings in each reading differs by more than ±0.04, repeat readings at 5-min intervals. When readings are constant, record.

9.5 Remove the pycnometer from the bath and allow it to come to room temperature. Rinse the outer surface with distilled water, with acetone, then with redistilled xylenes, and dry thoroughly with a chemically clean lint-free cloth, slightly damp with water. Allow to stand a few minutes, and then weigh to nearest 0.1 mg.

NOTE 2—In atmospheres of low humidity (60 % or lower), drying the pycnometer by rubbing with dry cotton cloth will induce static charges equivalent to a loss of about 1 mg or more in the weight of the pycnometer. This charge may not be completely dissipated in less than ½ h and can be detected by touching the pycnometer to the wire hook on the balance and then drawing it away slowly. If the pycnometer exhibits an attraction for the wire hook, it may be considered to have a static charge.

9.6 Repeat the above, but break the siphon when water has reached the 3 mark in the bulb arm, and in the next experiment, at the 0 mark in the bulb arm. Obtain the apparent volume for each filling by dividing the weight of water held by the pycnometer in each experiment by the density of water at the calibration temperature t. Calibration shall be made at 20, 40, and 50°C. Prepare a calibration curve for 20°C by plotting the sum of the two scale readings *versus* the apparent volume at 20°C. If the curve is not a straight line, and future checks do not correct it, discard the pycnometer. The line shall not be more than 0.0002 mL/unit from any one determined point.

9.7 Corresponding calibration curves shall be made for 40 and 50°C. These calibration curves are checked using the following equation:

$$V_2 = V_1(1 + ct) \quad (1)$$

where:

V_2 = apparent volume at test temperature,
V_1 = apparent volume at 20°C, and
c = cubical coefficient of expansion of borosilicate glass (9.9×10^{-6}/°C).

The calculated and determined curves at 40 and 50°C should check to within ±0.0002 mL/unit at all points. The calibration curves for higher temperatures shall be obtained by calculation.

10. Procedure

10.1 Weigh the clean, dry pycnometer, without the side arm, to 0.1 mg and record the weight.

10.2 Place a 10-mL sample beaker in the wooden rack (Fig. 2). Before attaching the side arm to the pycnometer, drain a few drops of sample through the side arm to wet the inside surface and reduce the chance of trapping air bubbles in the capillary during the filling operation. Place the side arm on the pycnometer, and place the assembly on the rack with the side arm dipping into the sample beaker as shown in Fig. 4.

10.3 In filling the pycnometer with very viscous oils or high-melting waxes, place the whole filling assembly in a hot-air oven to facilitate filling. An oven at approximately 100°C is usually hot enough for this purpose.

10.4 Apply gentle suction to the bulb arm of the pycnometer to start the siphoning action. The suction must be gentle to avoid the formation of bubbles. After siphoning is started, allow filling by siphoning to continue until the liquid level in

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS
Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:07:35 MDT

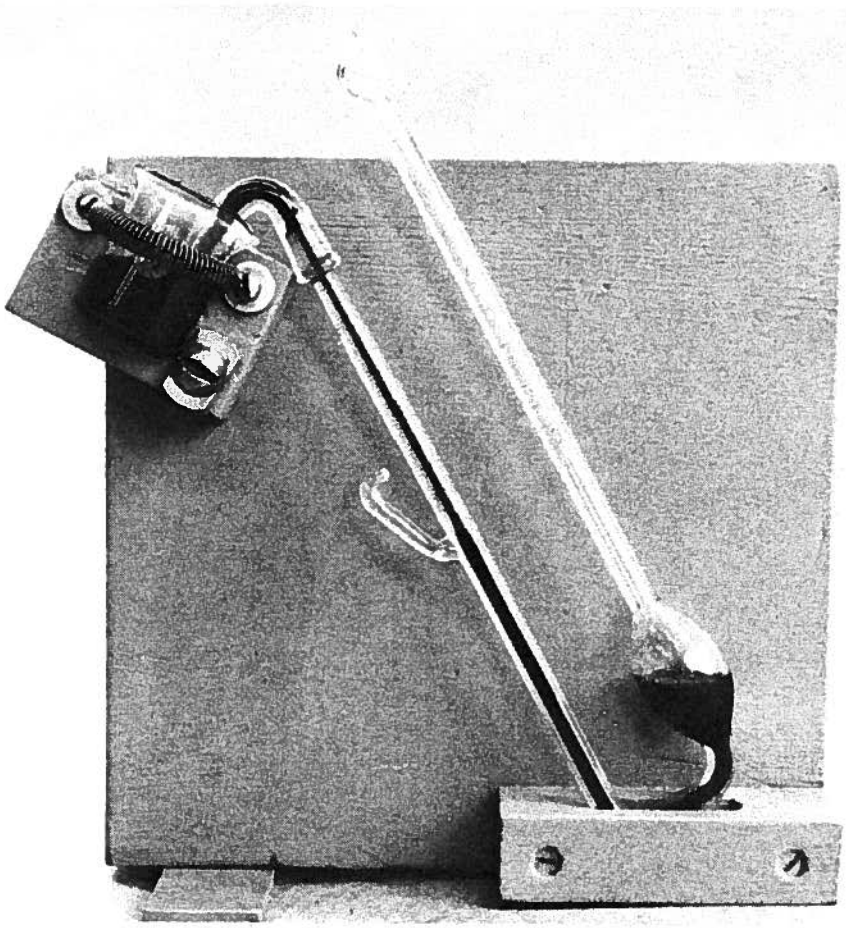


FIG. 4 Pycnometer Filling Assembly

the bulb arm ceases to rise. Then remove the pycnometer from the rack and place in the thermostated bath, in the same tilted position, until the oil ceases to contract. At this point, place the pycnometer in an upright position, and allow the liquid level in the bulb arm to reach the upper portion of the calibrated capillary, but not above 6.4. Stop siphoning by removing the side arm.

NOTE 3—With viscous oils, it will reduce drainage errors to fill to the 6.0 to 6.4 mark, and it may be necessary to apply a little suction to the long arm during cooling to prevent the meniscus in the bulb arm from falling. Maintain the meniscus at about the same level in the long arm throughout the whole determination.

10.5 After removing the side-arm cap from the short arm of the pycnometer, wipe the tip and ground joint of the pycnometer, and adjust it to an upright position in the thermostated bath. The bath liquid level shall be above the 6 mark on the pycnometer and below the ground glass tip of the pycnometer.

10.6 Allow 15 min for equilibrium to be obtained. After the stated 15-min time for coming to equilibrium, read the meniscus levels in both arms of the pycnometer to the nearest 0.2 of the smallest scale division. Wait 5 min and check readings. If the sum of the readings at the two different times do not agree to within ±0.04, repeat at 5-min intervals until checks are obtained. Record the sum of these readings and also record the corresponding apparent volume from the calibration curve for the same temperature.

NOTE 4—The final level of oil in the pycnometer should not be more than 5 mm below the tip of the ground glass end of the pycnometer, and the level in the long (bulb) side of the pycnometer should be no lower than it has been at any time during the procedure. With these precautions, drainage error (which is important with very viscous samples) is entirely eliminated.

10.7 Remove the pycnometer from the bath and tilt it so that the liquid moves down in the short arm and up in the bulb arm. Clean and dry the outside of the pycnometer as described in the calibration procedure (Section 9). Allow to come to balance room temperature. Weigh to the nearest 0.1 mg. Subtract the weight of empty pycnometer, without the side arm, to get the weight of sample.

11. Calculation

11.1 Calculate the density of the sample, corrected to vacuum, by the following equation:

$$\text{Density in vacuum, } d_v \text{ g/mL} = (W/V) + C \quad (2)$$

where:

W = weight of sample in air, g,
V = apparent volume, mL, and

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:07:35 MDT

C = vacuum correction, obtained from Table 1.

11.2 Calculate the relative density (specific gravity) of the sample at t_1/t_2 by dividing the density, as calculated in 10.1, by the density of water at the reference temperature, t_2, as obtained from Table 2. Relative density (specific gravity) at t_1/15.56°C (t/60°F where t is expressed in degrees Fahrenheit) can be changed to the conventional 15.56/15.56°C (60/60°F) relative density (specific gravity) by use of the appropriate Table 23 in Guide D 1250, provided that the glass expansion factor has been excluded.

11.3 In reporting density, give the test temperature and the units (for example, density at 40°C = x.xxxx g/mL). In reporting relative density (specific gravity), give both the test temperature and the reference temperature, but no units (for example, relative density (specific gravity), 40°C/15.56°C = x.xxxx). Carry out all calculations to five figures, and round off the final results to four figures.

12. Precision and Bias

12.1 The precision of the test method as obtained by statistical examination of interlaboratory test results is as follows:

12.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

Pycnometer Volume, mL	Repeatability, g/mL
10	0.000 15

12.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

Pycnometer Volume, mL	Reproducibility, g/mL
10	0.000 35

NOTE 5—If pycnometers of other than 10 mL in volume are used, this precision statement may not apply.

TABLE 1 Vacuum Corrections

	Correction[A] Plus		Correction[A] Plus
0.70	0.000 36	0.85	0.000 18
0.71	0.000 35	0.86	0.000 17
0.72	0.000 33	0.87	0.000 16
0.73	0.000 32	0.88	0.000 14
0.74	0.000 31	0.89	0.000 13
0.75	0.000 30	0.90	0.000 12
0.76	0.000 29	0.91	0.000 11
0.77	0.000 28	0.92	0.000 10
0.78	0.000 26	0.93	0.000 09
0.79	0.000 25	0.94	0.000 07
0.80	0.000 24	0.95	0.000 06
0.81	0.000 23	0.96	0.000 05
0.82	0.000 22	0.97	0.000 04
0.83	0.000 20	0.98	0.000 03
0.84	0.000 19	0.99	0.000 01

[A] This table applies for all air density values between 0.0011 and 0.0013 g/mL. For air densities outside this range, the vacuum correction shall be calculated from the equation $C = (d_a/0.998\ 23) \times [0.998\ 23 - (W/V)]d_a$ being the density of the air in the balance case in grams per millilitre.

TABLE 2 Density of Water[A]

Temperature,° C	Density, g/mL	Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL
0	0.999 840	21	0.997 991	40	0.992 212
3	0.999 964	22	0.997 769	45	0.990 208
4	0.999 972	23	0.997 537	50	0.988 030
5	0.999 964	24	0.997 295	55	0.985 688
10	0.999 699	25	0.997 043	60	0.983 191
15	0.999 099	26	0.996 782	65	0.980 546
15.56	0.999 012	27	0.996 511	70	0.977 759
16	0.998 943	28	0.996 231	75	0.974 837
17	0.998 774	29	0.995 943	80	0.971 785
18	0.998 595	30	0.995 645	85	0.968 606
19	0.998 404	35	0.994 029	90	0.965 305
20	0.998 203	37.78	0.993 042	100	0.958 345

[A] Densities conforming to the International Temperature Scale 1990 (ITS 90) were extracted from Appendix G, *Standard Methods for Analysis of Petroleum and Related Products 1991*, Institute of Petroleum, London.

12.2 *Bias*—The difference of results from the established value when compared to pure reference materials is not expected to be more than 0.000 35 g/mL. Specific bias has not been established by cooperative testing.

13. Keywords

13.1 density; gravity; pycnometer; relative density; specific gravity

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Sold to:Brown & Caldwell, 753356
Not for Resale,03/19/2012 17:07:35 MDT

Designation: D 6836 – 02 (Reapproved 2008)ε2

Standard Test Methods for Determination of the Soil Water Characteristic Curve for Desorption Using Hanging Column, Pressure Extractor, Chilled Mirror Hygrometer, or Centrifuge[1]

This standard is issued under the fixed designation D 6836; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Mercury warning and other minor changes were editorially added in November 2008.
ε[2] NOTE—"1n" was editorially corrected to "ln" in Eq 12 in March 2009.

1. Scope

1.1 These test methods cover the determination of soil water characteristic curves (SWCCs) for desorption (drying). SWCCs describe the relationship between suction and volumetric water content, gravimetric water content, or degree of water saturation. SWCCs are also referred to as soil water retention curves, soil water release curves, or capillary pressure curves.

1.2 This standard describes five methods (A-E) for determining the soil water characteristic curve. Method A (hanging column) is suitable for making determinations for suctions in the range of 0 to 80 kPa. Method B (pressure chamber with volumetric measurement) and Method C (pressure chamber with gravimetric measurement) are suitable for suctions in the range of 0 to 1500 kPa. Method D (chilled mirror hygrometer) is suitable for making determinations for suctions in the range of 500 kPa to 100 MPa. Method E (centrifuge method) is suitable for making determinations in the range 0 to 120 kPa. Method A typically is used for coarse soils with little fines that drain readily. Methods B and C typically are used for finer soils which retain water more tightly. Method D is used when suctions near saturation are not required and commonly is employed to define the dry end of the soil water characteristic curve (that is, water contents corresponding to suctions > 1000 kPa). Method E is typically used for coarser soils where an appreciable amount of water can be extracted with suctions up to 120 kPa. The methods may be combined to provide a detailed description of the soil water characteristic curve. In this application, Method A or E is used to define the soil water characteristic curve at lower suctions (0 to 80 kPa for A, 0 to 120 kPa for E) near saturation and to accurately identify the air entry suction, Method B or C is used to define the soil water characteristic curve for intermediate water contents and suctions (100 to 1000 kPa), and Method D is used to define the soil water characteristic curves at low water contents and higher suctions (> 1000 kPa).

1.3 All observed and calculated values shall conform to the guide for significant digits and rounding established in Practice D 6026. The procedures in Practice D 6026 that are used to specify how data are collected, recorded, and calculated are regarded as the industry standard. In addition, they are representative of the significant digits that should generally be retained. The procedures do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the objectives of the user. Increasing or reducing the significant digits of reported data to be commensurate with these considerations is common practice. Consideration of the significant digits to be used in analysis methods for engineering design is beyond the scope of this standard.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 **Warning**—Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website (http://www.epa.gov/mercury/faq.htm) for additional information. Users should be aware that selling mercury or mercury-containing products, or both, in your state may be prohibited by state law.

[1] These test methods are under the jurisdiction of ASTM Committee D18 on Soil and Rock and are the direct responsibility of Subcommittee D18.04 on Hydrologic Properties and Hydraulic Barriers.

Current edition approved Sept. 1, 2008. Published November 2008. Originally approved in 2002. Last previous edition approved in 2002 as D 6836 – 02.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 421 Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants

D 425 Test Method for Centrifuge Moisture Equivalent of Soils

D 653 Terminology Relating to Soil, Rock, and Contained Fluids

D 698 Test Methods for Laboratory Compaction Characteristics of Soil Using Standard Effort (12 400 ft-lbf/ft^3(600 kN-m/m^3))

D 854 Test Methods for Specific Gravity of Soil Solids by Water Pycnometer

D 2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D 3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D 4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D 5084 Test Methods for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter

D 6026 Practice for Using Significant Digits in Geotechnical Data

2.2 *API Standard:*

API RP 40 Recommended Practice for Core-Analysis Procedure[3]

3. Terminology

3.1 For common definitions of other terms in this standard see Terminology D 653.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *air entry pressure*—the air pressure required to introduce air into and through the pores of a saturated porous plate.

3.2.2 *air entry suction, ψ_a*—the suction required to introduce air into and through the pores of a saturated porous material.

3.2.3 *axis translation*—the principle stating that a matric suction ψ can be applied to a soil by controlling the pore gas pressure, u_g, and the pore water pressure, u_w, so that the difference between the pore gas pressure and pore water pressure equals the desired matric suction, that is, $\psi = u_g - u_w$.

3.2.4 *gravimetric water content, w*—the ratio of the mass of water contained in the pore spaces of soil or rock to the mass of solid particles.

3.2.5 *matric suction, ψ*—the negative gage pressure, relative to an external gas pressure acting on the soil water, that must be applied to a solution identical in composition to the soil water to maintain equilibrium through a porous membrane existing between the solution and the soil water. Matric suction is also referred to as matric potential, capillary suction, and capillary potential. By definition, matric suction is the difference between the pore gas pressure, u_g, and the pore water pressure, u_w, that is, $\psi = u_g - u_w$. In most cases the pore gas is air.

3.2.6 *osmotic suction, ψ_o*—the negative gage pressure derived from the measurement of the vapor pressure of water in equilibrium with a solution identical in composition with the soil water, relative to the vapor pressure of water in equilibrium with free pure water. Osmotic suction is also referred to as osmotic potential.

3.2.7 *porous membrane*—a porous polymeric membrane that can transmit water and has a air entry pressure exceeding the highest matric suction to be applied during a test.

3.2.8 *porous plate*—a plate made of metal, ceramic, or other porous material that can transmit water and has an air entry pressure exceeding the highest matric suction to be applied during a test.

3.2.9 *pressure chamber*—a vessel used to apply a gas pressure on the specimen and the soil pores to induce a specified matric suction.

3.2.10 *saturated water content*—volumetric or gravimetric water content when the specimen is saturated.

3.2.11 *soil water characteristic curve*—a graph of suction (matric or total) versus water content (gravimetric or volumetric) or saturation. The soil water characteristic curve is also referred to as the soil water retention curve, the soil water release curve, and the capillary pressure curve.

3.2.12 *total suction, ψ_t*—the negative gage pressure derived from the measurement of the vapor pressure of water in equilibrium with water in the soil pores, relative to the vapor pressure of water in equilibrium with free pure water. Total suction is the sum of matric and osmotic suction, $\psi_t = \psi + \psi_o$. Total suction is also referred to as total potential.

3.2.13 *volumetric water content, θ*—the ratio of the volume of water contained in the pore spaces of soil or rock to the total volume of soil and rock.

3.2.14 *water activity, a_w*—the ratio of vapor pressure of water in the soil gas to the saturated vapor pressure at the existing soil temperature. Water activity is also referred to as the relative humidity.

4. Summary of Methods

4.1 *Methods A-C*—Methods A-C yield soil water characteristic curves in terms of matric suction. Various suctions are applied to the soil and the corresponding water contents are measured. Two different procedures are used to apply the suction. In Method A, the matric suction is applied by reducing the pore water pressure while maintaining the pore gas pressure at the atmospheric condition. In Methods B and C, the pore

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, http://www.api.org.

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5805805100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

water pressure is maintained at atmospheric pressure, and the pore gas pressure is raised to apply the suction via the axis translation principle.

4.1.1 For all three methods, saturated soil specimens are placed in contact with a water saturated porous plate or membrane. The matric suction is applied by one of the two aforementioned procedures. Application of the matric suction causes water to flow from the specimen until the equilibrium water content corresponding to the applied suction is reached. Equilibrium is established by monitoring when water ceases to flow from the specimen. Several equilibria are established at successive matric suctions to construct a soil water characteristic curve.

4.1.2 The water content corresponding to the applied suction is determined in one of two ways. For Methods A and B, the volume of water expelled is measured using a capillary tube. The water content is then determined based on the known initial water content of the specimen and the volume of water expelled. For Method C, the water content is measured gravimetrically by weighing the specimen after removal from the apparatus.

4.2 *Method D*—Method D yields a soil water characteristic curve in terms of total suction. In contrast to Methods A-C, the water content of the soil is controlled in Method D, and the corresponding suctions are measured. Two different approaches are commonly used. In one approach, a set of specimens are prepared that are essentially identical, but have different water contents. Water contents are selected that span the range of water contents that will be used to define the soil water characteristic curve. In the other approach, a single specimen is used. The specimen is tested, dried to a lower water content, and then tested again. This process is repeated until suctions have been measured at all of the desired water contents.

4.2.1 In Method D, the water activity of the pore water is measured using a chilled mirror hygrometer (also known as a chilled mirror psychrometer) and then the total suction is computed using the Kelvin equation. In many cases, Method D is used to determine only that portion of the soil water characteristic curve corresponding to higher suctions (typically > 1000 kPa) and lower water contents. Under these conditions, the osmotic component of total suction is generally small, and the matric and total suctions are comparable. Thus, the data from Methods A-C and Method D can be combined to form a single soil water characteristic curve. An example of this type of soil water characteristic curve is provided in Section 11.

4.3 *Method E*—Method E yields a soil water characteristic curve in terms of matric suction (or capillary pressure). The specimen is contained in a support chamber that is subjected to a centrifugal force in a centrifuge. Different matric suctions are applied by varying the angular velocity of the centrifuge. Water displaced from the soil at a given angular velocity is collected and measured in a calibrated cylinder at the base of the support chamber. A soil water characteristic curve is measured by subjecting the specimen to a series of angular velocities (each corresponding to a matric suction) and measuring the volume of water displaced from the soil at each velocity.

5. Significance and Use

5.1 The soil water characteristic curve (SWCC) is fundamental to hydrological characterization of unsaturated soils and is required for most analyses of water movement in unsaturated soils. The SWCC is also used in characterizing the shear strength and compressibility of unsaturated soils. The unsaturated hydraulic conductivity of soil is often estimated using properties of the SWCC and the saturated hydraulic conductivity.

5.2 This method applies only to soils containing two pore fluids: a gas and a liquid. The liquid is usually water and the gas is usually air. Other liquids may also be used, but caution must be exercised if the liquid being used causes excessive shrinkage or swelling of the soil matrix.

5.3 A full investigation has not been conducted regarding the correlation between soil water characteristic curves obtained using this method and soil water characteristics curves of in-place materials. Thus, results obtained from this method should be applied to field situations with caution and by qualified personnel.

NOTE 1—The quality of the result produced by this standard depends on the competence of the personnel performing the test and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 are generally considered capable of competent and objective testing, sampling, inspection, etc. Users of this standard are cautioned that compliance with Practice D 3740 does not in itself ensure reliable results. Reliable results depend on many factors. Practice D 3740 provides a means of evaluating some of these factors.

6. Apparatus

6.1 *Hanging Column Apparatus (Method A)*—A hanging column apparatus consists of three parts: a specimen chamber, an outflow measurement tube, and a suction supply (Fig. 1). The specimen chamber consists of a glass or rigid plastic funnel containing a porous plate that is large enough to contain the specimen being tested. Such funnels are commonly referred to as "Buchner" funnels. A photograph of a funnel used for a hanging column apparatus is shown in Fig. 2. Water expelled from the specimen during the test is measuring using a capillary tube connected to the outflow end of the funnel. The other end of this capillary tube is connected to suction supply consisting of two reservoirs. The relative elevation of the two reservoirs is adjusted to develop a vacuum, which is transmitted to the capillary tube. The magnitude of the applied suction is measured with a manometer.

6.2 *Pressure Chamber (Methods B and C)*—Pressure chambers are used for the pressure extraction method to apply a gas pressure (typically air pressure) to the specimen and the gas in the pores. Typically the pressure chamber is a metallic vessel. The pressure chamber shall be pressure-rated, at the very least, for the maximum pressure to be applied to the vessel during the test. The pressure chamber shall have a sealed, non-collapsing outflow tube that connects the atmospheric pressure side of the porous plate (or membrane) to the outside of the pressure chamber. Schematics of two types of pressure chambers are shown in Figs. 4 and 5. Photographs of these pressure chambers are shown in Fig. 4. In some cases, the effects of

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

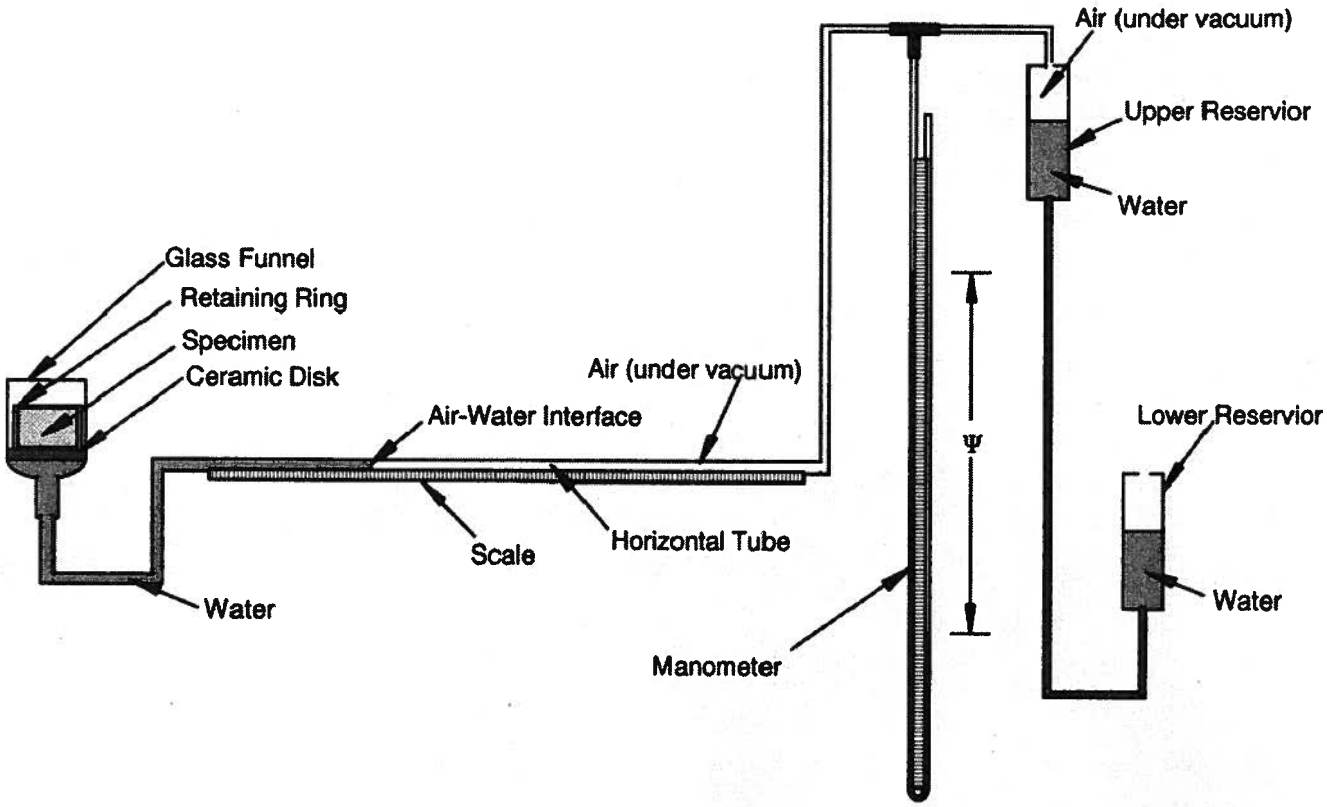


FIG. 1 Schematic of Hanging Column Apparatus

overburden pressure may wish to be simulated. For these cases, the pressure vessel may be equipped with a piston or a triaxial cell may be used.

6.3 *Regulated Pressure Source (Methods B and C)*—A regulated pressure source is used to apply gas pressure to the pressure chamber. Compressed gas is typically provided by an air compressor or bottled gas. The pressure source and associated regulators shall be capable of maintaining the desired pressure with an accuracy of ±0.25 % or better.

6.4 *Pressure Indicators (Methods A, B, and C)*—Pressure indicators are used to monitor the pressure applied in pressure chambers and the applied suction for the hanging column. Bourdon gages, mercury and water manometers, and pressure transducers are acceptable devices for measuring pressure in the pressure chamber. The accuracy of the measuring device must be within ±0.25 % of the matric suction being applied. For cases where both very large and very small suctions are to be applied, multiple pressure indicators may be used to provide sufficient accuracy for the various pressures to be applied. The manometer used to measure the applied suction in Method A shall have a resolution of 1 mm of water.

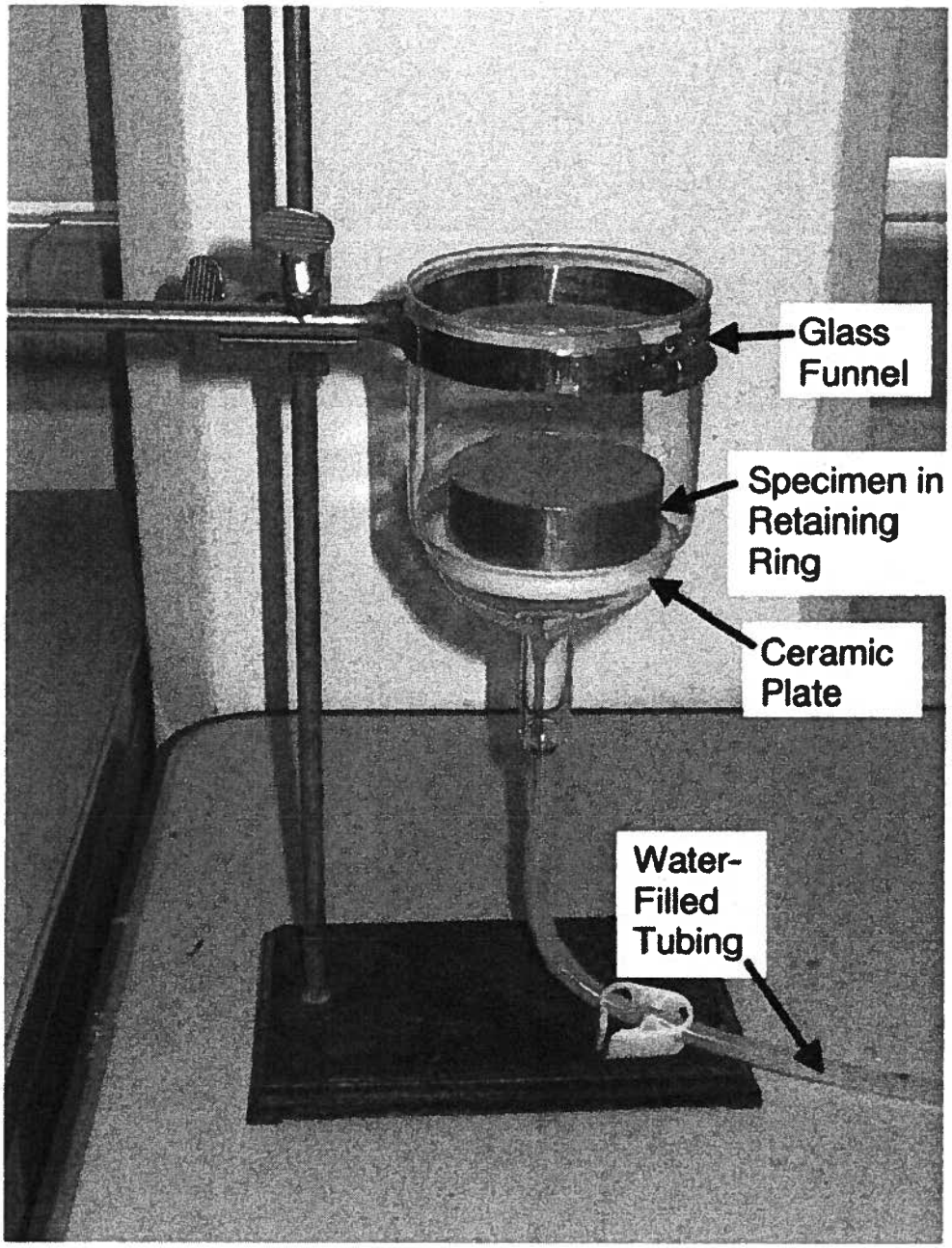
6.5 *Porous Plate (Methods B and C)*—A porous plate is used in the hanging column and pressure extractor to provide a saturated interface between the pore water and the water in the volume measuring system. Porous plates shall be fabricated from material that is hydrophilic and has an air entry pressure greater than the maximum matric suction to be applied during the test. Porous ceramic plates are typically usually used as porous plates. Porous plates are shown in the photographs in Figs. 4 and 5.

6.5.1 The porous plate shall be configured such that one side of the membrane is in contact with the specimen and is exposed to gaseous atmosphere in the pressure chamber. The other side of the plate shall be in contact with the outflow system (Fig. 3). A seal shall be provided that prevents gas in the chamber from contacting the side of the porous membrane in contact with the outflow system. This seal shall also prevent water from leaking from the outflow system and into the pressure chamber.

6.6 *Pressure Membrane (Methods B and C)*—A porous membrane is used in a pressure extractor to provide a saturated interface between the pore water and the water in the measuring system. The porous membrane shall be fabricated from a material that is hydrophilic and has an air entry pressure higher than the maximum suction to be applied during the test. A non-corroding porous material having sufficient strength and stiffness shall be used to support the porous material (a stainless steel screen typically is used). The pores in the supporting material shall be sufficiently large to preclude development of measurable capillary stresses. The membrane and supporting material shall be configured such that the unsupported side of the membrane is in contact with the specimen and is exposed to gaseous atmosphere in the pressure chamber. The support side of the membrane shall be in contact with the outflow system. A seal shall be provided that prevents

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT



Specimen is contained in a retaining ring.

FIG. 2 Photograph of Funnel Used for Hanging Column Apparatus.

gas in the chamber from contacting the side of the porous membrane in contact with the outflow system. This seal shall also prevent water from leaking from the outflow system and into the pressure chamber.

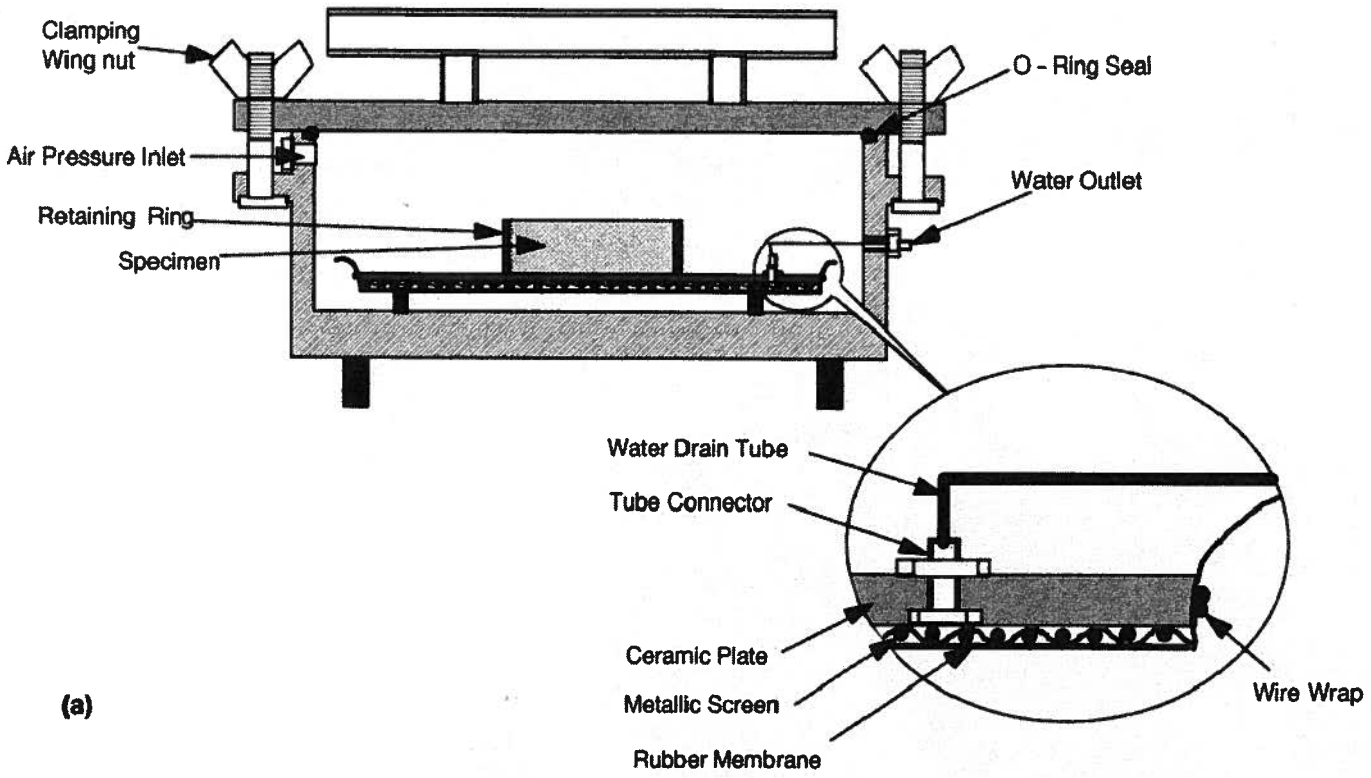
6.7 *Capillary Tube (Methods A and B)*—A capillary tube is used to measure the outflow from the specimen for Methods A and B. The capillary tube shall be free of dirt, oil, or other contaminants and include a scale that permits resolution of the volume expelled to 0.1 mL or better.

6.8 *Specimen Retaining Rings*—Specimens tested using Methods A, B, and C shall be retained on the porous plate or membrane using a retaining ring at least 5 mm in height and 25 mm in inside diameter. The wall thickness shall be sufficient to retain the soil without visible distortion. A photograph of a specimen in a typical retaining ring is shown in Fig. 6a. Retaining rings are typically constructed from stainless steel, acrylic, or polyvinyl chloride.

6.9 *Chilled Mirror Hygrometer (Method D)*—A chilled mirror hygrometer (also known as a chilled mirror psychrometer) shall be used to measure water activity to within 0.001. The chilled mirror hygrometer must be able to test specimens with a diameter of at least 20 mm and height of at least 5 mm. Water activity obtained from the chilled mirror hygrometer

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT



Schematic of pressure chambers with ceramic porous plates: (a) conventional chamber for single or multiple specimens and (b) chamber for single specimen

FIG. 3 Schematic of Pressure Chambers With Ceramic Porous Plates

shall be converted to total suction using the Kelvin equation. A photograph of a chilled mirror hygrometer is shown in Fig. 7.

6.10 *Specimen Retaining Dish (Method D)*—Specimens tested using Method D shall be contained in a specimen retaining dish with a diameter of at least 20 mm and height of at least 5 mm. The dish shall be made of a non-porous and non-corroding material such as stainless steel or plastic. A photograph of a specimen in a retaining dish is shown in Fig. 6b.

6.11 *Moisture Content Container*—A container meeting the requirements outlined in Method D 2216 shall be provided for determination of water content.

6.12 *Balance*—For Methods A-C, a balance shall be used that has a minimum capacity of 200 g and a sensitivity of 0.01 g. For Method D, a balance shall be used that has a minimum capacity of 50 g and a sensitivity of 0.001 g. All balances shall meet the requirements of Specification D 4753.

6.13 *Desiccator*—A desiccator shall be provided that has suitable size to hold specimens for cooling after removal from the oven. A desiccator is only recommended when moisture containers having close-fitting lids are not used (see Method D 2216).

6.14 *Drying Oven*—A thermostatically controlled drying oven capable of maintaining temperature at 110 ± 5°C as per requirements of Method D 2216.

6.15 *Temperature Controlled Centrifuge (Method E)*—A thermostatically controlled centrifuge capable of maintaining a temperature of 20 ± 3°C when operated between 0-10 000 RPM. A photograph of a centrifuge is shown in Fig. 8 with sealed sample support chamber and calibrated water collection tube. The centrifuge shall include a measurement device for recording outflow in the support chamber to the nearest 0.1 mL while the specimen is under centrifugation. A strobe light apparatus is normalized used for this purpose.

6.16 *Specimen Support Chamber (Method E)*—A chamber used to retain the specimen and the calibrated cylinder used to collect the displaced water. A schematic of a specimen support chamber is shown in Fig. 9.

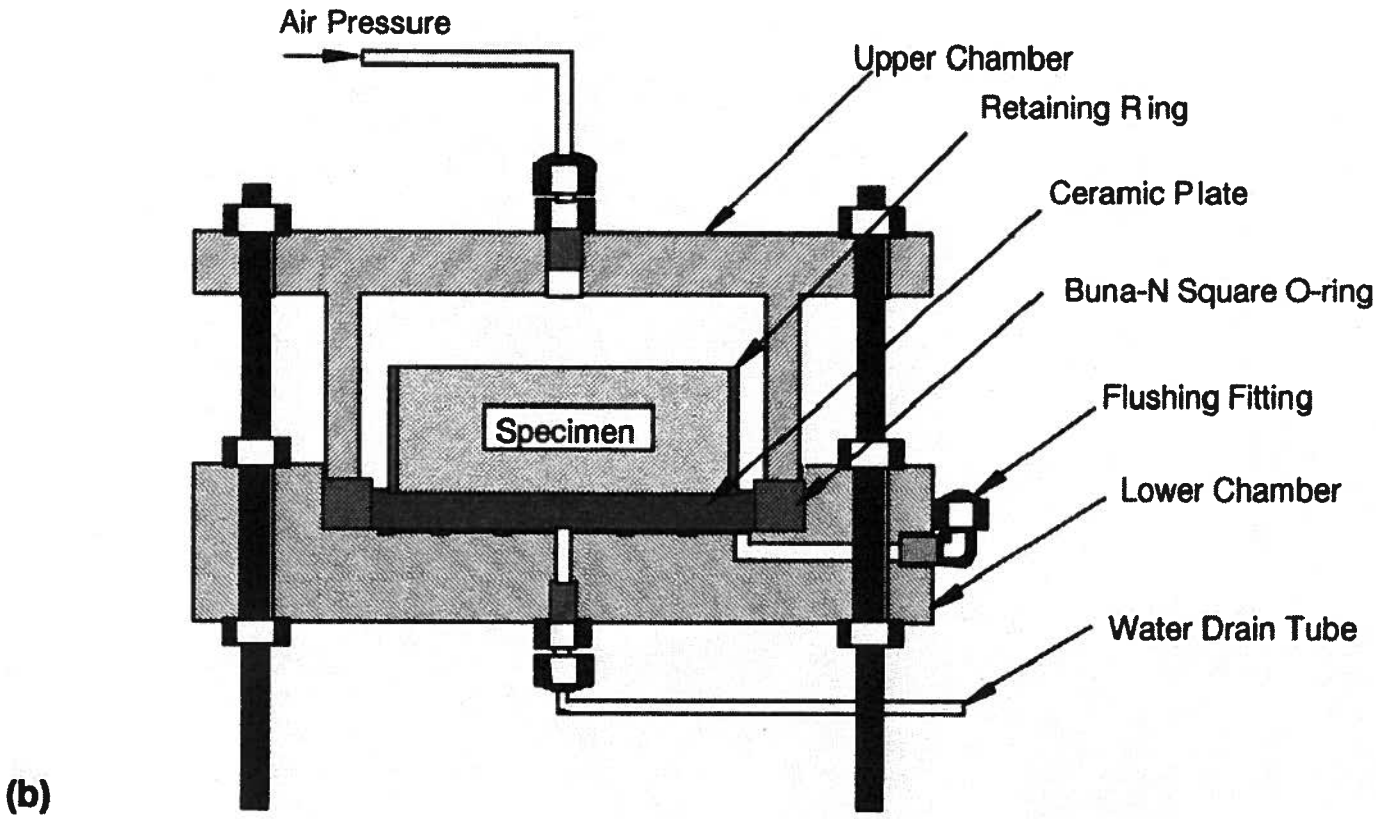
6.17 *Saturation Tray, Chamber, or Apron (Methods A-E)*—Plates, membranes and specimens are saturated in a saturation tray, a saturation chamber, or directly in pressure chamber using a saturation apron. Trays shall have a depth greater than the sum of the specimen height and thickness of the porous plate (or membrane). Saturation chambers are vessels filled with water in which a specimen in a retaining ring can be inundated for saturation. A vacuum is applied to the water in a saturation chamber using an external device. A saturation apron in the pressure chamber may be used to impound water around a specimen for saturation. Aprons are only used for saturating specimens.

6.18 *Miscellaneous Tools*—wire saw, beveled straightedge, spatula, and other small tools for trimming test specimens, 10 mL and 100 mL graduated cylinders, 25 mL syringe.

6.19 *Laboratory Environment*—The laboratory temperature shall be maintained within ± 3°C during the test. The apparatus shall be shielded from directly sunlight or other

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT



Schematic of pressure chambers with ceramic porous plates: (a) conventional chamber for single or multiple specimens and (b) chamber for single specimen

FIG. 3 Schematic of Pressure Chambers With Ceramic Porous Plates *(continued)*

sources of heat that may cause variations in temperature. If very precise measurements are required, the hanging column or pressure chamber may be isolated in a thermally insulated box.

7. Reagents

7.1 *Saturating Liquid*:

7.1.1 The saturating liquid is used to saturate the porous plates and membranes, the specimen, and the outflow measuring components of the apparatus.

7.1.2 The saturating liquid should be specified by the requestor. If no specific type of saturating water is specified by the requestor, the water shall be deaired 0.0025 M $CaCl_2$ solution or tap water. Distilled or deionized water shall not be used unless specified by the requestor. A biocide may be added to the water to minimize microbial growth during the test. If a biocide is added, the type and concentration of the biocide shall be included in the report.

7.1.3 *Deaired Water*—Deaired water shall be used. Water is usually deaired by boiling, spraying a fine mist of water into an evacuated vessel attached to a vacuum source, or by forceful agitation of water in a container attached to a vacuum source. To prevent dissolution of air back into the water, deaired water shall not be exposed to the atmosphere for prolonged periods.

8. Preparation

8.1 *Preparation of Specimens*:

8.1.1 *Specific Gravity*—Measure the specific gravity of solids on a sub-sample of the material to be tested following the method in Test Method D 854.

8.1.2 *Retaining Ring, Dish, or Mold*—Measure the inside diameter of the retaining ring, dish, or mold to the nearest 0.1 mm at three locations and record the average of these measurements as the diameter. Measure the height of the retaining ring, dish (inside height for dish), or mold to the nearest 0.1 mm at three locations and record the average of these measurements as the height. Weigh the retaining ring, dish, or mold and record its weight to the nearest 0.01 g (ring) or 0.001 g (dish).

8.1.3 *Specimens Prepared from Disturbed Samples*—Air dry and crush a representative sub-sample that has a mass at least twice that required to fill the specimen retaining ring. The sample may be crushed following the procedure described in Practice D 421. Remove all particles from the crushed sub-sample having a diameter exceeding one-half the height of the retaining ring. If requested, moisten the sample to the gravimetric water content specified using tap water. Prepare a specimen by placing a known mass of the moistened sub-sample into the retaining ring (Methods A-D) or a specimen mold (Method E). Tap or compact the soil to the density specified by the requestor. Trim the upper surface of the specimen so that it is level with the top of the retaining ring or mold. Weigh the specimen in the retaining ring or mold and

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

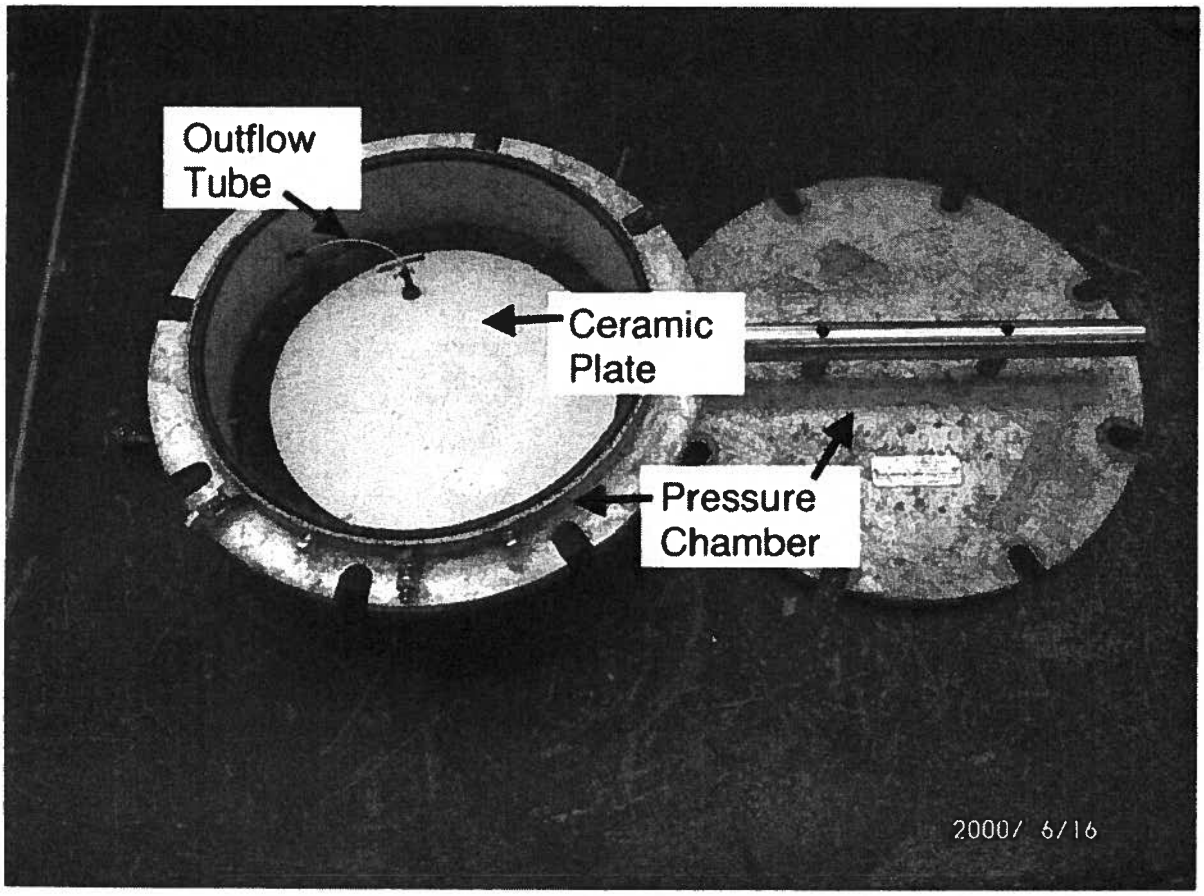


FIG. 4 Photographs of a Multiple-Specimen Pressure Chamber

record the weight to the nearest 0.01 g. Measure the gravimetric water content of the remaining material using Method D 2216.

8.1.4 *Specimens Prepared from Undisturbed Samples*—For Methods A-D, place the undisturbed sample on the bench and gently place a retaining ring on the surface of soil. Use trimming tools to gently remove soil that protrudes beyond the edge of the retaining ring so that the ring can slide over the soil specimen with little effort. Continue trimming until the soil fills the retaining ring. Trim the top of the specimen flush with the top of the retaining ring. Weigh the specimen in the retaining ring and record the weight to the nearest 0.01 g. Measure the gravimetric water content of the remaining material using Method D 2216. This procedure is also used to prepare specimens for testing in the chilled mirror hygrometer after being tested in the hanging column or pressure extractor.

8.1.4.1 A similar procedure is followed to prepare specimens for Method E, except no trimming ring or dish is employed. Specimens are trimmed from the undisturbed sample to form a right circular cylinder that fits inside the specimen support chamber. Typically specimens are 38 mm in diameter and 51 mm long (Fig. 9). When trimming is complete, measure the height of the specimen at three locations to the nearest 0.1 mm, and record the average of these measurements as the height. Similarly, measure the diameter of the specimen at three locations to the nearest 0.1 mm, and record the average of these measurements as the diameter. Weigh the specimen to the nearest 0.01 g. The sides of the trimmed specimen are wrapped in unsintered teflon tape. Stainless steel screens (220 mesh) are placed on the ends of the specimen.

8.1.5 *Specimens Compacted in the Laboratory*—Compact the specimen to the specified water content and density using the method of compaction specified by the requestor. For Methods A-D, place the compacted specimen on the bench and gently place a retaining ring on the surface of soil. Use trimming tools to gently remove soil that protrudes beyond the edge of the retaining ring so that the ring can slide over the soil specimen with little effort. Continue trimming until the soil fills the retaining ring. Trim the top of the specimen flush with the top of the retaining ring. Weigh the specimen in the retaining ring and record the weight to the nearest 0.01 g. Measure the gravimetric water content of the remaining material using Method D 2216.

8.1.5.1 A similar procedure is followed to prepare specimens for Method E, except no trimming ring or dish is employed. Specimens are trimmed from the compacted soil to form a right circular cylinder that fits inside the specimen support chamber. Typically specimens are 38 mm in diameter and 51 mm long (Fig. 9). When trimming is complete, measure the height of the specimen at three locations to the nearest 0.1 mm, and record the average of these measurements as the height. Similarly, measure the diameter of the specimen at three locations to the nearest 0.1 mm, and record the average of these measurements as the diameter. Weigh the specimen to the

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

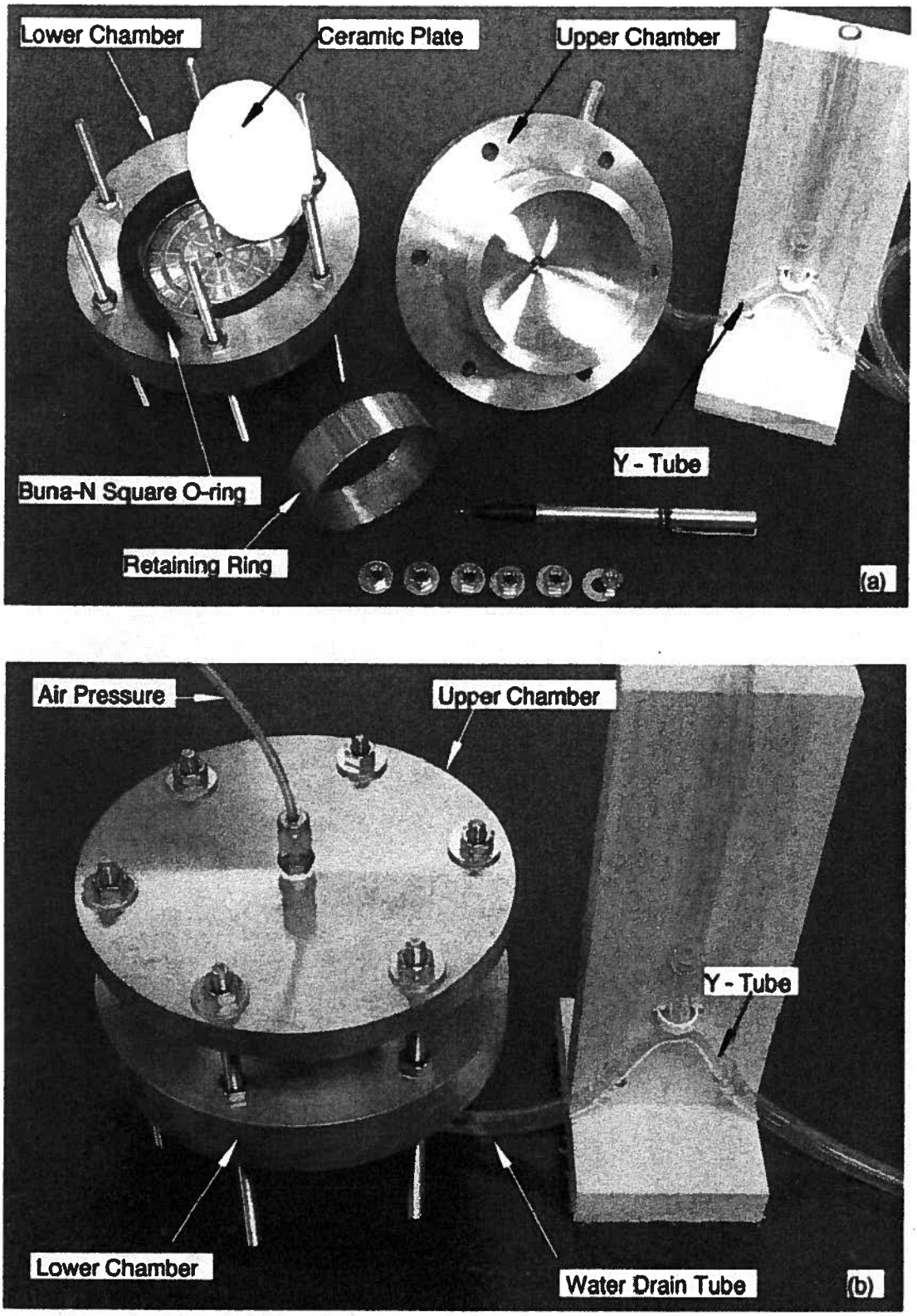
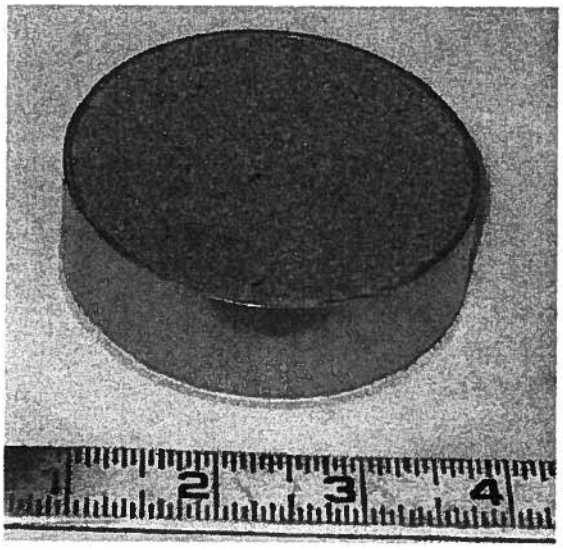


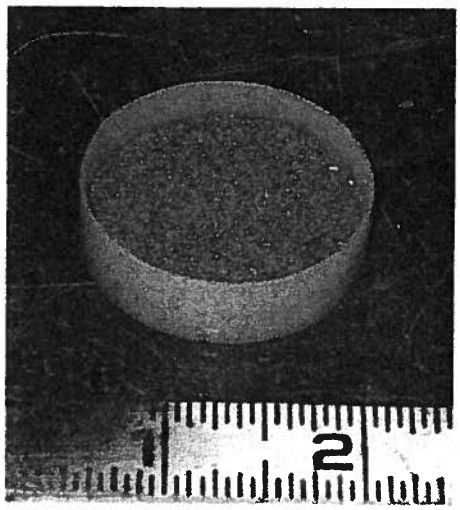
FIG. 5 Photographs of a Single-Specimen Pressure Chamber and Associated Equipment: (a) Open and (b) Assembled

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT



(a)



(b)

FIG. 6 Specimen in Retaining Ring (Methods A-C) (a) and Retaining Dish (Method D) (b)

nearest 0.01 g. The sides of the trimmed specimen are wrapped in unsintered teflon tape. Stainless steel screens (220 mesh) are placed on the ends of the specimen.

8.2 *Saturation of Funnel, Porous Plate, or Membrane:*

8.2.1 *Hanging Column (Method A)*—Immerse the funnel containing the porous plate in a saturation chamber filled with deaerated water under a vacuum of at least 75 kPa (Fig. 10). The funnel shall be immersed for at least 24 h and until no air bubbles are visible.

8.2.2 *Porous Plates (Methods B and C)*—Porous plates shall be submerged in a tray of deaired water or in a saturation chamber under vacuum for a period of at least 24 h and until no gas bubbles are visible. If a saturation chamber is used, the ceramic plates shall initially be soaked for 1 h in a saturation tray and then transferred to the saturation chamber. During saturation, a vacuum of at least 75 kPa shall be applied to the chamber for 24 h. At the end of the 24 h period, the vacuum shall be removed and re-applied, and the specimen inspected for escaping gas bubbles for 1 h. Repeat this procedure until no air bubbles are observed for at least two consecutive applications of vacuum.

8.2.3 *Porous Membrane (Methods B and C)*—Porous membranes are delicate and must be handled with care to prevent damage. A new membrane shall be used for each test. Before

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

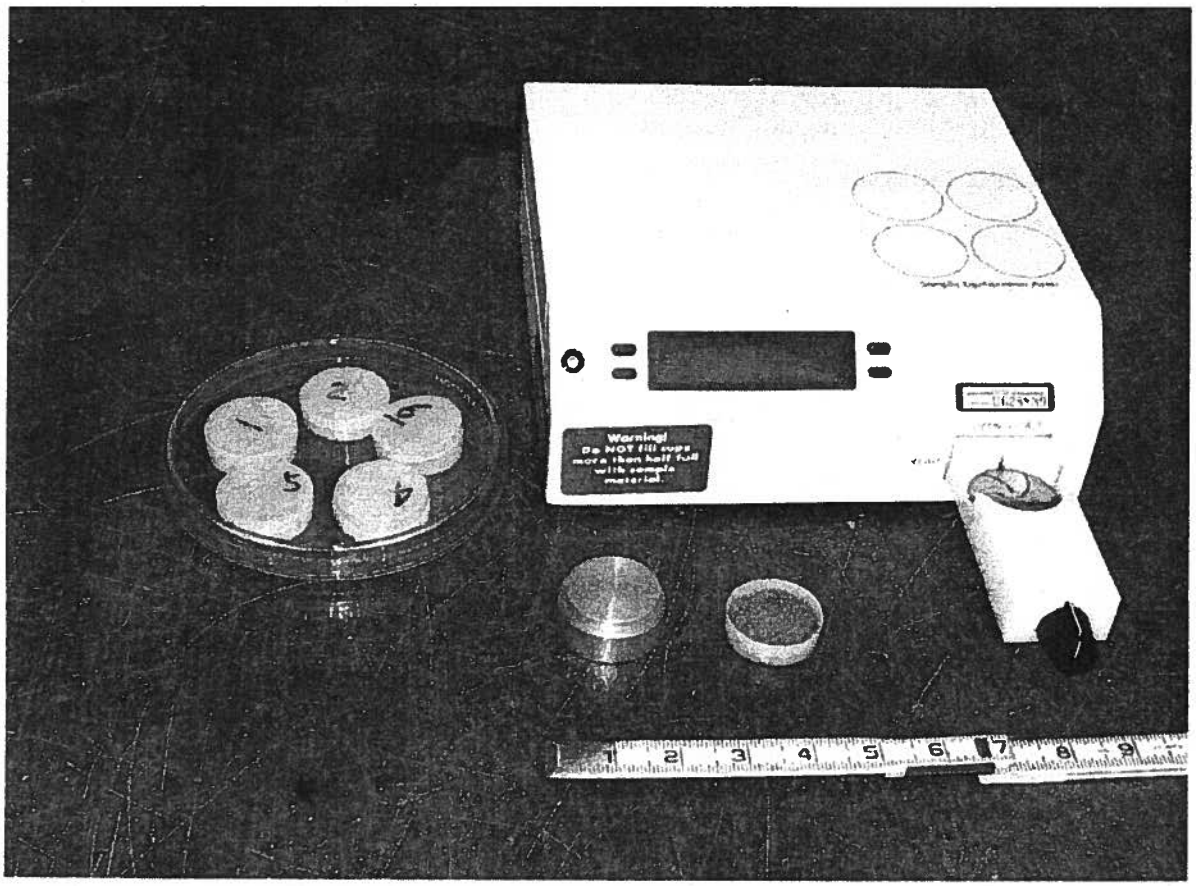


FIG. 7 Photograph of a Chilled Mirror Hygrometer With Open Sample Drawer and Specimen to be Inserted for Testing

handling, porous membranes shall be carefully placed in a pan of warm water for at least 30 min to permit softening. The softening water shall be the same water used for saturation. After softening, saturate the porous membrane by submerging in a tray of deaired water for at least 24 h and until no gas bubbles are visible.

8.3 *Saturation of Specimen (Methods A-E)*:

8.3.1 Specimens shall be saturated by inundation in a saturation tray, a saturation chamber, or directly in the hanging column or pressure chamber. The specimen should be maintained within the retaining ring during saturation to prevent distortion or sloughing. Specimens that are sufficiently cohesive can be placed in the saturation tray on top of a porous material (perforated plastic, wire screen, or non-woven geotextile) without the accompanying ceramic plate. Non-cohesive specimens generally need to be saturated while in contact with a ceramic plate so that the specimen does not become dislodged from the retaining ring. Saturation shall continue for at least 24 h and until no gas bubbles are visible. When the ceramic plate is included during saturation of the specimen, the plate shall be saturated beforehand using the procedure in 8.2.

8.3.2 During saturation in a saturation chamber, apply a vacuum to the chamber of at least 75 kPa for 24 h. At the end of the 24 h period, remove and reapply the vacuum, and then inspect the specimen for 1 h for escaping gas bubbles. Repeat this procedure until no air bubbles are observed for at least two consecutive applications of vacuum. Specimens saturated in a saturation chamber generally are sandwiched between two porous stones and two perforated plastic plates (Fig. 11). The plates are clamped together using bolts.

8.3.3 After saturation in a saturation tray or chamber, weigh the specimen and compute the degree of saturation. Ensure that the degree of saturation is 100 ± 3 %. If the saturation is less than 97 %, repeat the saturation procedure.

8.4 *Check of Porous Plate*:

8.4.1 When first used and every ten uses thereafter, the porous ceramic plate shall be tested to confirm that the air entry pressure is adequate and to check for clogging. Saturate the porous plate using the procedure in 8.2. Install the porous plate in the hanging column or pressure chamber using the methods in Section 9. Elevate the suction in the hanging column or the pressure extractor in increments equal to one-tenth the reported air entry pressure of the plate or the maximum suction to be applied using the apparatus, whichever is lower. Maintain each suction for at least 30 min, and record the total outflow at the end of each 30 min period. Graph the total outflow collected during each increment in suction vs. the total suction applied. Beyond initial expulsion of free water (Fig. 12), the volume of water expelled shall not exceed 0.4 mL. If the volume expelled is larger than 0.4 mL or a slope or break point in the graph exists, discard the plate.

8.4.2 Measure the saturated hydraulic conductivity of the saturated ceramic plate using the method in Test Methods D 5084. If the saturated hydraulic conductivity has decreased

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

FIG. 8 Photograph of a Temperature-Controlled Centrifuge With Various Specimen Support Chambers

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

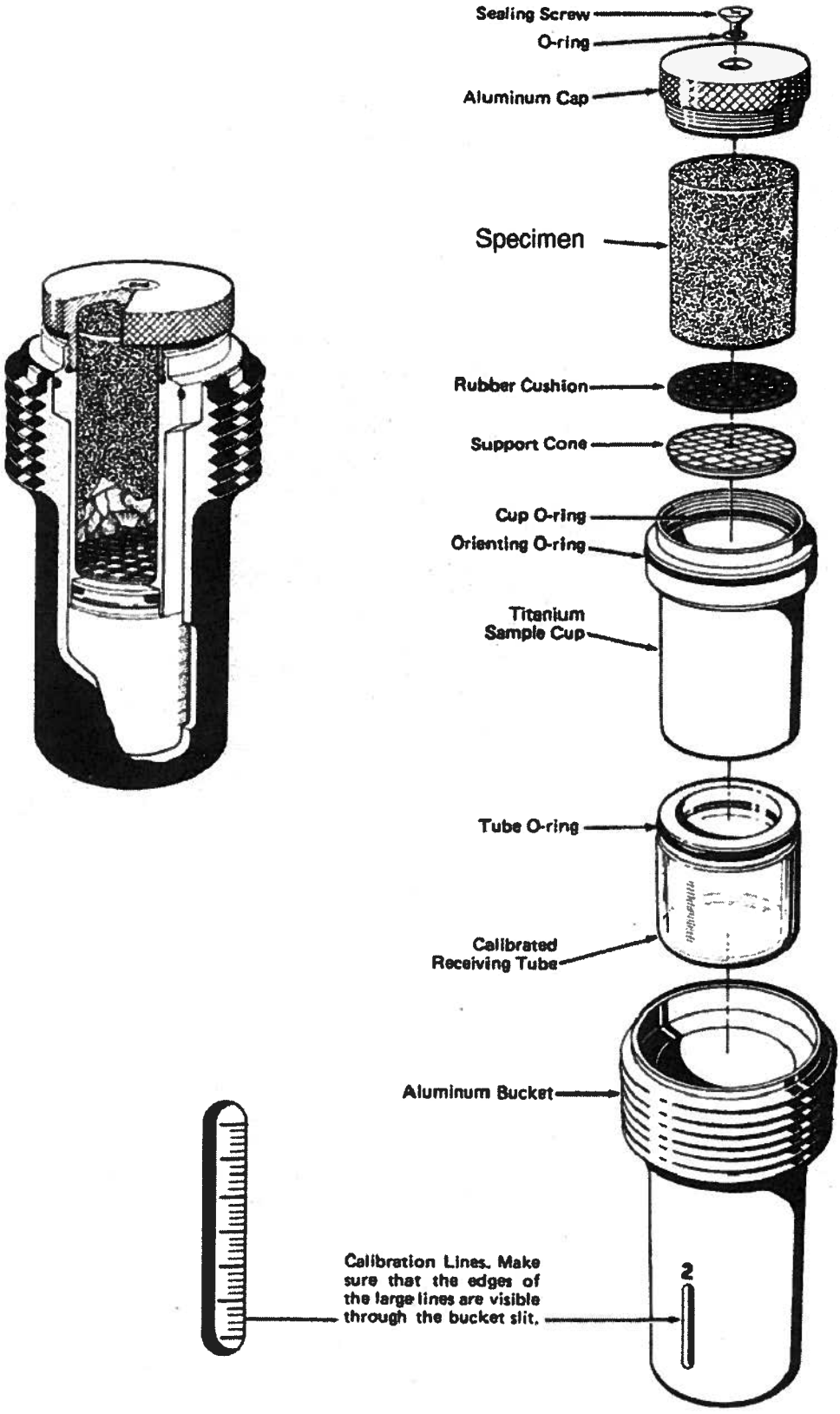


FIG. 9 Schematic of a Specimen Support Chamber

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

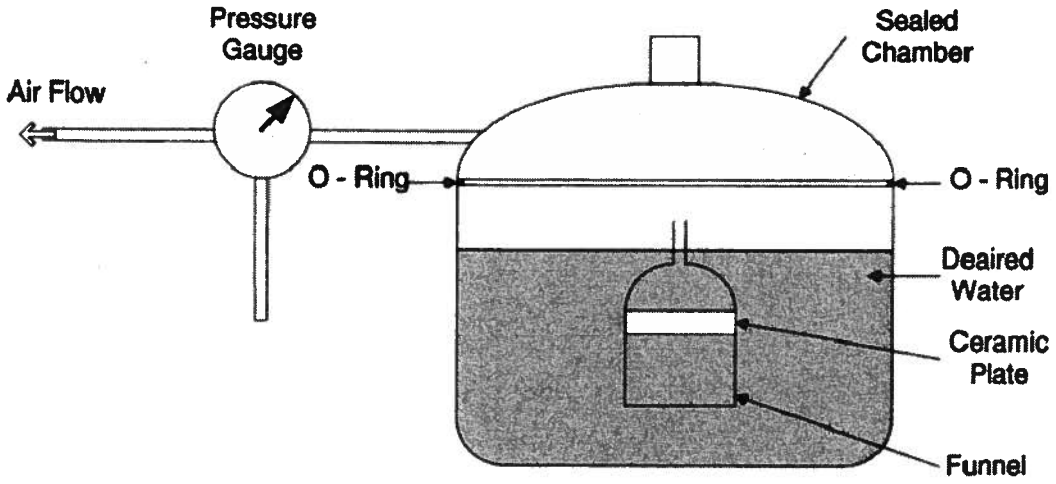


FIG. 10 Schematic of a Saturation Chamber for Saturating Plates and Specimens

more than a factor of five relative to the saturated hydraulic conductivity when new, cleanse the plate or discard the plate. After cleansing, re-check the hydraulic conductivity and the air entry pressure using the aforementioned procedure.

9. Procedure

9.1 *Hanging Column (Method A)*:

9.1.1 *Set-up Apparatus*—Use clear flexible plastic tubing to connect the saturated funnel to the capillary tube (Fig. 1). Use clear flexible plastic tubing to connect the other end of the capillary tube to the reservoir apparatus that applies the suction. Fill the funnel, the tubing connecting the funnel to the capillary tube, and a portion of the capillary tube with the same de-aired water solution used for saturating the specimen. Remove all air-bubbles from all portions of the apparatus filled with water.

9.1.2 *Set Reservoir and Funnel Elevation*—Adjust the relative elevation of the reservoirs so that the elevation difference ψ on the manometer is zero (Fig. 1). Adjust the elevation of the funnel so the top of the porous plate in the funnel is at the same elevation as the centerline of the capillary tube. Remove all excess water above the porous plate using a syringe.

9.1.3 *Insert and Equilibrate Specimen*—Place the specimen contained in the retaining ring on the porous plate. Twist the specimen approximately 45° to ensure good contact between the specimen and the porous plate. If contact is a concern, place a section of rigid screen or geonet on the upper surface of the specimen, and then apply a weight on top of the screen to provide a surcharge. A 1 kg weight typically is used. Secure a thin sheet of plastic film over the top of the funnel to prevent evaporation. Place a small hole in the plastic (for example, with a pin) to ensure equilibrium of air pressure. Allow the specimen to equilibrate for at least 48 h, and until movement of the air-water interface ceases. Record the position of the air-water interface in the capillary tube at the end of the equilibrium period.

9.1.4 *Suction Application*—After equilibration, apply the first increment of suction by adjusting the elevation of the water reservoirs (Fig. 1) until the manometer shows the desired suction has been applied. The first increment in suction should be no more than one-half the anticipated air-entry suction. Monitor the location of the air-water interface regularly until movement of the air-water interface has ceased for at least 24 h. Record the final location of the air-water interface. Then apply the next increment in suction using the same procedure.

NOTE 2—The increments of suction that should be applied depend on the pore size distribution of the specimen and the level of detail desired for the soil water characteristic curve. A common set of increments is 0, 5, 20, 40, 100, 200, 400, 600, 1000, 1500, 2000, and 4000 mm of water.

9.2 *Pressure Chamber with Volumetric Measurements (Method B)*:

9.2.1 *Number of Specimens*—Tests using Method B are normally conducted on a single specimen. Multiple specimens may be tested in a single pressure chamber only if a separate porous plate or membrane is used for each specimen, the plates (or membranes) are not in contact, and a separate outflow tube is provided for each plate.

9.2.2 *Set-up Apparatus*—Place a saturated porous plate (or membrane) in a pressure chamber (Fig. 3) and attach the outflow tube to the porous plate (or membrane). Use clear flexible plastic tubing to connect the outflow tube to the capillary tube as shown in Fig. 13. Saturate the outflow tubing, all connections between the outflow tubing and the porous plate (or membrane), and the flexible tubing connected to the capillary tube with the same de-aired water solution used for saturating the specimen. Remove all air-bubbles from the portions of the apparatus filled with water. Adjust the elevation of the pressure plate (or membrane) so the top of the porous plate is at the same elevation as the centerline of the capillary tube. Remove all excess water above the porous plate using a syringe. Mark the location of the water level in the vent (Y-) tube (Fig. 10). The water level in the vent tube should be at the elevation of the capillary tube and the surface of the porous plate. If the water level is not at this elevation, inspect the system for leaks.

9.2.3 *Insert and Equilibrate Specimen*—Place the specimen contained in the retaining ring on the porous plate (or membrane). Twist the specimen approximately 45° to ensure good contact between the specimen and the porous plate. If contact

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

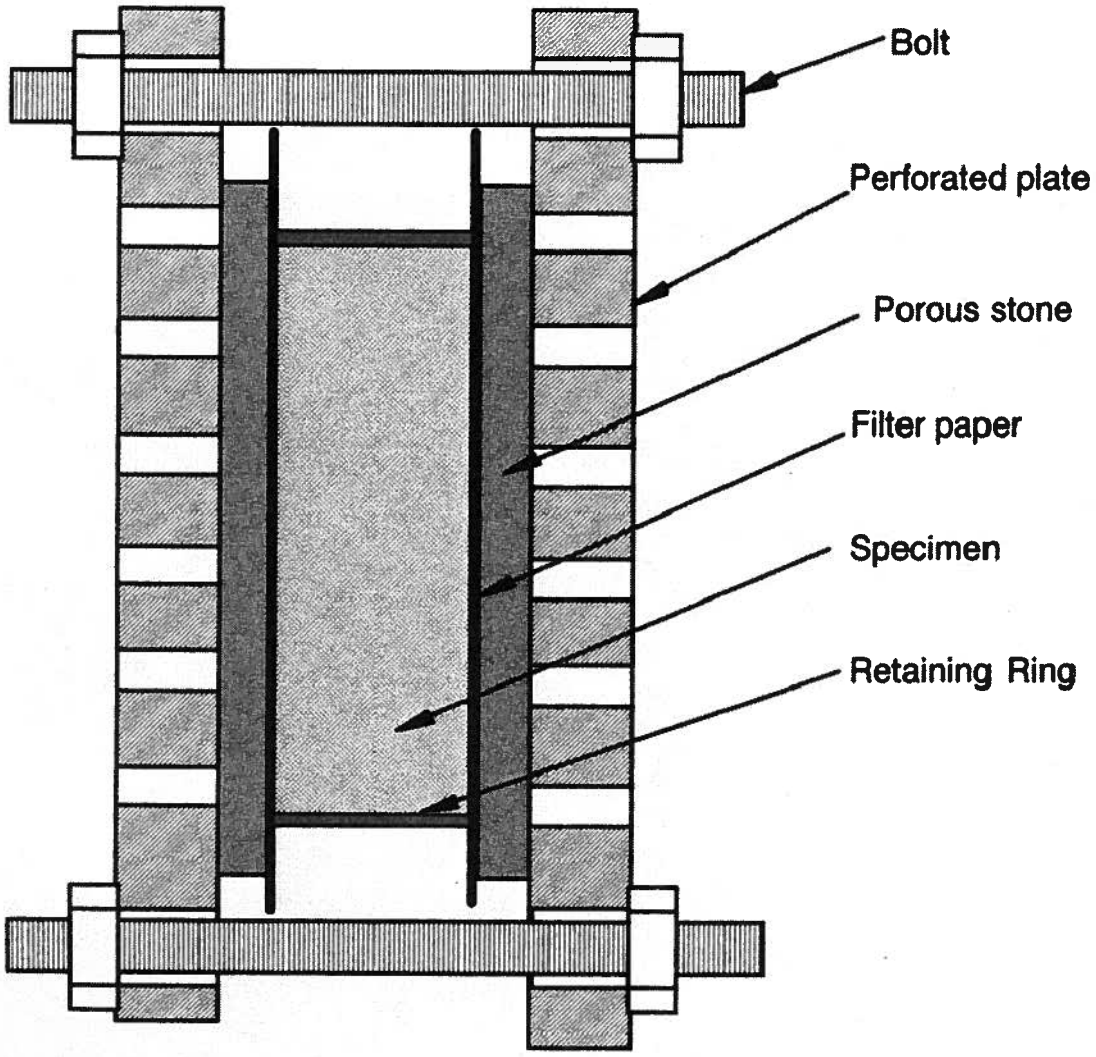


FIG. 11 Assembly Used to Contain Specimens When Saturating in a Saturation Chamber

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

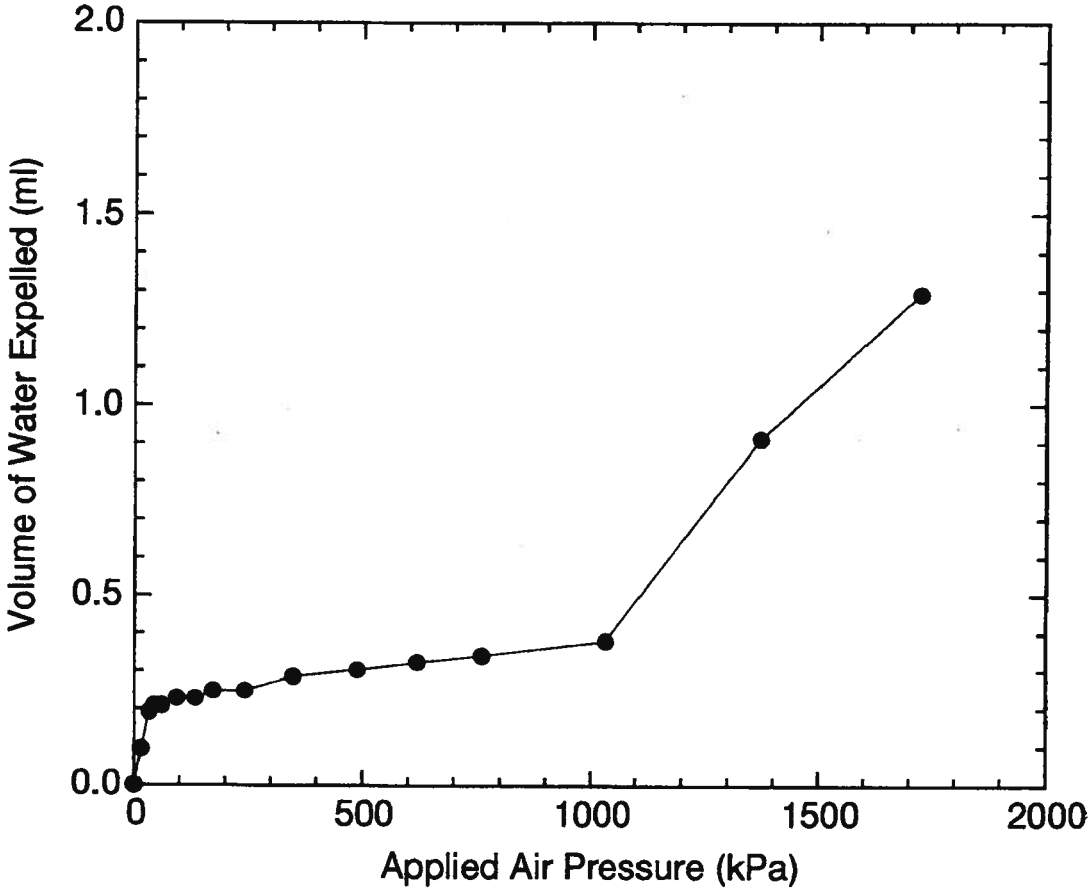


Plate functioned satisfactorily until a gas pressure exceeding 1000 kPa was applied. Subsequently, the volume of water expelled increased dramatically. The plate was discarded.

FIG. 12 Outflow from Check of a Ceramic Plate With a Reported Air Entry Pressure of 1500 kPa

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

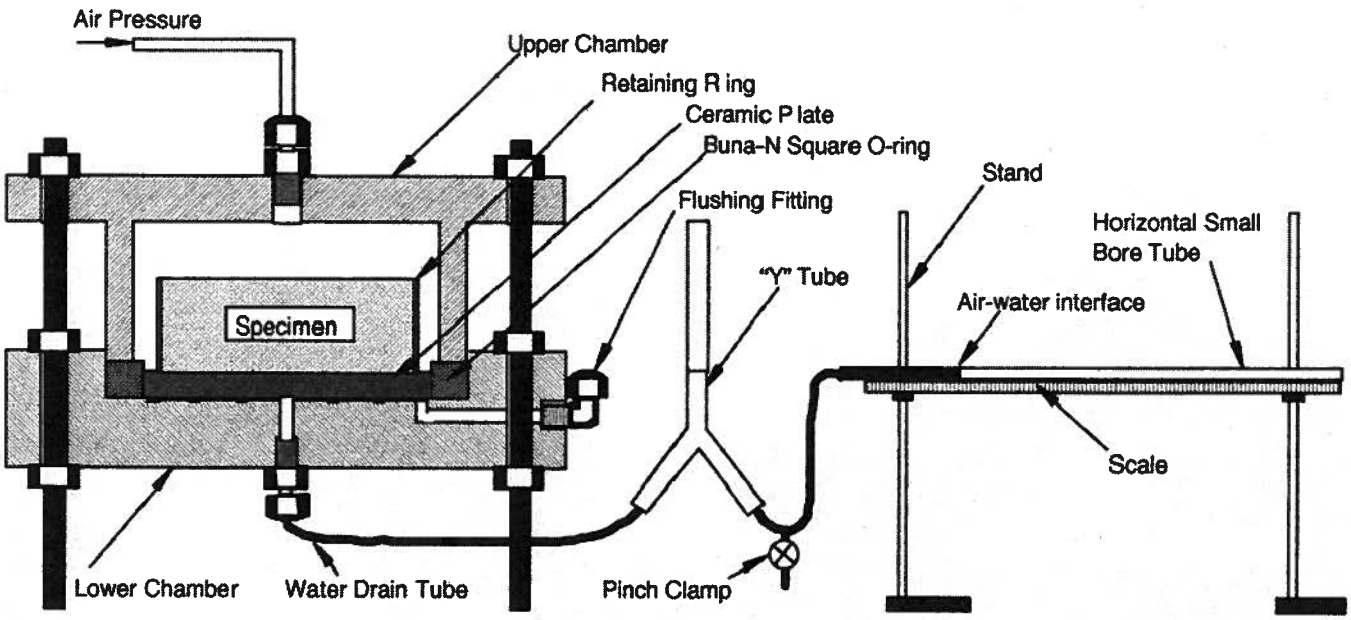


FIG. 13 Schematic of a Pressure Extractor With Ceramic Porous Plate and Capillary Tube for Measuring Outflow as Used In Method B

is a concern, place a section of rigid screen or geonet on the upper surface of the specimen, and then apply a weight on top of the screen to provide to a surcharge. A 1 kg weight typically is used. Close and seal the pressure chamber. Allow the specimen to equilibrate for at least 48 h, and until movement of the air-water interface in the capillary tube ceases. Record the position of the air-water interface in the capillary tube at the end of the equilibrium period.

9.2.4 *Suction Application*—After equilibration, apply the first increment of suction by adjusting the pressure regulators until the desired suction has been applied. The first increment in suction should be no more than one-half the anticipated air-entry suction. Monitor the location of the air-water interface regularly until movement of the air-water interface has ceased for at least 24 h. Record the final location of the air-water interface.

9.2.4.1 After equilibrium has been established, apply the next increment in suction using the same procedure and monitor movement of the air-water interface in the capillary tube until movement of the air-water interface has ceased and equilibrium has been established. For suctions less than 500 kPa, equilibrium is established when the air-water interface has not moved for at least 24 h. For suctions between 500 to 1000 kPa, equilibrium is established when the air-water interface has not moved for at least 48 h. For suctions greater than 1000 kPa, equilibrium is established when the air-water interface has not moved for at least 96 h.

NOTE 3—Regularly inspect the outflow tubing for air bubbles caused by diffusion of air through the porous plate or membrane. Gently tap the tube so that the bubbles flow to the vent tube where they can be expelled (Fig. 13).

NOTE 4—Suctions of 10, 50, 100, 300, 500, 1000, and 1500 kPa are often used to define the soil water characteristic curve using Methods B and C.

NOTE 5—At high suctions (> 1000 kPa), very small volumes of water may be expelled, especially from coarser soils. These volumes may be difficult to measure, and ensuring that equilibrium has been established can be difficult. Accuracy of the dry end of the soil water characteristic curve (that is, suctions exceeding 1000 kPa) can be checked by conducting additional tests using Method D. If the dry end of the soil water characteristic curve differs substantially when measured with Methods B (or C) and D, the data from Method D should be used to define the dry end of the soil water characteristic curve.

9.3 *Pressure Chamber with Gravimetric Measurements (Method C)*:

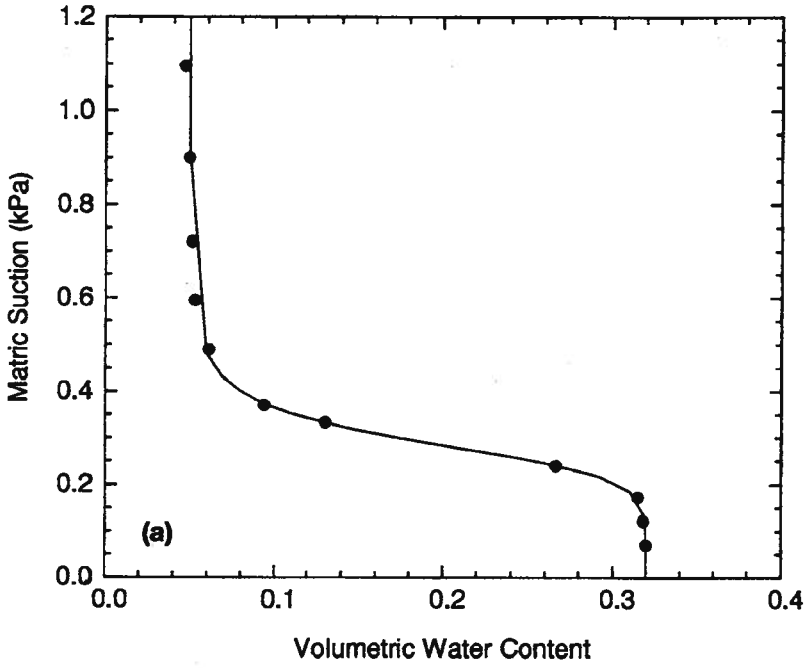
9.3.1 *Duplicates*—For Method C, duplicate tests should be performed when practical on specimens that are as close to identical as possible. Duplicate specimens shall be placed in retaining rings on opposite sides of the porous plate (or membrane). Specimens may not be in contact during the testing procedure.

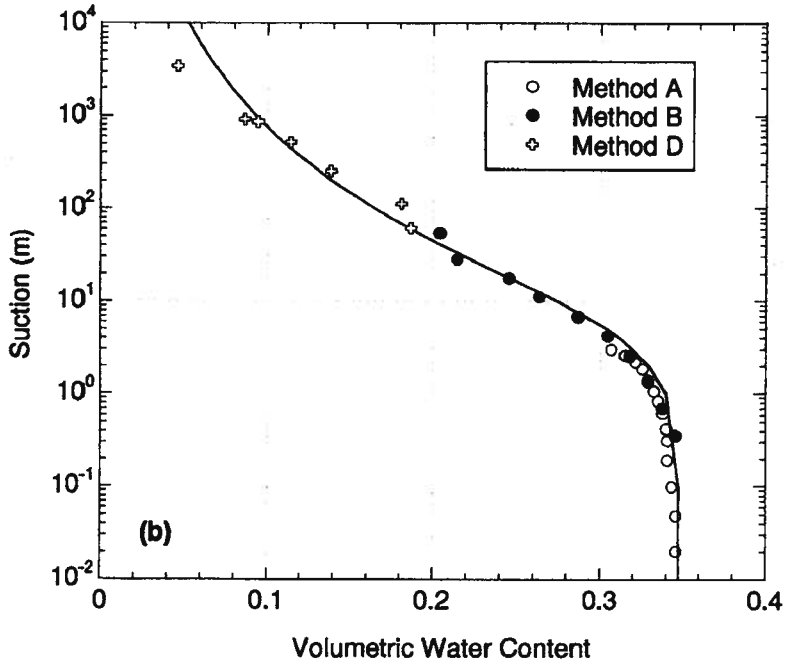
9.3.2 *Multiple Specimens*—Only soils of similar texture may be tested together in one chamber. The time required to reach equilibrium varies greatly with soil texture due to differences in pore size distribution. Consequently, soils of dissimilar texture should not be tested at the same time. When multiple specimens are tested, contact between the specimens shall be prevented.

9.3.3 *Set-up Apparatus*—Place a saturated porous plate (or membrane) in a pressure chamber (Fig. 3a) and attach the outflow tube to the porous plate (or membrane). Direct the outflow tube to a graduated cylinder or similar device that can measure volume with an accuracy of 0.1 mL or better. Saturate the outflow tubing and all connections between the outflow tubing and the porous plate (or membrane) with the same de-aired water solution used for saturating the specimen. Remove all air-bubbles from those portions of the apparatus filled with water. If more than one plate (or membrane) is used in a chamber, the porous plate (or membrane) shall not touch

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT



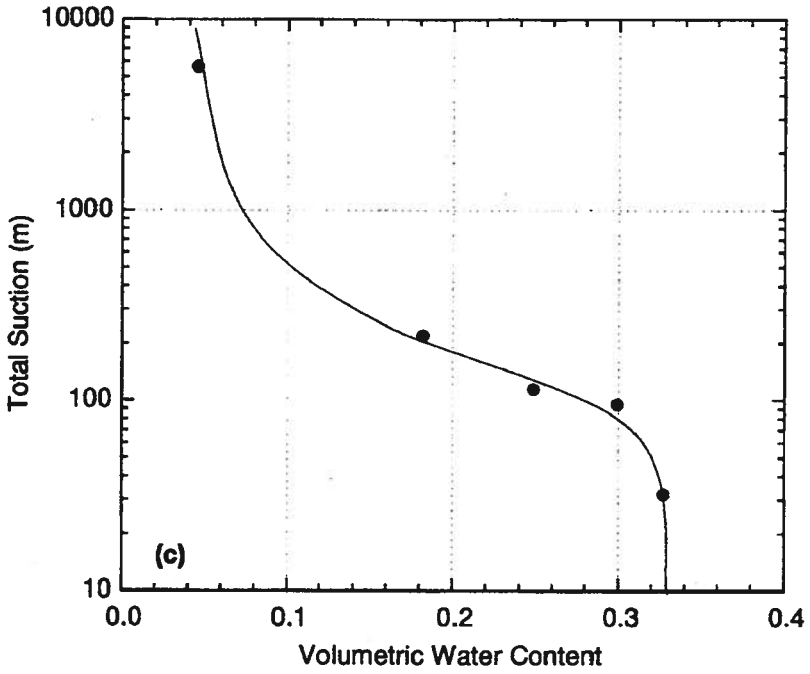


Soil water characteristic curves for (a) a clean medium sand obtained using Method A, (b) a sandy clay using a combination of Methods A, B, an D for low, moderate, and high suctions, and (c) a sandy silt obtained using Method C. All suctions reported in m of water.

FIG. 14 Soil Water Characteristic Curves

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT



Soil water characteristic curves for (a) a clean medium sand obtained using Method A, (b) a sandy clay using a combination of Methods A, B, an D for low, moderate, and high suctions, and (c) a sandy silt obtained using Method C. All suctions reported in m of water.

FIG. 14 Soil Water Characteristic Curves *(continued)*

any specimen except the specimens intended to be in contact with the plate. No contact may exist between the plates.

9.3.4 *Insert and Equilibrate Specimens*—Place the specimens on the porous plate (or membrane). Twist each specimen approximately 45° to ensure good contact between the specimen and the porous plate. Close the pressure chamber. Allow the specimen to equilibrate for at least 48 h. Record the volume of water in the graduated cylinder used to collect outflow at the end of the 48 h period.

NOTE 6—When testing soils using Method C that contain coarse fragments, or soils that may present difficulties in obtaining intimate hydraulic contact between the soil and the porous plate (or membrane), a 6-mm-thick layer of fine silica sand (that is, silica sand passing the No. 200 sieve) may be placed on the surface of the porous plate or membrane to ensure good hydraulic contact. When silica sand is used, a sheet of fine filter paper shall be placed between the soil and the silica sand to prevent penetration of sand into the soil specimen. This contact layer shall not be used in Method B. When contact is a concern for fine soils, the specimen may be surcharged using a weight (1 kg weight is typical). A section of rigid screen or geonet shall be placed between the upper surface of the specimen and the weight to ensure free access of air.

NOTE 7—Specimens tested using Method C are sometimes saturated in the pressure chamber. Saturation in the pressure chamber is generally by soaking in a pool of water that inundates the specimen. The elevation of the pool is increased gradually from an initial depth of approximately 3 mm (±1 mm) in 3-mm increments over a period of several hours until the water just covers the specimen. Saturation is assumed to be complete after the specimen has been inundated for 24 h. If the specimen is saturated in the chamber, all excess water on the plate (or membrane) must be removed (for example, using a syringe) before the chamber is closed and the test is initiated.

9.3.5 *Suction Application*—After equilibration, apply the first increment of suction by adjusting the pressure regulators until the desired suction (gas pressure) has been applied. The first increment in suction should be no more than one-half the anticipated air-entry suction. Record the volume of outflow in the graduated cylinder regularly until water has not been expelled for at least 24 h.

9.3.5.1 After equilibrium has been established, clamp the outflow tube to prevent backflow, exhaust the pressure, and open the pressure chamber. Quickly remove the specimens and their retaining rings from the porous plate (or membrane) using a wide-blade spatula. Immediately weigh the specimens. If the same specimens are to be used throughout the test, place the specimens back on the porous plate (or membrane) and twist each specimen approximately 45° to ensure good contact. Alternatively, a new specimen may be used for each increment provided that that all specimens used to define a soil water characteristic curve are practically identical. Close and seal the pressure chamber, remove the clamp on the outflow tube, and quickly raise the gas pressure to the next suction. Monitor the outflow volume until outflow has ceased for at least 24 h. Repeat this procedure until all suctions specified by the requestor have been determined. When suctions between 500 to 1000 kPa are being established, monitor the outflow volume until the outflow has ceased for 48 h. For suctions greater than 1000 kPa, monitor the outflow volume until outflow has ceased for 96 h. If concern exists regarding equilibrium at high suctions, check this portion of the soil water characteristic curve using the procedure described in Note 5.

NOTE 8—Re-establishing hydraulic contact between the soil specimen and the plate (or membrane) can be difficult after suction has been applied. If concern exists regarding hydraulic contact, separate specimens may be used for each increment. These specimens should be prepared so that they are as identical as possible. If separate specimens are used, the gravimetric water content of each specimen should be determined after testing using

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

the method in Method D 2216.

9.4 *Chilled Mirror Hygrometer (Method D)*:

9.4.1 *Procedure*—Place the soil specimen in a retaining dish and insert the dish into the chilled mirror hygrometer (Fig. 7). Measure the water activity to an accuracy of 0.001 using the hygrometer. Record the water activity.

9.4.1.1 If the soil water characteristic curve is to be determined using a series of specimens prepared at different volumetric water contents, measure the water activity of each specimen using the hygrometer. Then weigh each specimen to the nearest 0.001 g and determine the gravimetric water of each specimen using Method D 2216.

9.4.1.2 If a single specimen is to be used, measure the water activity at the first water content and then allow the specimen to dry by exposure to the atmosphere until the next water content is achieved. Monitor the change in water content by periodically measuring the weight of the specimen and dish to the nearest 0.01 g. After the specimen has dried to the next water content, record the weight of the dish and specimen to the nearest 0.001 g, seal the dish, and allow the specimen to equilibrate for 24 h. After equilibration, measure the water activity using the hygrometer as described in 9.4.1. Repeat this procedure until the water activity has been measured at each water content specified by the requestor. After the final measurement of water activity has been made, measure the final gravimetric water content of the specimen using the method in Method D 2216.

9.5 *Centrifuge (Method E)*:

9.5.1 *Procedure*—Place the specimen in the support chamber and seal the chamber (Fig. 9). Record the distance from the center of the axis of rotation to the bottom of the specimen (outer radius of rotation) to the nearest 0.1 mm. Record the distance from the center of the axis of rotation to the top of the specimen (inner radius of rotation) to the nearest 0.1 mm. Install the support chamber in the centrifuge, and begin centrifugation at an angular velocity corresponding to the lowest suction to be induced (see 10.4). Centrifuge for at least 120 min, and periodically record the volume of water displaced through centrifugation to the nearest 0.1 mL. Continue centrifugation until no additional water is displaced, and record the total volume of water displaced to the nearest 0.1 mL. Repeat the procedure at incrementally higher angular velocities to define the soil water characteristic curve.

NOTE 9—Angular velocities of 100, 200, 400, 800 and 1500 RPM are often used to define the soil water characteristic curve using Method E.

10. Calculations

10.1 *Saturated Volumetric Content*—The water content at zero matric suction is assumed to be the saturated water content. Compute the saturated gravimetric and volumetric water contents using the following methods if the test was conducted using Methods A-E. Saturated specimens are not tested with Method D.

10.1.1 For specimens saturated using a saturation tray or a saturation chamber and then tested using Methods A, B, or E, calculate the saturated gravimetric and volumetric water contents using the dry density recorded after preparation of the specimen, the gravimetric water content at saturation, and the volume of water imbibed or expelled during the saturation phase. The saturated gravimetric water content is computed as:

$$w_{sat} = \frac{M_{sat}(1 + w_m)}{M_m} - 1 \quad (1)$$

where M_m and w_m are the mass and gravimetric water content of the moist soil in the retaining ring after the specimen has been prepared (trimmed or compacted in ring), M_{sat} is the mass of soil in the retaining ring after saturation. The dry density is computed as:

$$\rho_d = \frac{M_m}{V(1 + w_m)} \quad (2)$$

where V is the volume of the retaining ring (Methods A and B). For Method E, V is the volume of the specimen and is computed as:

$$V = \frac{\pi d^2 L}{4} \quad (3)$$

where d is the diameter of the specimen and L is the height of the specimen, as described in 8.1. The saturated volumetric water content is computed as:

$$\theta_{sat} = \frac{\rho_d}{\rho_w} w_{sat} \quad (4)$$

where ρ_w is the density of water.

10.1.2 For specimens saturated directly in a pressure extractor using a saturating apron, compute the saturated volumetric and gravimetric water contents as:

$$\theta_{sat} = 1 - \frac{\rho_d}{G_s \rho_w} \quad (5)$$

and

$$w_{sat} = \frac{\rho_w}{\rho_d} \theta_{sat} \quad (6)$$

where ρ_d is computed using Eq 2.

10.2 *Water Contents at Other Suctions*:

10.2.1 *Methods A and B*—Compute the volumetric water content corresponding to each ith increment in suction, θ_i, as:

$$\theta_i = \theta_{i-1} - (x_i - x_{i-1}) \frac{F}{V} \quad (7)$$

where θ_{i-1} is the volumetric water content at the previous suction, x_i is the location of the air-water interface after equilibrium has been established at the ith suction as described in 9.2.4, x_{i-1} is the location of the air-water interface after equilibrium was established for the previous increment in suction, F is the scale factor for the capillary tube relating volume to displacement of the air-water interface, and V is the volume of the sample. When $i = 1$, $\theta_{i-1} = \theta_{sat}$ and x_{i-1} is the initial reading for the air-water interface that was recorded after initially establishing equilibrium, as described in 9.2.3. The gravimetric water content corresponding to each volumetric water content can be computed as:

$$w_i = \frac{\rho_w}{\rho_d} \theta_i \quad (8)$$

10.2.2 *Method C*—If duplicate specimens are tested, the gravimetric water content of the two replicate specimens shall be averaged for each suction, and the average shall be reported as the gravimetric water content for the applied suction. The

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

volumetric water content corresponding to each gravimetric water content can be computed using:

$$\theta = \frac{\rho_d}{\rho_w} w \quad (9)$$

10.2.3 *Method E*—Compute the volumetric water content corresponding to each *i*th increment in suction, θ_i, as:

$$\theta_i = \theta_{i-1} - \frac{V_{di}}{V} \quad (10)$$

where θ_{i-1} is the volumetric water content at the previous suction, V_{di} is the volume of water displaced by the *i*th suction (or *i*th angular velocity), and V is the volume of the specimen (Eq 3). Compute the *i*th suction in kPa induced as:

$$\psi_i = \beta(\rho_w - \rho_g)\omega_i^2(r_b^2 - r_t^2) \quad (11)$$

where ρ_w is the density of the pore liquid (typically water, 1.0 kg/L), ρ_g is the density of the pore gas (typically assumed to be zero), ω is the angular velocity of the centrifuge (rpm), r_b is the outer radius of rotation (m), r_t is the inner radius of rotation (m), and β is a constant = 0.00553.

10.3 *Total Suctions and Water Content—Method D*—Calculate the total suction for the *i*th test (ψ_{ti}) using the water activity reported by the chilled mirror hygrometer and the Kelvin equation:

$$\psi_{ti} = \frac{RT}{M} ln(a_w) \quad (12)$$

where a_w is the water activity, R is the gas constant, T is the laboratory temperature (°K), and M is the molecular mass of water. The value of R/M is 461 kPa/°K. Temperature in °K is obtained by adding 273° to the temperature in °C.

10.3.1 When suctions corresponding to various water contents are measured using a single specimen (that is, by repeatedly drying the specimen), the gravimetric water content corresponding to the *i*th measurement of suction, w_i, is computed as:

$$w_i = w_o + \frac{M_i - M_o}{\rho_d V} \quad (13)$$

where w_o is the gravimetric water content determined using Method D 2216 for the first suction measurement, M_o is the mass of the specimen for the first suction measurement, M_i is the mass of the specimen for the *i*th suction measurement, ρ_d is the dry density at which the specimen was prepared, and V is the volume of the specimen. Volumetric water contents corresponding to the gravimetric water contents can be determined using Eq 9.

10.3.2 When suctions are measured on individual specimens, the gravimetric water content of each specimen is measured using Method D 2216 after testing in the hygrometer. Volumetric water contents corresponding to these gravimetric water contents can be determined using Eq 9.

10.4 *Saturation*—If the soil water characteristic curve is to be reported in terms of saturation (S) rather than water content, compute the degree of saturation corresponding to each volumetric water content using:

$$S = \frac{\theta}{1 - \frac{\rho_d}{G_s \rho_w}} \quad (14)$$

or to convert gravimetric water content to saturation, use:

$$S = \frac{w}{\frac{\rho_w}{\rho_d} - \frac{1}{G_s}} \quad (15)$$

11. Report

11.1 The report shall include the following information:

11.1.1 Sample identifying information.

11.1.2 The type of water used to saturate and test the soil specimens.

11.1.3 The method of saturation (tray, saturation chamber, or saturating apron in a pressure chamber).

11.1.4 The method used to conduct the test (for example, Method A, B, C, D or E). For Methods B and C, state whether a porous plate or porous membrane was used, the air entry pressure of the porous plate or membrane. For Method D, state the brand and model of the hygrometer that was used. For Method E, state the manufacturer and model number for the centrifuge and specimen support chamber.

11.1.5 A description of the soil, any special characteristics of the specimen, and the selection and/or processing procedure (for example, removal of stones). For compacted materials, provide descriptive information on the method of compaction, the compaction water content, and the compacted dry unit weight.

11.1.6 Dimensions of test specimens.

11.1.7 Time required to reach equilibrium at each pressure step.

11.1.8 Approximate temperature of the soil during the test procedure (typically the air temperature of the laboratory) to the nearest 1°C.

11.1.9 A table reporting the gravimetric and volumetric water content corresponding to each suction.

11.1.10 For Methods A and B, report the gravimetric water content of the specimen after the last increment in suction was applied, as measured using Method D 2216. Report the corresponding volumetric water content, and the difference between this volumetric water content and that computed using Eq 6 for the final increment in suction.

11.1.11 The soil water characteristic curve; that is, a graph showing volumetric water content, gravimetric water content, or saturation as a function of suction. Three examples of soil water characteristic curves are shown in Fig. 12. The soil water characteristic curve in Fig. 12a was determined using Method A. Methods A, B, and D were combined to determine the soil water characteristic curve shown in Fig. 12b. The soil water characteristic curve in Fig. 12b illustrates how Method D can be used to define the dry end of the curve and Method A can be used to define the portion near saturation. The good match between the data from Methods B and D at the dry end indicates that equilibrium was established at the higher suctions when Method B was used. An example of a soil water characteristic curve determined solely with Method D is shown in Fig. 12c.

12. Precision and Bias

12.1 *Precision*—Test data on precision are not presented due to the nature of the geological materials tested by this test method. Having ten or more laboratories participate in a

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

round-robin testing program is considered to be infeasible and too costly. Also, producing multiple specimens that have uniform physical properties is considered infeasible and too costly. Any variation observed in the data is just as likely to be due to specimen variation as to operator or laboratory testing variation.

12.1.1 Subcommittee D18.04 on Hydrologic Properties of Soil and Rock is seeking data from the users of this test method that might be used to make a limited statement on precision.

13. Keywords

13.1 capillary pressure; chilled mirror hygrometer; gravimetric water content; hanging column; matric potential; matric suction; pressure membrane; pressure plate; soil; soil water characteristic curve; suction; total suction; volumetric water content; water activity

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).

Copyright ASTM International
Provided by IHS under license with ASTM
No reproduction or networking permitted without license from IHS

Licensee=Brown & Caldwell/5905905100, User=Isaac, Rick
Not for Resale, 03/29/2012 07:43:37 MDT

Recommended Practices for Core Analysis

RECOMMENDED PRACTICE 40
SECOND EDITION, FEBRUARY 1998

American
Petroleum
Institute

API ENVIRONMENTAL, HEALTH AND SAFETY MISSION AND GUIDING PRINCIPLES

The members of the American Petroleum Institute are dedicated to continuous efforts to improve the compatibility of our operations with the environment while economically developing energy resources and supplying high quality products and services to consumers. We recognize our responsibility to work with the public, the government, and others to develop and to use natural resources in an environmentally sound manner while protecting the health and safety of our employees and the public. To meet these responsibilities, API members pledge to manage our businesses according to the following principles using sound science to prioritize risks and to implement cost-effective management practices:

- To recognize and to respond to community concerns about our raw materials, products and operations.
- To operate our plants and facilities, and to handle our raw materials and products in a manner that protects the environment, and the safety and health of our employees and the public.
- To make safety, health and environmental considerations a priority in our planning, and our development of new products and processes.
- To advise promptly, appropriate officials, employees, customers and the public of information on significant industry-related safety, health and environmental hazards, and to recommend protective measures.
- To counsel customers, transporters and others in the safe use, transportation and disposal of our raw materials, products and waste materials.
- To economically develop and produce natural resources and to conserve those resources by using energy efficiently.
- To extend knowledge by conducting or supporting research on the safety, health and environmental effects of our raw materials, products, processes and waste materials.
- To commit to reduce overall emissions and waste generation.
- To work with others to resolve problems created by handling and disposal of hazardous substances from our operations.
- To participate with government and others in creating responsible laws, regulations and standards to safeguard the community, workplace and environment.
- To promote these principles and practices by sharing experiences and offering assistance to others who produce, handle, use, transport or dispose of similar raw materials, petroleum products and wastes.

Recommended Practices for Core Analysis

Exploration and Production Department

RECOMMENDED PRACTICE 40
SECOND EDITION, FEBRUARY 1998

American Petroleum Institute

SPECIAL NOTES

API publications necessarily address problems of a general nature. With respect to particular circumstances, local, state, and federal laws and regulations should be reviewed.

API is not undertaking to meet the duties of employers, manufacturers, or suppliers to warn and properly train and equip their employees, and others exposed, concerning health and safety risks and precautions, nor undertaking their obligations under local, state, or federal laws.

Information concerning safety and health risks and proper precautions with respect to particular materials and conditions should be obtained from the employer, the manufacturer or supplier of that material, or the material safety data sheet.

Nothing contained in any API publication is to be construed as granting any right, by implication or otherwise, for the manufacture, sale, or use of any method, apparatus, or product covered by letters patent. Neither should anything contained in the publication be construed as insuring anyone against liability for infringement of letters patent.

Generally, API standards are reviewed and revised, reaffirmed, or withdrawn at least every five years. Sometimes a one-time extension of up to two years will be added to this review cycle. This publication will no longer be in effect five years after its publication date as an operative API standard or, where an extension has been granted, upon republication. Status of the publication can be ascertained from the API Authoring Department [telephone (202) 682-8000]. A catalog of API publications and materials is published annually and updated quarterly by API, 1220 L Street, N.W., Washington, D.C. 20005.

This document was produced under API standardization procedures that ensure appropriate notification and participation in the developmental process and is designated as an API standard. Questions concerning the interpretation of the content of this standard or comments and questions concerning the procedures under which this standard was developed should be directed in writing to the director of the Authoring Department (shown on the title page of this document), American Petroleum Institute, 1220 L Street, N.W., Washington, D.C. 20005. Requests for permission to reproduce or translate all or any part of the material published herein should also be addressed to the director.

API standards are published to facilitate the broad availability of proven, sound engineering and operating practices. These standards are not intended to obviate the need for applying sound engineering judgment regarding when and where these standards should be utilized. The formulation and publication of API standards is not intended in any way to inhibit anyone from using any other practices.

Any manufacturer marking equipment or materials in conformance with the marking requirements of an API standard is solely responsible for complying with all the applicable requirements of that standard. API does not represent, warrant, or guarantee that such products do in fact conform to the applicable API standard.

All rights reserved. No part of this work may be reproduced, stored in a retrieval system, or transmitted by any means, electronic, mechanical, photocopying, recording, or otherwise, without prior written permission from the publisher. Contact the Publisher, API Publishing Services, 1220 L Street, N.W., Washington, D.C. 20005.

Copyright © 1998 American Petroleum Institute

FOREWORD

API publications may be used by anyone desiring to do so. Every effort has been made by the Institute to assure the accuracy and reliability of the data contained in them; however, the Institute makes no representation, warranty, or guarantee in connection with this publication and hereby expressly disclaims any liability or responsibility for loss or damage resulting from its use or for the violation of any federal, state, or municipal regulation with which this publication may conflict.

Suggested revisions are invited and should be submitted to the director of the Exploration and Production Department, American Petroleum Institute, 1220 L Street, N.W., Washington, D.C. 20005.

SCOPE

These recommended practices for core analysis replace API RP 40, *Recommended Practice for Core Analysis Procedure*, 1960, and API RP 27, *Recommended Practice for Determining Permeability of Porous Media*, 1952, (reissued 1956). In the first section of the new recommended practices, Planning a Coring Program, key factors to be taken into consideration in obtaining core samples are explained and advantages of different coring procedures are given. The second section, Wellsite Core Handling Procedures and Preservation, addresses documentation of coring conditions and how cores should be handled once they reach the surface, including marking and preservation. The third section, Core Screening and Core Preparation, describes how the condition and nature of core samples can be documented through core gamma logs and various imaging techniques, and how samples should be selected and prepared for basic testing. Also covered in the third section are methods of preserving samples prior to testing, and procedures for cleaning and drying samples. The fourth section, Fluid Saturation, explains how fluid saturations can be determined on different types of samples and the limitations of the various techniques. The fifth section, Porosity Determination, defines different types of porosity and explains the measurements. The sixth section, Permeability Determination, explains the theory and methods for measurement of permeability of porous media to a single phase. Relative permeability measurements to two or three phases are not covered in this document. The seventh section, Supplementary Tests, covers determination of grain size, brine salinity, oil gravity, and acid solubility. The eighth and final section, Reporting, supplies forms to assist in recording the details of core handling and testing methodology that could be critical in interpreting basic core analysis data.

Core analysis, like other technical areas, is continually evolving so that both methodology and costs are changing. The recommended practices provided here represent a snap shot in time of the consensus advice of a large international committee.

Geologic materials come in a vast range of chemical compositions and physical states. For unusual samples or extraordinarily accurate data, it may be necessary to develop special procedures.

CONTENTS

Page

1 PLANNING A CORING PROGRAM 1-1

2 WELLSITE CORE HANDLING PROCEDURES AND PRESERVATION 2-1

3 CORE SCREENING AND CORE PREPARATION 3-1

4 FLUID SATURATION 4-1

5 POROSITY DETERMINATION 5-1

6 PERMEABILITY DETERMINATION 6-1

7 SUPPLEMENTARY TESTS 7-1

8 REPORTING 8-1

SECTION 1—PLANNING A CORING PROGRAM

CONTENTS

Page

1 PLANNING A CORING PROGRAM 1-1
1.1 General 1-1
1.2 Coring Equipment 1-1
1.3 Conventional Coring Systems 1-2
1.4 Special Coring Systems 1-3
1.5 Wireline Sidewall Coring 1-4
1.6 Oriented Coring 1-5
1.7 Coring Bits 1-5
1.8 Fluid Discharge Characteristic of Core Bits 1-6
1.9 Core Catchers 1-6

Tables
1-1 Conventional Coring Systems 1-2
1-2 Special Coring Systems 1-3
1-3 Core Orientation Methods 1-5
1-4 General Coring Bit Guide 1-6
1-5 Core Catchers 1-6

Recommended Practices for Core Analysis

1 Planning a Coring Program

1.1 GENERAL

1.1.1 Scope

This section addresses the complexities of planning a coring program, the decisions to be made, and the factors that influence the choices.

1.1.2 Principle

A coring program is similar to many engineering projects. It begins with the premise that an investment will reap a reward. It progresses through a phase of exploring alternate sources of information; well tests, logs, previous cores, and cuttings or sidewall cores.

Planning begins by listing the objectives of the coring program. This is best done by a team of petrophysical, reservoir, geological, drilling, and production personnel. When discussing objectives, every expenditure must ultimately lead to producing more oil or gas at a lower unit cost. Constraints in budget, location, and timing will be placed on the program. Hole size, hole angle, temperature, pressure, and rock type will influence the selection of the coring tools. Planning becomes an interactive process where consensus is built and a detailed program formulated.

The keys to a successful coring operation are planning and communication.

1.1.3 Objective

The objective of every coring operation is to gather information that leads to more efficient oil or gas production. Some specific tasks might include the:

a. Geologic objectives:
   1. Lithologic information:
      (a) Rock type.
      (b) Depositional environment.
      (c) Pore type.
      (d) Mineralogy/geochemistry.
   2. Geologic maps.
   3. Fracture orientation.

b. Petrophysical and reservoir engineering:
   1. Permeability information:
      (a) Permeability/porosity correlation.
      (b) Relative permeability.
   2. Capillary pressure data.
   3. Data for refining log calculations:
      (a) Electrical properties.
      (b) Grain density.
      (c) Core gamma log.
      (d) Mineralogy and cation exchange capacity.
   4. Enhanced oil recovery studies.
   5. Reserves estimate:
      (a) Porosity.
      (b) Fluid saturations.

c. Drilling and completions:
   1. Fluid/formation compatibility studies.
   2. Grain size data for gravel pack design.
   3. Rock mechanics data.

1.1.4 Coring Fluids

1.1.4.1 The selection of a coring fluid should be based on four points:

a. Safety.
b. The primary objective of the coring program.
c. Environmental concerns.
d. Cost.

1.1.4.2 Safety takes precedence over all other factors. The drilling fluid must be designed to hold the expected formation pressures as well as clean, lubricate, and stabilize the borehole. The objectives of the coring program should influence the selection of the coring/drilling fluid. All coring fluids should be designed to have low static API filter loss and very low dynamic spurt loss to minimize core flushing.

1.1.4.3 Environmental concerns should also be considered and budgeted for. This may mean using a more expensive drilling fluid system to meet environmental objectives, or providing additional drilling fluid handling equipment to ensure containment.

1.1.4.4 Cost is important; still, it is a good practice to review the cost of the entire core analysis program and the expected benefits from it while pricing drilling fluid systems. Savings on drilling fluids may increase the cost of the core analyses, and put the accuracy of the core studies at risk.

1.1.4.5 The question of which drilling fluid is best for coring cannot be answered directly. Water-based, oil-based, foam, and air/mist drilling fluids have all been used to successfully cut cores. The best recommendation is to follow the criteria given above. Evaluating the needs of the drilling and core analysis program will lead to an appropriate selection.

1.2 CORING EQUIPMENT

1.2.1 Scope

This section presents an overview of coring tools, including guidelines for selecting coring tools for specific applications. Details of particular coring systems, and job specific coring recommendations should be obtained from appropriate service companies.

1.2.2 Principle

Coring equipment is designed to retrieve rock samples from deep in the earth for geologic and engineering studies. The tools do an excellent job of recovering core material, and specialized equipment has been developed to trap reservoir fluids and even seal in bottom-hole pressure.

1.2.3 Apparatus

With several notable exceptions coring systems consist of an inner core barrel suspended by a swivel assembly within an outer core barrel that is attached to the drill string. A coring bit is attached to the bottom of the outer barrel and a core catcher is fitted to the bottom of the inner core barrel. Drilling fluid is pumped down the drill string, through the swivel assembly, through the annulus between the inner and outer core barrels, and out the core bit.

1.3 CONVENTIONAL CORING SYSTEMS

1.3.1 Conventional Core Barrel

Conventional coring tools are available to cut cores with outer diameters from 1.75 to 5.25 inches (44.5 to 133.4 millimeters). Core length can run from 1.5 feet (.46 meter) for short radius horizontal well applications to over 400 feet (121.9 meters) for thick, uniform, consolidated formations. Hole size, hole angle, rock strength, and lithology will control the diameter and length of core that may be cut in one trip. The final selection of a particular system will depend upon the formation, location, and objectives of the coring program. Table 1-1 summarizes the conventional coring options available.

1.3.2 Heavy-Duty Conventional Core Barrels

Special heavy-duty coring tools have been developed to core harder than normal formations, and cut extended length cores. Heavy duty threads allow more torque to be applied to the bit, and improve the margin of safety against tool failure. Designed to cut cores up to 5.25 inches (133.4 millimeters) in diameter, these tools are especially attractive in situations where rig time is the largest coring expense. Heavy-duty coring systems are used to best advantage when coring longer lengths of homogeneous formations or when anticipating higher than normal torque loads.

The marine core barrel was the precursor to today's generation of heavy-duty core barrels. Developed to be stronger than existing coring systems, the tool was developed for use in offshore applications. The marine core barrel does increase the margin of safety against tool failure, but is restricted to cutting a 3-inch (76.2-millimeter) diameter core.

1.3.3 Core Barrel Liners

The use of a core barrel liner in a steel inner core barrel has two primary functions: to improve core quality by physically supporting the core material during handling and to serve as a core preservation system. PVC and ABS plastic, fiberglass, and aluminum have all been used as inner core barrel liners. The liners slip inside a conventional inner core barrel and are held in place by the core-catcher assembly and friction. Liners are typically 30 feet (9.14 meters) long. They may be cut shorter for special applications, but their maximum length is rarely more than 30 feet (9.14 meters) due to manufacturing and material handling limitations.

Liners are most often specified when coring unconsolidated or fractured formations. They are also appropriate when cutting hard rock in remote and offshore locations when immediate core preservation is required. Plastic liners are suitable up to temperatures of 180°F (82.2°C). Fiberglass liners may be used up to 250°F (121°C); 350°F (176.7°C) if special high temperature resin is used. Aluminum is generally recommended when temperatures in excess of 250°F (121°C) are expected. The disadvantage of core barrel liners is that

Table 1-1—Conventional Coring Systems

Inner Barrel	Core Length	Special Features
Mild steel	30 to 120 ft. (9.14 to 36.58 m)	Ready-made core preservation system. High temperature applications.
Mild steel	1.5 ft. (.46 m)	Designed for short-radius coring.
High strength steel	120 to >400 ft. (36.38 to >121.9 m)	Stronger barrel, includes additional inner and outer core barrel stabilization.
Fiberglass	30 to 90 ft. (9.14 to 27.43 m)	Ready-made core preservation system. Used for consolidated and unconsolidated formations. Maximum operating temperatures: normal resin 250°F (121°C), high temperature resin 350°F (176.7°C).
Aluminum	30 to 90 ft. (9.14 to 27.43 m)	Ready-made core preservation system. High temperature applications, maximum 350°F (176.7°C).
Steel with a plastic liner	30 ft. (9.14 m)	Ready-made core preservation system. Maximum temperature of 180°F (82.2°C). Reduces core diameter by $^1/_2$ in. (12.7 mm).
Steel with a fiberglass liner	30 ft. (9.14 m)	Ready-made core preservation system. Maximum temperature of 250°F (121°C). Reduces core diameter by $^1/_2$ in. (12.7 mm).
Steel with an aluminum liner	30 ft. (9.14 m)	Ready-made core preservation system. Maximum temperature of 350°F (176.7°C). Reduces core diameter by $^1/_2$ in. (12.7 mm).

they reduce the effective diameter of the inner core barrel by approximately 0.5 inch (12.7 millimeters).

1.3.4 Disposable Inner Core Barrels

Disposable inner core barrels serve the same general purposes as core barrel liners. They improve core quality by physically supporting the core material during handling and serve as a core preservation system. In addition, the outside diameter of the core is not reduced, as it would be with an inner barrel liner. Disposable inner core barrels are available in aluminum, fiberglass, and mild steel, and are manufactured in a variety of sizes to fit most conventional coring systems. In addition, the fiberglass inner core barrel has a low coefficient of friction that allows the core to slide more easily into the core barrel, thereby reducing the risk of core jamming.

1.3.5 Coring High Angle or Horizontal Well

Medium radius [290 to 700 feet (88.4 to 213.4 meters) radius] and extended length wells can be cored with conventional core barrels powered from the rotary table or by a downhole motor. Most cores will be cut without using a downhole motor, but cases will arise where the use of a mud motor is justified. Using a downhole motor enables coring to proceed without rotating the drill string. Typically a 30-foot (9.14-meter) long conventional core barrel would be placed ahead of the downhole mud motor. Mud motors produce high torque at low rotating speed for optimum coring power. Core barrel length and core diameter may be varied to accommodate drilling constraints. The inner core barrel is stabilized by fitting it with special roller bearing or bushing assemblies to centralize the inner core barrel. A special drop ball sub may be placed between the motor and core barrel to allow drilling fluid to flow through the inner core barrel, cleaning it of debris before coring. Activating the sub diverts the drilling fluid flow between the inner and outer core barrel for coring.

In some instances during coring it may be necessary to keep very tight control on the angle of the well. Coring without the downhole motor may improve well-angle control.

1.4 SPECIAL CORING SYSTEMS

1.4.1 General

Special coring systems have evolved to fill specific coring needs. Pressure-retained and sponge core barrels arose from a need for better oil saturation data. The rubber-sleeve and full-closure coring systems were developed specifically to improve the quality of cores cut from unconsolidated formations. Other special coring systems have equally unique capabilities, making them all useful to the engineers and geologists employing them. Table 1-2 summarizes some of the available special coring options.

1.4.2 Pressure-Retained Coring

Pressure-retaining core barrels are designed to retrieve cores maintained at reservoir pressure conditions. Accepted as the best method for obtaining core-based oil saturation data, pressure-retained cores also capture reservoir gases. The tool is especially useful when studying the feasibility of enhanced recovery projects and estimating the methane content of coal.

Pressure-retained core barrels are available in two sizes: 6-inch (152.4-millimeter) and 8-inch (203.2-millimeter) outside diameter that cut cores 2.50- and 3.75-inch (63.5- and 95.3-millimeter) outside diameter, respectively. The 6-inch (152.4-millimeter) outside diameter barrel cuts up to 20 feet (6.1 meters) of 2.5-inch (63.5-millimeter) diameter core while holding a maximum of 10,000 psi (69 MPa) pressure. The 8-inch (203.2-millimeter) outside diameter barrel cuts 10 feet (3.05 meters) of 3.75-inch (95.3-millimeter) diameter core while retaining a maximum of 5,000 psi (34.5 MPa) internal pressure. The maximum recommended operating temperature is 180°F (82°C).

Pressure core barrels are sophisticated tools requiring an on-site facility to service the barrel and handle the pressurized cores. Core handling procedures may be found in 2.2.5.

Table 1-2—Special Coring Systems

Coring System	Maximum Core Dimensions	Special Applications
Pressure-retained	3.75 in. x 10 ft. (5000 psi) [95.3 mm x 3.05 m (34.5 MPa)] 2.5 in. x 20 ft. (10000 psi) [63.5 mm x 6.1 m (69 MPa)]	Pressure-retained analyses, fluid saturations, gas volume and composition.
Sponge-lined	3.5 in. x 30 ft. (88.9 mm x 9.1 m)	Fluid saturations.
Full-closure	4.0 in. x 60 ft. (101.6 mm x 18.3 m)	Recovering unconsolidated formations.
Rubber-sleeve	3.0 in. x 20 ft. (76.2 mm x 6.1 m)	Recovering unconsolidated, fractured, or conglomeritic formations.
Wireline retrievable	2.75 in. x 30 ft. (69.9 mm x 9.1 m)	Coring is possible without tripping pipe.
Wireline percussion sidewall	1 in. x 1.75 in. (25.4 mm x 44.5 mm)	Samples obtained after drilling and logging.
Wireline drilled sidewall	.94 in. x 1.75 in. (23.9 mm x 44.5 mm)	Samples obtained after drilling and logging.
Sidewall corer	2.5 in. x 10 ft. (63.5 mm x 3.05 m)	Core obtained after drilling and logging.

1.4.3 Sponge-Lined Coring System

The sponge-lined coring system was developed to improve the accuracy of core-based oil saturation data. A sponge coring system does not trap reservoir gases, instead it traps oil expelled as the core is brought to the surface. The saturation information is very useful when evaluating enhanced oil recovery projects.

A sponge coring system has the advantage of being less expensive to operate than a pressure-retained coring system, while providing an opportunity to improve the accuracy of the core based oil saturation data. The sponge is stable to a temperature of 350°F (176.7°C). The sponge coring system is limited to cutting a maximum of 30 feet (9.14 meters) of 3.5-inch (88.9-millimeter) diameter core per trip.

1.4.4 Full-Closure Coring Systems

Full-closure coring systems were developed to improve the recovery of unconsolidated formations. These systems use core barrel liners or disposable inner core barrels, and a special core catching system to retrieve the troublesome rocks.

Full-closure coring technology allows the inner core barrel to slip gently over soft core with a minimum of disturbance, and then seal the core within the core barrel. This is done by using a full-closure core catcher assembly that allows unobstructed entry of the core into the inner core barrel, and then after coring seals off the bottom of the inner barrel. Full-closure coring systems are currently limited to cutting either 3.5-inch (88.9-millimeter) or 4-inch (101.6-millimeter) diameter cores. The recommended core length is 30 feet (9.14 meters). The smooth bore and the absence of an exposed core catcher may result in lost core if the tool is lifted off bottom before activating the full-closure core catcher.

1.4.5 Rubber-Sleeve Core Barrel

The rubber-sleeve coring system was the first system developed to improve the chances for recovering unconsolidated sands, conglomerates, and hard fractured formations. The rubber-sleeve barrel is unique in that the top of the inner barrel does not move relative to the core during coring. The outer barrel is drilled down around a column of rock that is progressively encased in a rubber sleeve. The rubber sleeve is smaller than the diameter of the core; it stretches tightly around the core, wrapping it securely and protecting it from the scouring action of the drilling fluid. The core is supported by the rubber sleeve thus, aiding in the recovery of soft formations that would not support their own weight.

There is only one size of rubber-sleeve core barrel, that cuts 20 feet (6.1 meters) of 3-inch (76.2-millimeter) diameter core per trip. The rubber sleeve itself is limited to temperatures no higher than 200°F (93°C). The tool is not recommended for use in holes with more than 45 degrees of inclination. In addition, coring must be stopped approximately every two feet to allow the tool to be reset; this might lead to core jamming in fractured formations. The system works best from fixed drilling structures, yet it can be operated from floating rigs if rig movement is minimal.

1.4.6 Wireline-Retrievable Core Barrel

Wireline-retrievable coring tools are operationally similar to conventional coring systems except they are designed for the inner core barrel to be pulled to the surface by a wireline. This speeds the coring operation by eliminating the need to trip the entire drill string for each core. A new section of inner core barrel is pumped down the drill string and latched into place for additional coring, or a drill plug is pumped down to facilitate drilling ahead.

Wireline-retrievable coring tools are usually smaller and lighter than conventional coring systems. This is an asset when they must be transported to remote locations or by helicopter. Unfortunately, the core diameters are limited since the entire inner core barrel assembly must pass through the drill string. Also, care must be taken to prevent “swabbing” oil or gas into the wellbore as the inner barrel is recovered.

1.5 WIRELINE SIDEWALL CORING

1.5.1 General

Wireline sidewall coring systems were developed to obtain core samples from a wellbore after it has been drilled and logged, and before casing is run. These tools may be positioned in zones of interest using data from gamma or spontaneous potential logs as guides. The samples provide small pieces of formation material, suitable for geologic and engineering studies.

1.5.2 Percussion Sidewall Coring

Most wireline sidewall cores are obtained by percussion sidewall coring systems. These tools shoot hollow, retrievable, cylindrical bullets into the wall of an uncased hole. The tool (gun) is lowered to the desired depth on a wireline, and then fired by electrical impulses controlled from the surface. The bullets remain connected to the gun by wires, and movement of the gun pulls the bullets, containing the samples, from the hole wall. Up to 66 samples, 1 inch (25.4 millimeters) in diameter by $1^{3}/_{4}$ inches (44.5 millimeters) in length, may be taken during one downhole trip. Different bullet “core barrel” designs are available for unconsolidated, soft, and medium-to-hard formations. It is wise to have more than one type of core barrel on location until acceptable core recovery can be shown.

The advantages of percussion sidewall coring are speed, low cost, and the ability to sample zones of interest after open hole logs have been run. The disadvantage is that the bullet usually alters the formation, shattering harder rock or compressing softer sediments. This reduces the quantitative value of the sidewall core analysis data. Percussion sidewall core

recovery tends to be low in very hard or fractured rock, and in very permeable unconsolidated sand.

1.5.3 Drilled Sidewall Coring

The rotary or drilled sidewall coring tool was developed to recover wireline sidewall core samples without the shattering impact of the percussion system. Suitable for hard-to-friable rock, the rotary sidewall coring tool uses a diamond-tipped drill to cut individual samples. Leverage applied to the drill snaps the sample from the sidewall. The drill and sample are retracted into the body of the tool where the sample is deposited. The tool is moved to a new location after depositing each sample. A maximum of 30 samples, $^{15}/_{16}$-inch (23.9 millimeter) diameter by $1^{3}/_{4}$-inch (44.5-millimeter) length, may be taken during one trip.

An advantage of the rotary sidewall coring system is that it produces samples of hard rock suitable for quantitative core analysis. Disadvantages are that it is more expensive than percussion sidewall coring in terms of rig time costs, and sample recovery tends to be low in unconsolidated formations.

1.5.4 Sidewall Coring Systems

Some new sidewall coring systems are coming on the market, and they merit discussion for two reasons. First, they are designed to acquire a larger, more-continuous core sample from a drilled and logged wellbore than is possible with existing sidewall coring tools. Secondly, the emergence of new tools confirms there is still room for improvement in the area of acquiring high quality, low cost core samples.

The first system is similar to a conventional core barrel. The sidewall coring system is designed to cut up to 10 feet (3.05 meters) of $2^{1}/_{2}$-inch (63.5-millimeter) diameter core. The tool is attached to a conventional drill string and lowered to the zone of interest. There an integral arm pushes the core barrel against one side of the wellbore. From then on the tool is operated as a conventional core barrel. The second system uses a removable whipstock to direct a conventional core barrel out into the formation. Both systems address the need to acquire quality core samples after logging.

1.6 ORIENTED CORING

1.6.1 General

Oriented cores are used to orient fractures, stress fields, and permeability trends. Exploration, production, and drilling operations use the information to explore for fractured reservoirs, design waterfloods, and plan horizontal wells.

Oriented cores are typically cut using a conventional core barrel fitted with a special scribe shoe, and a device for recording the orientation of the primary scribe knife relative to magnetic north. Laboratory methods used to orient cores are correlation of the core with borehole imaging logs and the paleomagnetic method. Table 1-3 lists methods commonly used to orient cores.

Table 1-3—Core Orientation Methods

Method	Location	Comments
Multishot survey	Well	Must stop drilling to take reading.
Electronic survey	Well	Records orientation versus time.
Paleomagnetic method	Laboratory	Orients one continuous interval.
Log correlations	Laboratory	Requires correlatable features in core and wellbore.

1.7 CORING BITS

1.7.1 General

Coring bits are a basic part of the coring system. Unfortunately for the experts and novices alike, coring bits come in a bewildering array of styles. Fortunately, general bit/formation guidelines are available from manufacturers to aid in selection of the proper bit. With a little background information, it is possible to make informed decisions on cutter types, bit profile, and hydraulic considerations for the range of anticipated coring conditions. Final bit selection should be guided by the goals of the coring program, coupled with a confirmation that the bit has proven itself in the field for similar applications.

The hardness (compressive strength), abrasivity, and variability of the rocks to be cored will have the greatest influence on cutter selection. General guidelines suggest use of smaller, more impact-resistant cutters as the formations get harder.

Low-invasion, face-discharge core bits designed for unconsolidated-to-medium strength formations can be used in harder or more abrasive rocks, but bit life may be drastically reduced.

The information presented in Table 1-4 provides an overview of the types of coring bits that are available. Specific details on coring bits and recommendations for particular applications should be obtained from service companies.

1.7.2 Natural Diamond Bits

Natural diamond core bits are used when the formation is too hard (high compressive strength) and/or abrasive for other type cutting elements. Large natural diamonds can be surface-set in a tungsten carbide matrix, or fine diamond chips can be dispersed in matrix to form what is called an impregnated diamond bit. Impregnated natural diamond bits are for ultra-hard formation applications.

1.7.3 Polycrystalline Diamond Compact—PDC

Polycrystalline diamond compact (PDC) cutters are man-made diamond materials that consist of a layer of micron sized diamond grit sintered together and bonded to tungsten carbide studs. The thickness of the polycrystalline diamond layer is only 0.020 to 0.060 inches (.51 to 1.52 millimeters). PDC bits are used to efficiently core formations ranging from

Table 1-4—General Coring Bit Guide

Rock Properties	Rock Type	Core Bit
Ultra-hard, abrasive rock	Quartzite, Igneous Rocks	Impregnated natural diamond.
Hard, abrasive rock	Sandstone, Shale, Siltstone	Natural diamonds surface set or TSP cutters.
Hard, non-abrasive rock	Limestone, Dolomite, Anhydrite	TSP cutters.
Medium to hard rock with abrasive layers	Sandstone, Limestone, Shale	TSP or surface set natural diamonds.
Soft to medium strength rock	Sandstone, Chalk, Shale	PDC cutters, low fluid invasion design.
Soft rocks, no sticky layers	Salt, Anhydrite, Shale	PDC or roller cone cutters.
Soft, sticky rock	Gumbo Clay	PDC cutters, face discharge.

very soft to medium hard. The bits are designed to cut by shearing resulting in a rapid rate of penetration. Due to the geometry of the PDC cutter, they are susceptible to impact damage, and therefore, are not recommended for very hard, highly fractured, or cherty formations.

1.7.4 Thermally Stable Diamond—TSP

Thermally stable (diamond) product, TSP, is similar to PDC in that it is also a man-made diamond material. The main difference in the TSP material is that it has a higher range of thermal stability due to the leaching of the metal catalyst used in the sintering process of manufacture. These cutters are suitable for formations generally considered too hard and/or abrasive for PDC cutters. They are not recommended for soft formations.

1.7.5 Roller Cone

The roller cone core bit uses four rotating cones set with tungsten carbide inserts or hard-faced milled-tooth cutters for coring purposes. The cutters in the cones roll and impact the hole bottom and fail the formation in compression by a chipping action. Due to the slow cutting action (chipping-compressive failure) and the number of moving parts, roller cone core bits are not commonly used.

1.8 FLUID DISCHARGE CHARACTERISTIC OF CORE BITS

1.8.1 Throat Discharge

Throat discharge core bits are designed to have 100 percent of the fluid pass between the core shoe and the inside diameter of the core bit (the “throat”). Throat discharge bits are designed to clean the inside diameter of the core bit, removing cuttings from this area to ensure a very smooth entry of the core into the core barrel. The cleaning action reduces the tendency of hard and/or brittle formations to jam.

1.8.2 Face Discharge

Face discharge core bits are designed to divert some fluid that would normally pass through the throat of the bit to the face of the bit. This cleans the face of the bit and reduces the amount of fluid scouring the core as it enters the core barrel. Face discharge bits are recommended for use in soft and friable formations.

1.8.3 Low-Invasion Profile

The low-invasion profile coring bits are designed to maximize penetration rate, and minimize drilling fluid filtrate invasion into the core. The design incorporates face discharge ports, a reduced number of cutters, and a diminished clearance between the inner core barrel and the bit face. Use of low-invasion profile core bits is recommended for soft to medium strength formations. Harder formations would slow the rate of penetration and possibly damage the cutters.

1.9 CORE CATCHERS

1.9.1 General

The single most critical part of every coring system is the core catcher that holds the core in the barrel as it is brought to the surface. Table 1-5 lists the core catchers available and suggests those most appropriate for specific rock types.

Many situations call for combining two or more catchers to ensure success. Sequences of friable sand interbedded with shale might require both slip and flapper type catchers. Full-closure catchers, run primarily to ensure success when coring unconsolidated sand, also incorporate split-ring or slip-type catchers to improve core recovery in case coring ends in hard rock.

Table 1-5—Core Catchers

Type	Recommended Usage
Split-ring, spring	Consolidated formations.
Collet	Where formation characteristics are unknown.
Slip	Consolidated formations, normally run with flapper catcher or with orientation knives.
Dog or flapper	Consolidated, fractured, and unconsolidated formations where geology is unknown.
Basket	Unconsolidated formations, normally run with another core catcher type.
Full closure	Friable to unconsolidated formations to provide positive full closure.

SECTION 2—WELLSITE CORE HANDLING PROCEDURES AND PRESERVATION

CONTENTS

Page

2 WELLSITE CORE HANDLING PROCEDURES AND PRESERVATION 2-1
2.1 General 2-1
2.2 Core Handling Procedures 2-2
2.3 Field Sampling and Analysis 2-6
2.4 Rock Types and Special Considerations in Handling 2-7
2.5 Preservation of Cores for Analysis 2-12
2.6 Recommendations for Core Handling to Preserve Wettability 2-15
2.7 Precautions 2-15
2.8 Reading List 2-16

Figures
2-1 Core Marking 2-3
2-2 Core Analysis Wellsite Data. 2-8
2-3 Basic Core Analysis Laboratory Data 2-9

2 Wellsite Core Handling Procedures and Preservation

2.1 GENERAL

2.1.1 The recommendations included in this document may involve the use of hazardous materials, operations, and equipment. This document does not address all of the safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2.1.2 Wellsite core handling procedures and preservation should follow the best possible practices because the value of all core analysis is limited by this initial operation. The objectives of a core handling program are as follows:

a. Obtain rock material that is representative of the formation.
b. Minimize physical alteration of the rock material during core handling and storage.

The major problems confronting those handling and preserving reservoir rocks for core analysis are as follows:

a. Selection of a nonreactive preservation material and a method to prevent fluid loss or the adsorption of contaminants.
b. Application of appropriate core handling and preservation methods based upon rock type, degree of consolidation, and fluid type.

Different rock types may require additional precautions in obtaining representative core data (see 2.4). All core material should be preserved at the wellsite as soon as possible after retrieval to minimize exposure to atmospheric conditions.

2.1.3 The terminology that has evolved to describe the state of core preservation is important historically, but may be confusing because it is sometimes not used consistently. For example, the term “native state” has been often used to designate core drilled with oil-based mud or lease crude for the purpose of making accurate water saturation measurements. Similarly, “fresh state” has often been used to imply that core was drilled with bland, water-based drilling fluid and preserved at the wellsite to limit evaporative losses. This term has also been used to include cores cut with oil-based mud. In the interests of consistency, the following terminology is recommended:

2.1.3.1 fresh core: Any newly recovered core material preserved as quickly as possible at the wellsite to prevent evaporative losses and exposure to oxygen. The fluid type used for coring should be noted, e.g., fresh state (oil-based drilling fluid), fresh state (water-based drilling fluid).

2.1.3.2 preserved core: Similar to fresh core, but some period of storage is implied. Preserved core is protected from alteration by any of a number of techniques (see 2.5).

2.1.3.3 cleaned core: Core from which the fluids have been removed by solvents. The cleaning process (sequence of solvents, temperature, etc.) should be specified.

2.1.3.4 restored-state core: Core that has been cleaned, then reexposed to reservoir fluids with the intention of reestablishing the reservoir wettability condition. This is often the only alternative available, but there is no guarantee that reservoir wettability is restored. The conditions of exposure to the crude oil, especially initial water saturation, temperature and time, can all affect the ultimate wettability.

2.1.3.5 pressure-retained core: material that has been kept, so far as possible, at the pressure of the reservoir in order to avoid change in the fluid saturations during the recovery process.

None of these terms alone adequately describes the state of the core; a full description of drilling mud, handling, preservation, and subsequent treatment is required.

2.1.4 For testing, the core must be sampled. In order to obtain a representative core analysis from the formation(s) of interest, it is recommended that the entire core be sampled. The entire core section should be retained. Wellsite core sampling can be important for a variety of reasons (see 2.3.1). If sampling of the core is required, it should be performed with an awareness that the sampling procedure may impact future core analysis efforts and results. Wellsite sampling must be minimal to maintain the integrity of the core. Samples for lithology description, for example, can be taken from small broken pieces of core without damaging any of the intact rock. If intact, measurable lengths of core are removed, a note or block should be left in their place describing the sample length, lithology, the reason for removal, and any other pertinent information. If samples are required from within an intact core segment, a non-percussive sampling method should be employed. The objective of a standard core-sampling procedure is to obtain samples under a uniform procedure so that the results will be independent of human bias. The selection of samples is fairly simple for uniform formations. However, where a formation contains widely varying lithology and heterogeneous porosity types (such as conglomerates, cherts, vugular or fractured reservoirs, and interlaminated shales and sands), the proper selection of representative samples requires greater care. A qualified person (engineer, geologist, etc.) should follow an established sampling procedure to minimize statistical bias.

2.1.5 The prescribed core handling and preservation procedures are applicable to all conventionally cored rock material. Many of the same practices apply to sidewall cores and drill cuttings. These recommended procedures have been selected as those which will yield core materials for the most reliable and representative core analysis. The success of any given technique is directly related to the rock properties of the core. Handling procedures should also be based on the technology used to retrieve the rock material and the objectives of the coring program. A review of core preservation materials is also presented. Each coring job and reservoir should be carefully examined prior to the design of a wellsite handling and preservation program.

2.2 CORE HANDLING PROCEDURES

2.2.1 General

Several methods are available for core acquisition. Conventional, full-diameter, continuous coring techniques can be divided into two groups; those that employ a standard reusable steel inner barrel, and those that utilize disposable inner barrels or liners. Other coring methods such as the sidewall devices and wireline-retrieved coring apparatus obtain rock material using special equipment. Special coring processes, including pressure-retained and sponge methods, are available to obtain core and fluid analysis results more representative of in situ conditions.

Consolidated core material obtained with a standard reusable inner barrel should be removed from the barrel as soon as possible after reaching the surface to minimize drilling fluid imbibition. Among the possible undesirable effects of fluid imbibition are as follows:

a. Changes in fluid saturations, geochemical and gas solution equilibrium.

b. Changes in wettability.

c. Mobilization of interstitial clays and fine-grained minerals.

d. Clay swelling and associated degradation of mechanical properties.

Any delay in removal of the core from the barrel should be reported. Various rock types and coring methods require varying levels of attention and can be broken into two major categories:

a. Basic handling—This category requires minimum training and/or experience and includes:

1. Standard reusable steel inner barrel used to obtain core on fairly homogeneous consolidated rock.

2. Wireline sidewall core acquisition from percussion or rotary coring.

b. Special handling—This category requires extensive training and/or equipment and includes:

1. Disposable inner barrels and oriented core barrels used to obtain core from fractured or unconsolidated rock that may require mechanical stabilization (Skopec, et al., 1992).

2. Pressure-retained core barrel to maintain core at reservoir pressure to minimize fluid expansion from pressure reduction and fluid expulsion as the core is brought to the surface (Sattler, et al., 1988).

3. Aluminum core barrel with sponge liner inside a standard steel inner barrel to trap fluids during expansion from pressure reduction as the core is brought to the surface (Park, 1983).

The use of any core inner barrel liner reduces the diameter of the resultant core.

2.2.2 Removal of the Core From a Standard Reusable Steel Inner Barrel

The core should be removed from the inner core barrel in a horizontal position whenever possible. Care should be exercised to minimize the mechanical shock during extraction. The core should be allowed to slide out of the core barrel by slightly elevating the top end of the core barrel. If the core will not slide a rod may be used to push the core from the barrel. It may be necessary to gently tap the core barrel with a hammer to initiate movement of the core. However, do not hammer the core barrel in a manner that imparts severe mechanical shock to the core. In all physical manipulations, attempt to expose the core to the minimum mechanical stress. If the core cannot be removed by the foregoing method, it should be pumped out of the barrel with a fluid. If this is necessary, a suitable piston arrangement should be used that will prevent fluids from directly contacting and contaminating the core. The coring fluid should be used if pumping directly with fluids is necessary. The use of fresh water or other fluids foreign to the core should be avoided. If water is forced past the piston and contacts the core, erroneously high water saturation values may be obtained in subsequent core analysis as any excessive pressuring of the barrel may cause fluid to penetrate the core. Any difficulty or irregularity encountered while removing the core from the barrel should be noted, e.g., pressure used if pumped out with fluid, loss of core material, etc.

2.2.3 Labeling and Logging of the Core

The core should be laid out and boxed on the rig floor if space is available. Alternatively, the pipe rack can be used for this purpose. The labeling and logging of the core should not interfere with the drilling and/or coring operation. If the decision is made to handle the core on the rig floor, place appropriately marked trays, boxes, or troughs near the core barrel. If the core is to be laid out on the catwalk, prepare a cleared area and place the core between two lengths of drill pipe.

Care must be taken to maintain the orientation, and to preserve the correct sequence of the core pieces. The key point is

that the core must be labeled and marked in such a way that the entire cored interval can be reassembled at some future time. The core should be protected from temperature extremes, moisture, and dehydration, i.e., direct sunlight, hot engines, rain, strong wind, and low relative humidity. Core preservation materials and equipment should be close to the core handling area to facilitate a rapid operation. Accurate measurements of recovery must be made and recorded. Any recovery in excess of the core cut should be reported, as well as nonrecovery. Assign nonrecovery and overrecovery to the bottom of each core, unless some special observation indicates that an exception should be made. All such exceptions should be noted. The following data and observations may be helpful in determining the origin of overrecovery and nonrecovery:

a. Drilling parameters—drilling time, torque, rate of penetration, pump pressure, etc.
b. General conditions of the core—continuity, broken sections, induced fractures, etc.
c. Condition of the bottomhole coring assembly.

Label the core depths from top to bottom and assign overrecovery and underrecovery to the bottom. The top of the next core should be given the drilled depth. This means that in the case of overrecovery there will be the same depth in two cores. However, these cores will be distinguishable from each other because of their core numbers. Core depths must be adjusted to log depths before correlations can be made between log properties and core properties and between cored and uncored wells. Core-to-log adjustment can be made using detailed core descriptions or core scans.

It is recommended that all core barrels be laid down on the catwalk or rig floor before removal of core. The following guidelines are appropriate for laying out and marking the core:

a. The bottom of the core comes out of the barrel first and the first piece of core should be placed at the bottom of tray, box, or trough, with each succeeding piece being placed closer to the top.

b. Caution must be exercised in maintaining the proper sequence and core orientation to ensure that individual core segments are not out of place or turned upside down. Any portion of the core that is badly broken should be put in thick plastic bags and placed in its proper position.

c. Fit the core together so that the irregular ends match, then measure total recovery.

d. Do not wash the core (see 2.4, 3.5, and 3.6). If excess drilling fluid is on the core surface, it may be wiped off with a clean drilling fluid saturated cloth and wrung out as often as needed.

e. With red and black indelible markers, taped together, stripe the core from top to bottom with parallel lines (see Figure 2-1). The red line should be on the right as the individual performing the marking is facing from the bottom of the core towards the top. Arrows pointing toward the top of the core should be used to avoid confusion.

f. With an indelible marker or paint stick, starting from the top, draw a line across the core at each foot and label each line with appropriate depth.

g. To obtain reliable core analysis, speed is essential in removing, laying out, labeling, and preserving the core to minimize any alteration due to exposure (see 2.5).

h. The core should be preserved (see 2.5) and placed in numbered containers for transportation to the laboratory. It is recommended that the entire core interval be preserved at the wellsite, with sampling being reserved for the controlled conditions at the laboratory.

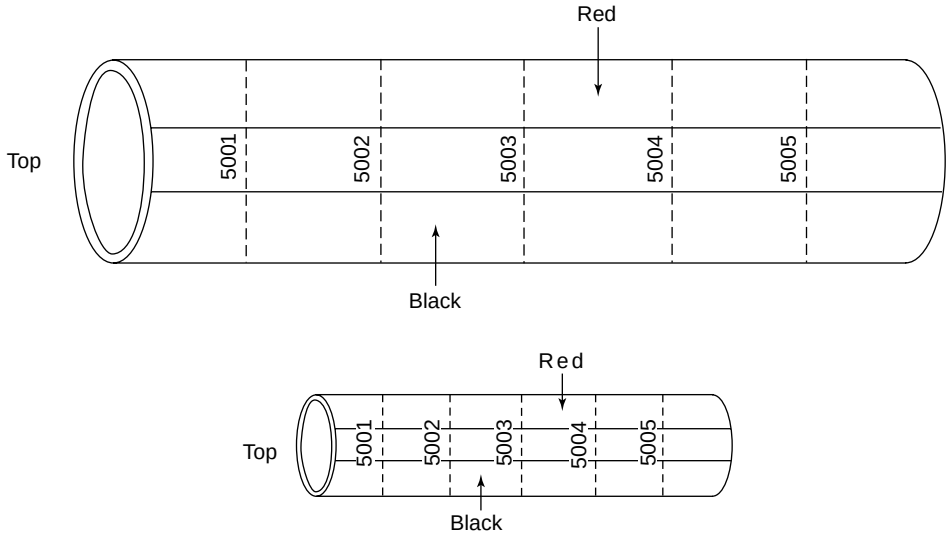


Figure 2-1—Core Marking

Even a few minutes exposure, depending on atmospheric conditions, can cause a significant loss of both water and light hydrocarbon fractions from cores. If the core is accidentally washed with water, allowed to remain in the core barrel, or let stand before preservation, then this information should be noted.

2.2.4 Handling Liners and Disposable Inner Barrels

Inner core barrel liners and disposable inner barrels improve the recovery of poorly consolidated or fractured formations. They are made of plastic, fiberglass, or aluminum and are rated to various temperatures. When coring unconsolidated or poorly consolidated formations, select the liner or disposable barrel to withstand the circulating temperature. Hard beds such as shale are best cored using either fiberglass or aluminum to prevent jamming and consequently poor core recovery. Certain coring fluid additives such as caustic react with aluminum barrels causing release of aluminum ions, that may react with the core to alter its surface properties.

When coring a poorly consolidated formation, to avoid rock compaction it is advisable to cut short lengths, 30 feet or less depending upon the rock strength. In long lengths, the lower section of core may be over compacted and damaged by the weight of the overlying material. Damaged core is of limited use for core analysis. When coring fractured formations, short core lengths may also be beneficial to decrease the risk of jamming.

The core barrel should be brought to the surface smoothly. During the last five hundred feet the core should be surfaced slowly to minimize gas expansion that can severely damage unconsolidated core if the pressure is reduced too quickly. Where gas expansion damage is expected, a perforated liner or a perforated disposable inner barrel can be used to provide a means for gas escape. All perforations must be sealed if the liner or disposable inner barrel is used as a core preservation container. Alternatively, the entire perforated section can be placed in plastic bags to prevent fluid loss.

A core-filled liner can be lowered to the catwalk, within the metal inner core barrel, using a system of pulleys anchored to the end of the catwalk. The barrel should not be banged on the rig and should be lowered gently onto the catwalk. Core-filled inner barrels flex, particularly those made of fiberglass or plastic, and should be supported by a splint. The splint should be attached to the inner barrel while it is hanging in the derrick.

a. Wedge the core barrel to prevent rotation and remove the core catcher. Transfer the core catcher material to a suitable length of liner or disposable inner barrel. The core catcher material is generally too disturbed to be used for quantitative core analysis.

b. A catwalk extension can be used to remove the entire core-filled liner from the inner barrel without flexing.

1. If the entire liner is removed or if handling a disposable inner barrel, feel inside and locate the top of the core. Cut the liner at this point. Label the core with orientation lines (red on the right and black on the left), depths, and other identification (see 2.2.3). Label depths every foot starting from the top.

2. (Preferred Method) If there is no room to remove the entire liner, pull out a little over 3 feet (0.91 meter) at a time using adequate support to prevent it from flexing. Label each 3-foot (0.91-meter) length with orientation lines and with a number to represent its position in the sequence of cut lengths. Label depths on the 3-foot (0.91-meter) lengths once the entire recovered interval is processed and the top of the core is located.

c. Cut the liners and core into 3-foot (0.91-meter) lengths using an air or electric powered circular saw. Fiberglass and aluminum liners should be cut using a mounted circular saw. Be careful to avoid vibration and rotation of the core. Clamps on the core barrel should be used with caution to avoid damaging the rock. Alternatively, the entire 30-foot (9.1-meter) length of core can be capped and shipped with a splint attached to the liner or inner barrel to prevent it from flexing. The core can then be cut in the laboratory to any specified length. This minimizes the handling of the core at the wellsite; however, preservation, sampling, and shipping procedures become more difficult.

d. Physically stabilize the 3-foot (0.91-meter) lengths using a nonreactive casting material (e.g., epoxy) to fill the annular space between the core and liner. Alternatively, the annulus can be filled with a nonreactive fluid in order to prevent evaporation. As a minimum precaution, the 3-foot (0.91-meter) lengths should be sealed with standard end caps.

e. Transfer the lengths to labeled boxes and cushion them for the trip to the laboratory. Use screws to attach wood lids to wood core boxes to avoid core damage caused by hammering.

2.2.5 Pressure-Retained Core

Pressure retaining core barrels are designed to obtain the best possible in situ fluid saturation. This method of coring offers an alternative to the conventional core barrel that loses pressure upon its retrieval to the surface. To enable the fluid saturations to be measured in the laboratory, the core must go through extensive handling. The core barrel assembly is placed in a special core service unit and the drilling fluid is flushed from the annulus between the inner and outer barrel using a nonreactive fluid while maintaining proper back pressure on the entire system. The entire core barrel assembly is then placed in a freezer box filled with dry ice (see 2.5.2.2).

To this point in the handling of a pressure-retained core, the work should be performed by trained service company personnel to their specifications.

The following procedures performed on frozen cores should be supervised by the operating company:

a. Remove the pressure-retained inner barrel from the ice, place in a safety shroud, and cut to the desired lengths.
b. Place each section back on ice, as it is cut. Take care to ensure that sections are laid down in such a way that top and bottom and position in the core section can be clearly identified. (see 2.2.3).
c. Raise one end of the core section at a time and place core cap with label on each end, securing with a hose clamp (see 2.2.4).
d. The labels should be marked with the company's name, recovered pressure, legal location, depth range of core, and processed core depth.
e. Place processed core sections in insulated shipping boxes and pack with dry ice. Mark the box with core numbers and depths along with company, location, and shipping information. If insulated shipping boxes are in transit for more than 24 hours, additional dry ice may be required.

2.2.6 Sponge Core

The sponge coring assembly is designed to improve the measurement of reservoir fluid saturations. As the core is brought to the surface, fluid that would otherwise be lost by expulsion because of pressure reduction is trapped by an absorbent polyurethane sponge surrounding the core. The coring assembly consists of 6 pre-cut 5-foot (1.52-meter) lengths of aluminum liner run within a standard steel inner barrel. The sponge core is, for the most part, then handled according to procedures established in 2.2.4. In most cases, the liner must be pumped from the coring assembly. The pre-cut liner is stored and preserved in PVC shipping tubes filled with a suitable nonreactive fluid. The PVC shipping tube is sealed with one rigid and one rubber gas expansion end cap. For orientation purposes, each sponge liner section is beveled on one end.

Once the sponge core arrives at the laboratory, it is milled open and both the core and sponge are extracted of all reservoir fluids (see 4.3.4).

2.2.7 Wireline Sidewall Coring

Wireline sidewall cores are extracted from the formation by a variety of means. Percussion sidewall coring involves the use of an explosive charge that propels a hollow projectile into the formation. Because of the forces produced by the entry of the percussion core bullet into the formation, compaction, fracturing, and rearrangement of rock grains occur; great care must be exercised when handling this core material.

Alternatively, wireline sidewall cores may be mechanically drilled from the formation with a rotary bit. Damage is minimized with this technique; however, this method is not feasible in all types of rock. If the sample breaks during removal from the coring tool, it should be pieced together and any damage should be noted. Other sidewall sampling devices involving the use of pressure-actuated coring receptacle samplers are also available.

When using sidewall coring techniques, fragile samples should be placed in plastic or glass jars with metal lids. Paper or other materials capable of absorbing liquids should not be placed in jars to act as sample cushioning material. Heat-sealable plastic laminates are an acceptable preservation technique for the drilled sidewall samples. All samples should be stabilized and cushioned during transport to the laboratory (see 2.5.2.1) and be accurately labeled (see 2.2.3).

2.2.8 Wireline-Retrieved Continuous Coring

In wireline-retrieved continuous (WRC) coring operations, the core barrel is recovered while the drill string remains in the borehole. Trip time is reduced and consequently the method can be less expensive than conventional coring. Typically, long vertical intervals are cored continuously, and certain wells may be cored from surface to total depth.

2.2.8.1 Depth Marking of WRC Cores

Good communication between the wellsite core retrieval personnel and the driller is necessary for accurate depth marking of WRC core. In conventional coring, the driller provides the top and bottom depths of the cored interval. This may cause some confusion in assigning core depths, because conventional cores are referenced from the top of the cored interval. Because WRC coring is continuous, it is best to use the bottom of the previous coring run as the top of the subsequent run. Monitoring for possible fillup between runs is suggested to minimize errors from this cause. Maintaining sound core accounting procedures in the form of a realtime spreadsheet should minimize the potential for errors in determining the top of the next interval. The spreadsheet information should include core run number, top of cored interval, bottom of cored interval, percent recovery, and a column noting the point from which core marking started.

All WRC core should be marked and labeled in accordance with 2.2.3, except assignment of depth should be modified in accordance with the following procedures. The conventional procedures for assigning core depths are not always appropriate for WRC core. In WRC coring, core dropped in one coring run may be retrieved in the next coring run, so the true depth of the current core may be in the previously cored interval. The retrieval of "dropped core" also means that the volume of overrecovered core is larger than is commonly encountered in conventional coring. Given the larger volumes of core involved, it is more important to use a core marking procedure that avoids assigning the same depth to more than one piece of core. If overrecovery occurs, the core depth marking can start with the bottom of the core, with the depth of that piece of the core being assigned the bottom reported

by the driller. Core marking should then move from bottom to top. If 100 percent or greater recovery was also obtained in the previous coring run, depth assignments should follow conventional procedures, with the depth marking beginning at the top.

For underrecovery, the following equation should be used to account for missing core:

$$\begin{aligned} \text{Missing core} &= \text{Depth Drilled} - \text{Core Length} \\ &= [CDD - PDD] - [CBL + PC - VOID] \end{aligned} \quad (1)$$

Where:

MC = missing core length, feet (meters).
CDD = current driller's depth, feet (meters).
PDD = previous driller's depth, feet (meters).
CBL = core barrel length, feet (meters).
PC = protruding core, feet (meters).
$VOID$ = void in core barrel, feet (meters).

In underrecovery, the core depths should be labeled from top to bottom, assigning underrecovery to the bottom of the cored interval.

2.2.8.2 Alternate Method for Depth Marking of WRC Cores

An alternate to the method discussed in 2.2.8.1 is to assign depths in the same manner as conventional cores (see 2.2.3). As for conventional cores, there may be underrecovery or overrecovery. Starting at the top, mark the core with depth marks every foot through to the end of the core. No attempt should be made to resolve underrecovery or overrecovery intervals before core-to-log adjustment. If depths are consistently marked from the top of each core run and each core run has a unique number, there may be the same "depth" in two consecutive cores, but they will be distinguished by their core run number. If an attempt is made to adjust for overrecovery at the wellsite, this can result in tremendous confusion with cores relabeled multiple times.

2.2.8.3 Washing of WRC Cores

As for conventional coring, care should be exercised to avoid damaging the rock; e.g., by washing it with inappropriate fluid(s). If there is any ambiguity about damaging effects, err on the side of caution, for example, by avoiding washing the core.

2.2.8.4 Analysis of WRC Cores

The extent to which the WRC core is analyzed varies from operator to operator. The core analysis procedures for WRC cores differ significantly from those used for conventional cores. In conventional coring operations, the core is returned to a laboratory for analysis and, at some future time, the core analysis results are adjusted to downhole log depths. With WRC core, much of the analysis is performed in the field, and in some cases, the core may never be archived. Typically, a wellsite geologist should describe the section with sufficient lithological detail to enable core-to-log adjustment and correlation. The geologist should note the hydrocarbon shows, porous intervals, and facies changes. Some operators use mobile laboratories through which they perform fairly sophisticated analyses of the WRC core, including core gamma scanning, ultraviolet photography, gas chromatography, and measurements of porosity, density, magnetic susceptibility, mineralogy, and acoustic properties.

2.2.9 Oriented Core Barrel

Orientation of the core barrel is accomplished using electronic, multi-shot instruments and specialized core scribing equipment. Alternatively, paleomagnetic rock signature can be used for core orientation purposes. Strict handling procedures must be followed to ensure that orientation data are positively depth correlated and matched to the proper piece of core material. This is particularly critical in fractured rock units where disposable inner barrels and liners are commonly used.

2.3 FIELD SAMPLING AND ANALYSIS

2.3.1 General

In general, sampling of recovered core material at the wellsite is not recommended. If it is necessary to sample immediately, precautions must be taken to minimize the exposure time of the core. Sampling should be quick, efficient, and performed in accordance with sound safety practices. Always obtain samples using the least damaging or contaminating method available. The entire core should be saved in all cases.

Specific reasons for wellsite sampling include, but are not limited to, a) chip sampling for lithologic description and/or mineralogical determination, b) measurement of basic rock properties, c) fluid compatibility-completion testing, d) wettability studies, e) observation of oil fluorescence/cut, f) anelastic strain recovery measurements, and g) methane desorption studies for the analysis of coal. All sections removed from the cored interval should be recorded on the field data sheet (see 2.3.3) and physically represented in continuous core sections using rigid spacers. The removed sample should be preserved, labeled, and packaged in a manner consistent with the test desired. Any additional pertinent data should accompany the samples to the laboratory or be available for wellsite analysis. Other special wellsite core analysis techniques are feasible if adequate mobile facilities are available to run tests under controlled conditions.

Hammering can damage the core and may preclude core analysis. If possible, chip samples should be taken at naturally occurring breaks in the core or with a precision trim saw. The sample size should be kept to the minimum necessary to perform the desired analysis. Place samples in individual

bags and preserve the fluid saturation with a suitable preservation technique (see 2.5).

If a detailed core examination is to be made at the wellsite, sampling and core handling should be done quickly, and only if the examination is critical to the success of the well objective.

2.3.2 Transportation and Logistics

The transport method should be expedient and provide protection against damage from environmental changes, mechanical vibration, and mishandling. Other important factors to consider when choosing the mode of transport include, a) distance and remoteness from the wellsite to the laboratory, b) onshore or offshore conditions and terrain, c) core material competence, d) weather conditions, e) type of preservation or packaging, and f) cost.

In all cases, precautions must be taken to securely stabilize the core material. In air transport, the storage cabin may not be pressurized, and this can be a factor in the core preservation. Do not stack core in such a manner as to damage the core material. Commercial carriers unaccustomed to transporting fragile materials should be used with caution. For safety reasons, cores packed in dry ice may have to be treated as a "chemical" for transportation purposes.

In all methods of transportation, a transmittal letter or contents documentation form with pertinent shipping information should accompany the shipment. A separate copy of this letter should be sent to the recipient via surface mail or fax. All applicable U. S. Department of Transportation regulations should be followed in the shipment of core materials. When standard core boxes are used they can be palletized, banded, and shipped as is. Cores, particularly those of unconsolidated materials, may be frozen or refrigerated at the wellsite for preservation and stabilization during transportation and storage. If freezing is used, the core must be entirely frozen before shipment to avoid mechanical damage. Frozen core is usually crated in insulated containers and packed with dry ice. Refrigerated core is usually shipped in self-contained refrigerator units. A temperature monitoring and recording device should accompany the core to ensure the desired conditions are maintained during transportation (see 2.5.2.2).

2.3.3 Data Sheet

A suitable data sheet should be provided for and completed by the wellsite engineer or geologist, to supply as complete a record as possible of the conditions of coring. This information will be valuable in qualifying the interpretation of the core analysis data. Further, this record may suggest either that certain additional tests be run to supplement the basic tests, or that other tests would not yield significant data. This will result in the most useful analysis for the least time and cost. Figures 2-2 and 2-3 are example forms, and the use of these or similar forms is recommended.

It is important to have as much pertinent data as possible accompany the core material. The following is a list of desirable information:

a. Well identification, API well number, elevation, vendor names and contacts, as well as telephone/telefax numbers and addresses.
b. Drilling fluid type, contents, and measured data.
c. Core type and equipment used.
d. The formation(s) cored, with the top and bottom driller's depth.
e. Designation of critical coring information and any pertinent coring notes, i.e., total coring/trip time, difficulties, and recovery.
f. Formation water salinity and production fluid data.
g. Preservation guidelines. Exposure time.
h. Analysis requested.
i. Coring log and drilling records.
j. A core description.
k. Well logs and mud logs (if available).

2.4 ROCK TYPES AND SPECIAL CONSIDERATIONS IN HANDLING

2.4.1 General

The term "rock type" is used to describe the major distinguishing feature(s) of core material. This can refer to the degree of consolidation, presence of fractures or vugs, composition (shale), or physical properties (e.g., low permeability) of the rock. Geological descriptions of rock are more complex and classification schemes have been devised to categorize specific rock types with respect to texture, type of cementation, grain size, etc. Many special considerations must be taken into account when designing a wellsite core handling program. Paragraphs 2.4.2 through 2.4.13 include general guidelines for various rock types.

2.4.2 Consolidated Rock

Consolidated rocks are hardened as a result of cementation. They need no special treatment at the wellsite. Cementation in rocks is defined as the process of precipitation of cementing materials around the solid grain surfaces. Rocks can be described as consolidated, poorly consolidated (friable), or unconsolidated, depending on the degree of compaction and cementation. Common consolidated rocks include limestone, dolomite, sandstone, and chert.

2.4.3 Unconsolidated Rock

Unconsolidated rocks have little or no cement and are essentially compacted sediments. Poorly consolidated rocks have minor cement but not enough to make them hard. These rocks are best cored using an inner core barrel liner or a disposable inner barrel (see 2.2.4). Care must be taken to prevent

Prepared By: _____ *Date:* M M D D Y Y Core Number _____

Operator _____ Coring Company_____ Core Analysis Company_____

Contact/Phone _____ Contact/Phone _____

Well Identification _____ Field/Reservoir _____ Address _____

API Number _____ ❑ Vertical Well ❑ DeviatedWell ❑ Sidetrack _____

County/State/Country _____ Legal Location/Block_____ Elevations: _____ Ground/Mean Sea Level _____ Kelly Bushing ____

Persons Requesting Work _____ Phone _____ Fax _____ Address _____

Report Results To _____ Phone _____ Fax _____ Address _____

Alternate _____ Phone _____ Fax _____ Address _____

Rig Contract _____ Phone _____ Fax _____ Address _____

Coring/Drilling Fluid
Type/Contents _____ Weight _____ ppg Funnel Viscosity _____ sec Water Loss _____ cm^3/30 min Chlorides _____ ppm pH _____ Tracer _____

Type of Core
❑ Conventional _____ Inner Barrel Type _____
❑ Sponge ❑ Oriented Barrel
❑ Pressure Retained ❑ Other _____
Sidewall: ❑ *Percussion or* ❑ *Mechanically Drilled Number Attempted* _____ *Number Recovered* _____

Analysis Planned
❑ Plug Size or ❑ Full Diameter
Method
❑ Fluid Saturation _____
❑ Porosity _____
❑ Grain Density _____
❑ Permeability _____
❑ Surface Gamma Log _____
❑ Special Instructions _____

Transportation
❑ By Service Company _____
❑ Ground _____
❑ Air _____
❑ Other _____
Carrier _____
Date shipped _____

Preservation
Method ❑ Plastic Laminate/Type _____
❑ Freeze ❑ Dry Ice or ❑ Liquid Nitrogen
❑ Refrigerate _____ °F _____°C
❑ Core Inner Barrel
❑ Core Wrap and/or ❑ Dip-Type ❑ Resination
❑ Other

Exposure Time/Climate _____
Notes _____

Laboratory/Long Term Preservation _____
Special Instructions _____
Post Core Analysis Instructions/Core Material Distribution _____

Formation(s) Depths (Drillers)
_____ Interval Cut _____
Expected Core Point _____ Interval Recovered _____
_____ Interval Cut _____
Expected Core Point _____ Interval Recovered _____
_____ Interval Cut _____
Expected Core Point _____ Interval Recovered _____

Well Inclination _____ Core Diameter _____Units_____
Total Core Recovered_____ Bit Type _____
Length Cored _____Units _____ Percent Recovery _____
Coring Time _____ Trip Time _____
Estimated Connate Water Salinity _____ ppm Chlorides or Estimated R_W _____ @ _____ ❑ °F ❑ °C
Estimated Production ❑ Oil
❑ Condensate
❑ Dry Gas

Notes _____

Attach Coring log And Core Description

Figure 2-2—Core Analysis Wellsite Data

General Information
Analysis Requested Date [M][M][D][D][Y][Y] API # _____
File # _____

Requested By: _____

❑ Full Diameter or ❑ Plug : Size _____ Units _____

Method	*Special Instructions*
❑ Saturation	_____
❑ Porosity	_____
❑ Permeability	_____
❑ Other	_____

Objective of the tests as agreed by parties involved _____

_____

Transportation and Inventory

Carrier _____ Date Shipped _____ Core Arrival Date _____

Core condition on arrival:

❑ Preserved ❑ Frozen ❑ Unpreserved ❑ Cleaned ❑ Other _____

Correlation Depths: Driller's _____ Log _____ TVD _____ Units _____

Allocation of the missing cored interval: ❑ Bottom ❑ Middle ❑ Top _____

Screening

❑ CT ❑ X-ray ❑ NMR ❑ Fluroscopy ❑ Other _____

Sample Handling

Core Gramma (yes/no, type) _____

Lab Preservation (prior to analyses) _____

Sampling Method

Sample Sleeve ❑ Yes ❑ No, Type _____

Nominal Sample Diameter/Length_____ Units _____

Plugging/Trimming Fluid_____

Treatment prior to testing (flushing, evaluation, resaluration, etc.) _____

_____

Core Disposition (removed samples, storage location, preservation, etc.)_____

_____

Lab Analyst _____

Other Service

❑ Slab ❑ Resination ❑ Photo: ❑ BW ❑ Color ❑ U.V. ❑ Other _____

Supplementary Tests _____

_____

Methods and Conditions

Cleaning:

Methods	Conditions
❑ No Cleaning	Solvents _____
❑ Dean-Stark	Temperature _____Units _____
❑ Soxhlet	Pressure _____Units _____
❑ CO2/Solvent	Time _____
❑ Flow Through	Volume and Rate_____ Units _____
❑ Others	Others _____

Drying:

Methods	Conditions
❑ Convection oven	Temperature _____ Units _____
❑ Vacuum oven	Time _____
❑ Humidity oven	Relative Humidity % _____
❑ Others	

Porosity:

Methods

Pore Volume	Grain Volume	Bulk Volume
❑ Boyle's Law	❑ Boyle's Law	❑ Caliper
❑ Saturation	❑ Archimedes	❑ Archimedes
❑ Summation-Of-Fluids	❑ BV-GV	❑ Mercury Displacement
❑ BV-GV	❑ Other	❑ GV+PV
❑ Other		❑ Other

Conditions/Fluids

Confining Stress (Magnitude and Type)_____Units _____

Gas _____ Liquid_____

Pressure _____

Permeability:

Methods	Conditions
❑ Steady State	Fluid Type _____
❑ Unsteady State	Confining Stress _____Units _____
❑ Probe	Sleeve Durometer _____
❑ Empirical	Pore Pressure _____Units _____
❑ Not Measured	Klinkenberg: ❑ Measured ❑ Empirical ❑ No Correction
❑ Others	Inertial Factor: ❑ Measured ❑ Empirical ❑ No Correction

Saturation:

Methods	Conditions
❑ Distillation Extraction (DS)	Temperature _____Units _____
❑ High Temperature Retort	Fluids _____
❑ Others	Water Density_____ Units _____
	Oil Density _____ Units _____
Correction for Salt:	❑ Corrected for Water Volume
	❑ Corrected for Oil Weight ❑ Not Corrected

Quality Assurance: (Page number in report where the information is provided) _____

_____

Comments/Remarks: _____

Data Anomalies: _____

Figure 2-3—Basic Core Analysis Laboratory Data

the core from disintegrating. This includes making sure the core is brought to the surface, laid down smoothly, and preserved in such a way that it will survive transportation (see 2.5.2).

2.4.4 Unconsolidated Rock—Light Oil and Gas

It is critical to preserve unconsolidated cores containing light oil in an efficient and expedient manner. Any unnecessary movement of the core should be avoided. The two methods commonly used to preserve this rock type involve environmental methods, such as freezing or chilling and mechanical stabilization with epoxy, foam resin, etc. Unconsolidated core containing light oil is susceptible to significant fluid loss during handling at the surface. As with most rock types, by the time the core has been brought to the surface, it has undergone mechanical stress relief due to the removal of overburden pressure and has had varying amounts of gas expansion when internal pore pressure is lost. The degree to which these effects will alter the core depends on depth, reservoir pressure, oil gravity, fluid properties, sediment type, and coring procedures. Care must be taken to ensure that pressure does not build-up within the core barrel during handling. The inner core barrel liner or disposable inner barrel can be predrilled with holes [$^1/_8$ inch (3.18 millimeter) diameter] to avoid pressure buildup. Cored intervals should be limited in length to prevent possible rock damage under its own weight. As the core is raised through the upper 500 feet (152 meters) of the well, the core barrel should be retrieved at a slow rate to minimize chances of disaggregating the rock and causing damage to the core. If freezing is used to stabilize unconsolidated materials, the core should not be transported before it is fully frozen, as partial freezing can cause structural damage to the core (see 2.5.2.2).

Filling the annulus between the disposable inner barrel or liner and core with coring fluid is recommended for stabilization when epoxy or a permanent setting material is not used; however, this procedure can alter rock fluid saturation and wetting characteristics. When epoxy, resin, or foam injection are used, the drilling fluid must be fully displaced or drained from the annulus. The casting material must completely conform to and encase the core surface.

2.4.5 Unconsolidated Rock—Heavy Oil

The greatest difficulty in handling unconsolidated rocks that contain viscous heavy oil is prevention or minimization of delayed core expansion. The expansion is the result of slow gas evolution from the heavy oil, with no possibility of short-term drainage because of the low mobility to gas. Swelling of the rock may easily continue until the gas phase becomes continuous, and this may require volumetric expansion in excess of 6 percent to 8 percent. In liners, unconsolidated heavy oil sandstones will expand radially to fill empty annular space. Once the swollen core is tight in the liner, further gas evolution may cause a piston action, causing core to extrude from the ends of the liner sections leading to overrecoveries as high as 5 percent. It is tempting to simply cut off the extruded core and discard it, but this must be avoided. Extruded material is kept at the liner end from which it came, the plastic end caps may aid in keeping it slightly retained.

Improving core quality in unconsolidated heavy oil sandstones requires the following considerations:

a. Provide mechanical restraint to expansion.
b. Provide a means to allow gas drainage.
c. Provide mechanical strength to the core.

Item a involves use of liners that have an internal diameter only slightly larger [$^1/_8$ inch (3.18 millimeters)] than the core bit, to reduce radial expansion. During handling and preservation, flexing of liners, heating of core, and prolonged exposure of liner ends must be avoided. Axial restraint will help reduce the tendency to extrude the core. This restraint may be provided in a number of ways, including:

a. Rigid liner caps rather than rubber end caps, with the end caps stapled to the liner at a number of points, and secured with screw clamps.
b. Cutting liner segments to precise lengths and placing in a strong core box so that the ends of the box provide axial restraint. Alternatively, cores can be wedged into the core boxes with pieces of wood planks cut to lengths.
c. Special handling techniques devised by various operators may also be used, including special storage cylinders or axial retention methods.

Gas evolves slowly from heavy oil, and will continue for months. It is recommended that pre-perforated liners be used in all areas to shorten the gas flow path and eliminate piston effects. The pre-drilled holes [$^1/_8$ inch (3.18 millimeter) diameter] should be spaced no further apart than the radius of the liner. Post-drilling of liners is not recommended as an acceptable alternative because it prolongs core handling and may lead to damage. If required, liner and core segments may be placed in cylinders and repressured with an inert gas (N_2) to slow or stop gas evolution, and to avoid oxidation.

Freezing of unconsolidated heavy-oil core may be necessary, although in general, freezing is not well understood. Freezing has the following effects: a) it reduces gas evolution speed and volume, b) it enhances oil viscosity that restricts expansion, and c) it freezes interstitial water that gives the core some mechanical strength to restrict expansion and fracturing. Because the pore water is usually saline, the temperature will have to be reduced below –40°F (–40°C) to ensure full mechanical benefits (see 2.5.2.2).

Core handling during transportation and storage for unconsolidated materials containing heavy oil must maintain the mechanical restraint and low temperature. When preparing core sections for analysis, the temperature should be allowed to rise slowly, so that evolved gas can dissipate; mechanical

restraint should be fully maintained until the core is equilibrated, a process that may take weeks because of the high oil viscosity and low relative permeability.

2.4.6 Vuggy Carbonates

Large vugs can weaken the core material and cause difficulties with recovery. In many cases, core recovery is reduced in friable vuggy intervals. Standard consolidated core preservation methods should be used on this rock type (see 2.5).

2.4.7 Evaporites

Salt rocks are generally quite competent, and, except for their solubility, may be considered as consolidated rocks. Core containing salts in continuous sequences or as vug and fracture fillings should not be washed with fresh water under any circumstances. Because the physical properties of salt rocks can be altered by small changes in moisture content, cores containing salts must be immediately wiped to a surface dry state and preserved. Transportation and storage of cores containing salts must always be undertaken keeping the soluble nature of the material in mind. Cores of evaporite, anhydrite, gypsum, or calcite present no special core handling problems.

2.4.8 Fractured Rock

Many reservoir rocks are naturally fractured. Disposable inner barrels or liners made of aluminum or fiberglass are recommended for coring fractured rock (see 1.6 and 2.2.4). An oriented core can be useful in determining fracture strike and in situ stress direction (see 1.6 and 2.2.4).

2.4.9 Rocks Rich in Clay Minerals

Clay minerals may be present in small quantities in rocks, yet have a major impact on rock properties. Some of the major concerns in rocks containing clay minerals include:

a. The presence of smectite (a swelling clay mineral), even in very small quantities (1 percent), is of importance in core handling because of swelling potential, high cation exchange capacity, and osmotic suction potential.
b. Interstitial clay minerals can be physically mobilized by changes in fluid content, chemistry, or mechanical disturbance, leading to pore throat blocking or changes in surface wetness characteristics of pores or other physical changes.
c. Clay minerals in contact with their natural pore fluids are in thermodynamic equilibrium, and exposure to other fluids will alter this leading to changes in clay mineral activity, exchangeable cations, and consequent changes in mechanical and flow behavior.
d. Smectitic shales and sandstones may swell when confining stress is removed if free water is available, even if the free water has properties identical to the interstitial fluids.

Any excess fluid or mud cake should be immediately wiped from cores of smectitic materials and clay mineral rich materials, followed by immediate preservation (see 2.5.2).

2.4.10 Shale

In addition to the recommendations for clay mineral containing rocks (see 2.4.9), there are special issues relating to handling highly fissile shale. These materials have fissility planes of low strength that may split spontaneously, even if core is handled with great care. Once a fissile shale core has split, it may be impossible to obtain specimens large enough for core analysis.

It is recommended that fissile shale cores be handled in the following manner:

a. Avoid excessive handling or movement of the core.
b. Remove any excess water.
c. Preserve immediately to stop desiccation.
d. Masking tape or fiberglass packaging tape may be wrapped around core segments perpendicular to the fissility planes to reduce further splitting. Alternatively, heat-shrinkable plastics can be used.

Shales have low permeability and slow internal transfer of moisture will inevitably take place between beds of different mineralogy and fabric during long-term storage. If the core is totally unconstrained, this may result in delayed splitting even without desiccation or handling. Fissile shales are exceptionally sensitive to temperature changes, and should be maintained at a constant temperature during transportation and storage. Freezing of shales must not be allowed, since this leads to massive microfissuring and internal moisture movement.

Oil shales with organic chemical volumes in excess of 20 percent are sensitive to temperature and oxidation, and must be preserved with particular speed if detailed analysis is required. The much stronger matrix-supported oil shales are typically more quartzose and with organic chemical volumes less than 20 percent and in general do not require special handling.

2.4.11 Low Permeability Rock

Evaporation of fluids, a problem with all core materials, is a particular difficulty in low permeability and low porosity core where the percentage change in saturation may be much greater for the same volume of fluid evaporated. The time period before core is protected from evaporation is critical for these samples. The presence of clay minerals may make damage by evaporation irreversible in some samples (see 2.4.9).

2.4.12 Coal

In situ gas content, gas sorption behavior, permeability, relative permeability, cleat and fracture analysis, core composi-

tion, and mechanical behavior are the major interests in coal analysis for coalbed methane production. Gas desorption studies may be performed at the wellsite with desorption canisters. Procedures for handling of coal core should include instructions for these special studies. Wireline retrieved core barrels, core barrels with disposable inner barrels or liners, and pressure-retained core barrels have been used to cut coal cores.

Gas content and gas desorption rate are commonly measured from coal by canister desorption methods using conventional cores, mechanically-drilled sidewall cores, wireline-retrieved continuous cores, or drill cuttings. Coal segments are sealed in a canister and isothermally maintained while the volume of gas evolved from the sample is measured. Gas content measurement by canister desorption requires an estimate of gas volume lost while bringing the core to the surface and before sealing the samples in the canister. Gas content must be normalized to a mineral free matter basis. Since the gas evolved will not be 100 percent methane, the composition of the gas must be analyzed.

Pressure-retained core technology, that does not require an estimate of lost gas, offers a more accurate means to determine the total in situ gas content of a coalbed. The volume of gas evolved from the coal core in the pressure barrel is measured as a function of time, temperature, and pressure. If pressure-retained methods for coring coal are used, a special internal temperature sensor is incorporated, and no coring fluid flushing is carried out (see 2.2.5). In one procedure, the entire pressure-retained barrel is returned to bottomhole temperature and allowed to equilibrate for up to several days before the pressure is allowed to dissipate and the device is disassembled. During the temperature and pressure dissipation phase, changes should be gradual to minimize both the magnitude of the pressure gradient and the temperature gradient. The former may cause the coal to fracture internally, whereas rapid cooling may cause tensile cracks to open in the outer part of the coal core. These may affect both permeability and mechanical behavior. Alternatively, the pressure-retained coal core can be cut into one foot sections and placed in desorption canisters for measurement of the gas content and gas desorption rate.

Coal core must be handled with care at the wellsite because of its heterogeneous nature, and because the methane found in small pores within the core is under pressure. Careless treatment, liner flexing, or a sharp blow to the core barrel may cause the core to fragment, rendering the core of little use for core analysis. The internal gas pressure may aid this tendency to deteriorate, and time is required to permit gas dissipation. Coring fluids in contact with coal intended for fluid flow or mechanical tests should not contain materials capable of altering the coal structure.

Fresh coal oxidizes when exposed to air, potentially changing its surface properties and sorption characteristics. Exposure time to air must be minimized, and special handling may include canisters to receive coal core segments, followed by flushing of the cylinders with inert gas or methane.

White or yellow pens or paint crayons should be used to mark the surface of the core lengths. As with any core, preservation of moisture and minimization of exposure time is advised. Freezing of coal core is not recommended, nor is it considered necessary.

2.4.13 Diatomite

Diatomites are generally high-porosity, low-permeability rocks composed of opaline-quartz phases with varying amounts of detrital material. Diatomites are cored with disposable inner barrels or liners (see 2.2.4).

Diatomite can be preserved by environmental means, wrapping, etc. Freezing of diatomite is not recommended. Temperature should be controlled to maintain a constant temperature [35 to 40°F (1.67 to 4.44°C)] during wellsite and transportation operations.

2.5 PRESERVATION OF CORES FOR ANALYSIS

2.5.1 General

The preservation of a core is an attempt to maintain it, prior to analysis, in the same condition as existed upon its removal from the core barrel. In the process of cutting a core, recovering it, and bringing it to the surface, the fluid content of the rock is altered by unavoidable changes in pressure, temperature, etc. Pressure-retained core methods attempt to minimize these effects (see 2.2.5). Careless or incorrect practices in handling and preservation cause further alteration of the core and its fluids, thereby making the core even less representative of the formation.

Preservation and packaging of cores may vary depending upon the test(s) required, the length of time before testing, and the potential of performing wellsite tests. If the core samples are to be used to determine fluid saturation or for special core analysis, it is necessary that they be preserved for transportation to the laboratory. Evaporation and migration of fluids as well as oxidation within the sample must be avoided to obtain reliable core analysis. An additional objective of the preservation program is to prevent breakage of the cores during shipment and storage. Consolidated core may be durable enough not to require special handling procedures. However, special care should be taken with unconsolidated or fractured rock, etc. (see 2.4).

The use of unprotected glass jars, easily deformable plastics (if not properly stabilized), paper cartons, non-rigid containers, and air-tight cans are not recommended for core preservation purposes.

2.5.2 Methods of Preserving Cores

There is no one best preservation method. Experience can help determine the most satisfactory method for the rock type

in question. The choice of method will depend on the composition, degree of consolidation, and distinguishing features of the rock. Therefore, general use of one specific method of preservation will not apply to all rock types. The techniques required to preserve cores for testing may depend upon the length of time for transportation, storage, and the nature of the test to be performed. Some variation in the method of preservation may depend upon whether the cores will be analyzed locally or whether they must be prepared for long-distance shipping. Preferred methods to preserve cores for laboratory analysis include one or more of the following:

a. Mechanical stabilization.
b. Environmentally controlled preservation using chilling, regulated humidity, or freezing, if necessary (see 2.5.2.2).
c. Heat-sealable plastic laminates.
d. Plastic bags.
e. Dips and coatings.
f. Sealing in disposable inner barrels, liners, and tubes.
g. Anaerobic jars.

2.5.2.1 Mechanical Stabilization

All rock types should be mechanically stabilized prior to shipment to the laboratory. This is particularly true for unconsolidated rock (see 2.4.4 and 2.4.5). Core that has been cut using plastic, fiberglass, or aluminum liners/disposable inner barrels can be cast using resin, wax, or foam to fill the annular space between the core and the sleeve. Resin has low viscosity and will fill fine fractures. However, it is only poured into the annulus and is not under enough pressure to displace pore fluids in the rock and therefore does not impregnate the core.

Mechanical stabilization for well consolidated cores may also be as simple as wrapping the core in bubble wrap or other suitable cushioning materials. All core material should be considered fragile and handled carefully (see 2.3.2). Care should be taken to avoid disturbing poorly consolidated or fractured core prior to mechanical stabilization.

2.5.2.2 Environmental Preservation

Controlling environmental conditions to which the core is subjected by chilling or maintaining a humid environment can help to preserve the core (refer to core preparation information in Section 3). Core chilling is used primarily to minimize fluid evaporation and provide mechanical stabilization. This technique is useful in preventing the core from drying; however, its effectiveness is subject to the coring fluid type and the reservoir rock and fluid properties. When chilling core, it is still necessary to mechanically stabilize the rock for transport to the laboratory (see 2.5.2.1).

Cores that are preserved by freezing should be frozen by application of dry ice, liquid nitrogen, or placement in an electrically-operated freezer unit. Freezing may result in the migration or diffusion of fluids within the core structure or breakage of the core. Freezing can cause significant evaporative losses through sublimation. Unconsolidated cores that have been frozen can be packed with a $^1/_4$ inch (6.35 mm) thick layer of frozen brine (surface ice cake) to reduce the sublimation process. This measure is critical if freezing is used for long-term core storage. Structural damage to the core may occur if it dehydrates while frozen.

The practice of freezing core is most common for unconsolidated rocks (see 2.4.4 and 2.4.5). The full effect of freezing on a core's petrophysical properties is unknown. The freezing of consolidated core with interstitial water is not well understood. Expansion of ice crystals may cause irreversible structural damage to core. Freezing may affect the properties of rock flushed with fresh water more than those flushed with saline drilling fluid filtrate. These effects will decrease with decreasing water saturation. If it is necessary to allow the core to warm to ambient temperature before testing, the condensation of moisture from the atmosphere onto the core surface must be prevented. Thawing of the core may cause some redistribution of the fluids within the core matrix.

The fluid saturation and reservoir (mineral) properties can also be preserved by controlling the relative humidity of the core environment with specially designed ovens. This technique has wide applicability and is most effective with rocks containing moisture sensitive clay minerals and/or chemically bound water contained in minerals (see 2.4.9).

2.5.2.3 Heat-Sealable Plastic Laminates

Several heat-sealable plastic laminates are available. Aluminum foil or mylar may be used to add rigidity to the laminate. The laminated core preservation package should act as an impermeable barrier to water vapor and gases, and be resistant to chemical alteration and degradation by fluids. Laminates are easy to use and the preservation process can be performed quickly. Care should be taken to prevent tears or punctures in the laminate. A clean, flat surface is required for smoothing the laminate prior to sealing. All core should be prewrapped and taped with durable plastic or other material to cover the core ends and sharp edges. The packaged core segment should be labeled with well and depth information.

The heat sealing process is critical to the success of using this preservation method. The heat sealer must be set to the proper temperature in accordance with manufacturer's specifications to obtain an effective seal. Any discontinuity in the seal will negate the barrier properties of the material. Some laminates are available in tubular form that requires the sealing of only two ends rather than four. The head space in the preservation package should be minimized; however, enough material should be used to prevent weakening if the package will be opened and resealed more than once. In some cases, it may be advisable to evacuate the gas space where the loss of light-end hydrocarbons is not an issue. An inert gas such as nitrogen can also be blanketed over the rock to minimize oxi-

dation. When core material degasses, the laminate package will inflate; this will pose no problem if the package is sealed properly. If necessary, the evolved gas may be sampled directly with a standard gas syringe and the package resealed. The preservation package must be labeled and mechanically stabilized for shipping (see 2.5.2.1). At no time should the preservation package be subjected to extremes of temperature.

2.5.2.4 Plastic Bags

Plastic bags are only recommended for short-term preservation. Core samples should have a minimum of air space between the core and bag wall. Any excess bag can be folded against the core wall and taped to assure a tight fit. As always, clear labeling and proper stabilization procedures should be followed.

2.5.2.5 Dips and Coatings

Dips and coatings are used when cores are not to be tested within a few hours or days and when the material is to be transported over long distances. Dip coatings can also be used with plastic laminates to add mechanical integrity.

CAUTION: Cores should never be dipped directly into any molten wax or plastic material.

All core should be prewrapped with a heat-sealable laminate or plastic film and aluminum foil prior to dipping. All core segments should be labeled with well and depth information. The purpose of the plastic film wrap is to prevent contact of the core and pore fluids with the aluminum foil outerwrap. Such contact can cause oxidation of the foil and loss of its moisture and oxygen barrier properties. The following procedures should be used with the wrap and dip method:

a. Prepare a heating vat for dip-coating several hours prior to preserving the core. Observe all safety precautions. Follow the dip manufacturer's recommendations for handling. Overheating the dip can cause the coating to be ineffective.

b. Wrap the core tightly in plastic film that will conform to the surface of the rock, crimping the free ends together. Several layers of high quality plastic film are desirable to prevent puncturing.

c. Wrap the core with several layers of aluminum foil, crimping the free ends together. Avoid puncturing the aluminum wrap.

d. Tie a wire around the core to make a handle.

e. Dip the foil-wrapped core sample in the molten coating material. A liberal amount of dip coating should encase the core; a $^1/_8$- to $^1/_4$-inch (3.18- to 6.35-millimeter) thick coating is recommended. This is accomplished through the use of multiple dips, allowing each dip coating to harden prior to the application of additional dip material. It is recommended that the dip coating be hardened by suspending the core in air by means of the wire handle.

f. The wire handle should be cut flush with the dip coating. Additional dip should be applied to the wire end to eliminate a pathway for evaporation or oxidation.

Coating material must have certain properties, as follows:

a. It must be dimensionally stable over long periods of time.

b. It must not react with oil or water and not contain acids, oils, solvents or any other liquid that may be exuded when set.

c. Permeability to gases, oils, and water must be low when set.

d. It should have a low melting point, preferably below 200°F (93.3°C) maximum and have a fairly low viscosity when melted. Higher melting points are acceptable if exposure time to the dip is minimized.

e. When removed from heat and exposed to ambient conditions, it should be dry and set tack-free within 5 to 15 seconds.

f. When set, it should be tough but pliable, slightly elastic but with good tensile strength, and not melt at temperatures below 180°F (82.2°C).

As with all core preservation methods currently used, the long term effectiveness of dips and coatings remains uncertain.

2.5.2.6 Disposable Inner Barrels, Liners, and Rigid Tubes

A convenient means of core preservation is possible when disposable inner barrels or liners made of plastic, aluminum, or fiberglass are used (see 2.2.4). The core may be preserved as-is by sealing the ends of the cut inner barrel or liner. This is not recommended as a long term preservation method, but will allow the core to be processed quickly and without special equipment. Holes in the inner barrel should be sealed prior to shipping. The core can be rapidly shipped to the laboratory for sampling and testing, and, if required, additional preservation of the sealed containers is possible (see 2.5.2.2). When a conventional core barrel is used, steel, aluminum, or plastic tubes with suitable couplings, caps, or O-ring seals may be used to preserve the core. To be used effectively, the tube and end pieces must be nonreactive with the core and its fluids. Segments may be stabilized according to guidelines established in 2.5.2.1 to minimize damage in shipment.

2.5.2.7 Anaerobic Jar

Immersion of the core in liquid within an anaerobic jar can be used to prevent oxidation, evaporation, and drying during the handling of core. The anaerobic vessel is an elongated jar with a sealable lid, into which a liquid can be introduced and any free oxygen removed. The immersion liquid must be compatible with the core and pore fluids, and be able to main-

tain the current wettability of the sample (see 2.6). Typically, the following fluids are used for immersion:

a. Deoxygenated formation brine or synthetic formation brine with biocide.
b. Crude oil.
c. Depolarized refined mineral oil.

As always, follow all safety precautions when using anaerobic jars for preserving reservoir samples.

2.6 RECOMMENDATIONS FOR CORE HANDLING TO PRESERVE WETTABILITY

2.6.1 General

Wettability alteration can take place during coring, core treatment at the wellsite, or during the period of storage before measurements are made in the laboratory. Only core treatment at the wellsite is discussed here, although the measures recommended can only be successful in preserving wettability if adequate precautions are taken at each step of the core recovery and analysis process.

The validity of many laboratory core tests depends on maintenance or reestablishment of reservoir wetting conditions; however, there is rarely an initial reference point to define in situ wettability. The practices recommended here are intended to provide measures of wettability as early as possible after removal of core from the formation so that a reference point for wettability is established. Cores should not be exposed to air for any longer than is necessary.

Wettability tests should be made at the wellsite. A simple observation of imbibition of water and oil droplets placed on the core surface should be recorded routinely. In many cases, this will be the extent of wellsite wettability testing, but much more comprehensive tests can be initiated at the wellsite as well. Some special facilities would be required, including:

a. The capability to cut core plugs from newly recovered whole core. Any delay in cutting core plugs allows capillarity and diffusion to distribute surface active drilling fluid constituents to the uninvaded portions of the core. Synthetic reservoir brine or refined laboratory oil should be used as the cutting fluid.
b. Core holders and core flooding equipment to allow flushing of plugs are needed. It may be possible to avoid damage done by an unstable oil phase (one from which asphaltenes or paraffins are deposited because of changes in pressure and temperature) if the oil phase is quickly replaced by a stable, non-damaging oil.
c. Imbibition test facilities are also needed since tests of imbibition rate and extent are the best indicators of wettability in porous media.

Requirements for maintaining wettability will vary from one reservoir to another and to some extent will have to be experimentally determined. Suitable core preservation methods to control fluid loss are described in 2.5.2.

2.7 PRECAUTIONS

2.7.1 General

The fundamental objective of core analysis is to obtain data representative of in situ reservoir rock properties. Coring, handling, and preservation should be conducted in such a manner as to prevent both loss of the interstitial fluids and contamination with foreign fluids. The core should never be washed with water or oils prior to preservation. Suitable coring and handling procedures must be adopted to obtain laboratory data that are meaningful. Alteration of the rock can occur during coring, handling, preservation, sampling, and preparation prior to or during analysis. For a reliable determination of the fluid content of cores, a uniform procedure must be designed for handling and preservation. It should be emphasized that a properly designed program will benefit not only the near term user, but also future users of the core material. Some precautions in the handling and packaging of samples are listed:

a. All cores should be preserved as soon as possible after removal from the core barrel. Even after preservation, the samples should not be exposed to extreme conditions. The long term effectiveness of core preservation materials currently in use is unknown. A tested and trusted material should be used if the core is to be stored for extended periods of time.
b. Always minimize the gas head space in core preservation containers to prevent evaporation losses during core storage. This procedure will also minimize condensation losses on the inside surface of the container and help prevent breakage of the more loosely consolidated samples during shipment.
c. To minimize fluid loss, do not contact the core with cloth, paper, or any other dry material with fine capillaries.
d. Do not dip or coat the core directly with any fluid.
e. Follow stringent handling instructions for the processing and preservation of unconsolidated core (see 2.2.4 and 2.5).
f. Do not preserve an unconsolidated rock or other rock type in the same container as a rock of a vastly different lithology. This will minimize the potential of mechanical damage to weak samples.
g. Should any core be exposed to harsh handling conditions or washing, this should be noted. All pertinent data must be supplied with the core (see Figures 2-2 and 2-3).
h. Label each preservation container properly. In cases where confidentiality is an issue, this data should be coded with numbers and referenced to a master list.
i. It is recommended to have a company representative familiar with wellsite handling and preservation present during the coring operation. If this is not possible, explicit written instructions should be given to the service company

representative responsible for this activity. Upon arrival at the laboratory, cores should remain in a preserved state until ready for analysis.

j. ALL APPLICABLE SAFETY REGULATIONS MUST BE FOLLOWED WHEN HANDLING CORING EQUIPMENT AND CORE MATERIAL. The wellsite team should be protected, as necessary, against exposure to hazardous material using overalls, gloves, protective eyewear, etc. A hard hat, steel-toed shoes, and hearing protection are also recommended. Where toxic gases such as hydrogen sulfide are present, appropriate personal protective breathing apparatus must be available. Safety training is required for all wellsite workers prior to handling coring equipment, processing equipment, machinery, and core material.

2.8 READING LIST

1. Anderson, B., "Wettability Literature Survey—Part 1: Rock-Oil-Brine Interactions and the Effects of Core Handling on Wettability," *Journal of Petroleum Technology* (October 1986) 1125-1144.

2. Auman, J. B., "A Laboratory Evaluation of Core Preservation Materials," Society of Petroleum Engineers Reprint 15381, 1986.

3. Cornwall, C. K., "Core Preservation—An Alternative Approach," Society of Core Analysts European Conference Reprint, 1990.

4. Cuiec, L. E., "Evaluation of Reservoir Wettability and Its Effect on Oil Recovery," *Interfacial Phenomena In Petroleum Recovery*, N. R. Morrow, ed., Marcel Dekker, Inc., NY, 1990, 319-375.

5. Hunt, P. K. and S. L. Cobb, "Core Preservation With a Laminated, Heat-Sealed Package," SPE Formation Evaluation Symposium, December 1988.

6. Morrow, N. R., "Wettability and Its Effect on Oil Recovery," *Journal of Petroleum Technology* (December 1990) 1476-1484.

7. Park, A., "Coring Part 2: Core Barrel Types and Uses," *World Oil*, April 1985.

8. Park, A., "Coring Part 2: Planning the Job," *World Oil*, May 1985.

9. Park, A., "Improved Oil Saturation Data Using Sponge Core Barrel," SPE Production Operations Symposium, February 27 - March 1, 1983, Oklahoma City, OK, p. 87-91.

10. Sattler, A. R., Heckes, A. A., and Clark, J. A., "Pressure Core Measurements in Tight Sandstone Lenses During the Multiwell Experiment," *SPE Formation Evaluation Journal* (1988), 645-650.

11. Skopec, R. A., Mann, M. M., Jeffers, D., and Grier, S. P., "Horizontal Core Acquisition and Orientation for Formation Evaluation," *Society of Petroleum Engineers Drilling Journal*, Vol. 7, No. 1, p. 47-54, 1992.

12. Swanson, R. G., "Sample Examination Manual," American Association of Petroleum Geologists, Tulsa, OK, 1985.

13. Worthington, A. E., Gidman, J., Newman, G. H., "Reservoir Petrophysics of Poorly Consolidated Rocks, Part I. Wellsite Procedures and Laboratory Methods," Society of Core Analysts Conference (1987) Paper Number 8704.

SECTION 3—CORE SCREENING AND CORE PREPARATION

CONTENTS

Page

1 CORE SCREENING AND CORE PREPARATION 3-1
3.1 General. 3-1
3.2 Core Description 3-1
3.3 Core Gamma Logs and Core Spectral Gamma Logs 3-2
3.4 Core Imaging 3-2
3.5 Core Sampling and Core Preparation (Basic Core Analysis). 3-4
3.6 Core Cleaning 3-5
3.7 Drying 3-7
3.8 Sample Preservation 3-7

Tables
3-1 Selected Solvents and Their Use. 3-6
3-2 Core Sample Drying Methods. 3-7

3 Core Screening and Core Preparation

3.1 GENERAL

The recommendations included in this document may involve the use of hazardous materials, operations, and equipment. This document does not address all of the relevant safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices prior to use and to comply with all applicable regulatory requirements regarding use and disposal of materials and equipment.

3.2 CORE DESCRIPTION

3.2.1 Principle

The purpose of core examination and description is the recognition of lithological, depositional, structural, and diagenetic features of whole or slabbed core. Qualitative and quantitative core descriptions provide the basis for routine core analysis sampling, facies analysis, and further reservoir studies such as reservoir quality and supplementary core analyses. A description provides a permanent, accessible record of the core.

3.2.2 Apparatus and Supplies

The following equipment is recommended for use in standard descriptions of the core:

a. Logging form for systematic data collection.
b. Microscope or hand lens.
c. Length measurement scale.
d. Grain size scale.
e. Appropriate chemicals such as:
   1. Water or brine to improve visibility of geological structures.
   2. Dilute HCl acid to identify carbonate minerals.
   3. Alizarin red to differentiate calcite and dolomite.
   4. Hydrocarbon solvents to facilitate the detection of oil fluorescence under ultraviolet light.
f. Coring log, drilling report, mud logs, well site information on lost core.
g. Core gamma log.
h. Ultraviolet light.

3.2.3 Precautions

The following precautions should be observed:

a. If the samples being described have been preserved for special testing, exposure of the samples to air and chemicals should be avoided until tests on the preserved core have been completed.
b. Choose an appropriate logging format. The objective of a logging form should be to accurately represent the core. When describing core, it is important to collect and record data in a systematic manner.

3.2.4 Procedures

The following procedures should be used:

a. Lay out the core to be analyzed on a viewing table.
b. Check the amount of core with the coring report and ensure that no core material has been lost during transportation. Note any damage or alteration to the core during well-site handling and transportation.
c. Check the numbering and order of boxes or containers against cumulative depth.
d. Check core continuity and orientation with respect to the top of the core. If the core has been marked in accordance with 2.2.3, the top of the core is up when the red scribe mark is on the right.
e. Check the order of core segments in the boxes; look for matching “breaks” or scribe marks from one piece to the next and from one box to the next.
f. Measure and mark footage on each box. Mark core to the nearest $^1/_2$ inch (1 centimeter).
g. If downhole gamma-ray logs are available, a comparison should be made with the core gamma-ray logs (see 3.3) to verify that the cored interval depths are consistent with well log depths.
h. Assess the entire cored sequence before commencing. Seek out distinguishing features such as units, contacts, unique markers (e.g., bentonite, coal).
i. Record major features such as the following using standardized nomenclature and abbreviations (see Section 8):
   1. Core lithology (shale, sandstone, limestone, etc.).
   2. Color.
   3. Bedding (thickness, bed contacts, erosional markers).
   4. Obvious sedimentary structures.
   5. Texture (grain size, angularity/roundness and sorting).
   6. Composition (grains, cement, fossils).
   7. Porosity types.
   8. Diagenetic and tectonic features.
   9. Note any oil stain and related fluorescence.
j. Minimize centimeter-by-centimeter descriptions. Some fine scale features may be important and should be recorded.
k. Record nonreservoir rocks, e.g., mudrocks, in addition to potential pay sequences. Subtle changes in lithology may assist correlation between core and well logs and definition of relevant zonation.

l. Record fracture information such as width, extent, density, fracture orientation if core is oriented, and presence of cementation or mud. Where possible, differentiate between natural and coring induced fractures.

3.3 CORE GAMMA LOGS AND CORE SPECTRAL GAMMA LOGS

3.3.1 Principle

Naturally occurring gamma-ray emitters (radiogenic daughters of uranium and thorium along with potassium-40) give a measurable gamma-ray response that can be recorded with depth. If such a record measured at the surface is compared with the gamma-ray readings taken from a downhole gamma-ray log, the results can frequently be used to adjust the depth of the core to coincide with the open-hole logging depths and to identify zones where core has been lost.

3.3.2 Apparatus

The recommended apparatus consists of a conveyer for moving core, lead shielding to reduce background gamma radiation, and suitable gamma-ray detectors. A typical detector system consists of a shielded scintillation crystal coupled with a photomultiplier. The scintillation crystal is normally sodium iodide doped with thallium [NaI(Tl)]. Other scintillation crystals include cesium iodide (CsI) and bismuth germanate (BiGeO).

Signals from the detectors are processed and the gamma-ray events are energy-sorted and counted. Standard gamma-ray units should provide total gamma counts in APIU units. With the spectral gamma units these counts are converted into potassium, uranium, and thorium concentrations, and standard gamma-ray log API units. Signals are displayed on a computer graphics screen or plotted in downhole log formats and scale for direct comparison with downhole logs.

3.3.3 Procedures

The following procedures should be used:

a. Set the recorder readout to correspond to the scale used with the downhole gamma-ray log.
b. Log the core gamma-ray response from bottom to top. This conforms with the sequence used in downhole logs.
c. Core gamma-ray logging should be carried out smoothly, without interruption, in the correct order and spacing with matching core breaks fitted together.
d. Record a repeat section near the bottom of the core to establish the reproducibility of the measurement.

3.3.4 Advantages

Core gamma-ray logging is widely available and is used in general practice to correlate core depth with log depth. The spectral gamma-ray apparatus will differentiate uranium, thorium, and potassium concentrations and can be used to better identify and differentiate shale, particularly in sandstone cores with large amounts of potassium feldspar and mica.

3.3.5 Limitations

This technique is unable to detect low gamma-ray activity and can suffer from significant background interference. Analysis of samples for spectral gamma-ray response requires a slower rate of logging than that used to determine total gamma-ray response.

3.3.6 Calibration

The apparatus should be calibrated before evaluating any core material. Calibrations are sensitive to the size of core and the gamma-ray energy range. Calibration should be done by measuring the gamma-ray response to calibration tubes containing pure samples of the elements of interest. The total gamma-ray apparatus requires a calibration tube containing potassium (K-40), uranium (U-238), and thorium (T-232) of known activities. The calibration procedure for the spectral gamma unit requires the same elements but in individual tubes. A blank calibration tube should be measured to ensure a minimal amount of background gamma-rays are interfering with the apparatus. The speed of the conveyor should be slow enough, to provide an acceptable ratio of signal to background noise. A ratio of three times the square root of the count rate to background count rate greater than one is acceptable, i.e.:

$$\frac{3 + \sqrt{\text{count rate}}}{\text{background count rate}} > 1$$

3.3.7 Accuracy

The precision of the measurement varies with the square root of the count rate.

3.4 CORE IMAGING

A recorded image of the core is essential. This record will provide information that can be used if viewing of the core is not possible. The record may include visual images of the surface features of the core using photographic techniques, visual representations of the internal structures of the core such as x-radiographs, computed x-ray tomographs, magnetic resonance images, or acoustic images. Any record should include information on core depth along with a scale or legend indicating intensity range of the recorded image.

In addition to photographic representations, modern imaging techniques provide quantifiable information for the core that can be used as the basis for routine core analysis sampling, facies analysis, reservoir quality studies, supplementary core analyses, and downhole log interpretations.

3.4.1 Photography

3.4.1.1 Principle

Core is normally photographed under both natural (5,500 K) and ultraviolet light (254-365 nm) along with a standard color scale. The natural light photographs show lithology and sedimentary structures and allow specific features recorded in the core description to be examined. Ultraviolet light photography can highlight hydrocarbon-bearing zones by causing most oils to fluoresce in shades ranging from orange-brown for heavy oils to bright yellow for high gravity oils; condensates can appear as a very light white to blue-white color. Non-hydrocarbon bearing zones will appear as purple regions, although some minerals such as chalky-limestones also appear purple.

3.4.1.2 Advantages

Core photography provides a visual record of the core that can be used to reconstruct damaged core pieces, minimize core handling, and identify the location of samples if photographed following sampling.

3.4.1.3 Limitations

Photographic colors may not represent true colors of the core. Highlighting of features may require wetting the core surface for photography. A scale and color bar are required if adjustments are necessary.

3.4.2 X-Ray Techniques

3.4.2.1 Principle

X-ray techniques can be used to noninvasively examine the internal character of a core. An x-ray beam is directed through the core and variations in attenuation of the incident x-ray beam are measured. In order of increasing definition, these techniques are: fluoroscopy, x-radiography, and computed tomography (CT scanning).

The utility of these methods relies on their sensitivity to density contrasts within the core. Consequently, non-porous, dense areas will be contrasted against porous, low-density areas. High definition of density variations within the core requires multi-directional measurements or spatial separation of the contrasted areas.

3.4.2.2 Fluoroscopy

In this technique, the core is moved across an x-ray source. The beam, attenuated by the core, impinges on a fluorescent screen, is intensified, and is recorded by a video camera. The captured image can be viewed on a monitor, recorded on a video cassette, and converted to digital format for later processing and viewing. The output from this technique is a continuous image along the length of the core.

Because the core is moving, the resolution of the resulting image may be diminished. It does, however, enable identification of density variations, some geological structures, and areas of lost core within plastic or fiberglass sleeves. It is also useful in identifying fractured and badly broken zones.

3.4.2.3 X-Radiography

A core sample is positioned between an x-ray source and an x-ray sensitive film. Attenuation of the x-ray beam by the core is captured on film. The developed film can be viewed on a light table, or used to make a black and white print.

It is possible to obtain multiple images of the same piece of core and display them side-by-side. The core should be first x-rayed to obtain an initial image and is then rotated 90 degrees in order to obtain a second image. This is useful in orienting sleeved core prior to slabbing because it can establish the bedding direction.

During x-radiography, the core sample is stationary and provides a higher resolution image than the fluoroscopy method. X-radiography enables detection and evaluation of internal geological structures such as bedding planes, fractures and nodules, lithological changes, and bulk density.

3.4.2.4 Computed Tomography (CT)

In CT, the core is scanned by a highly collimated x-ray beam. Detectors on the opposite side of the core measure the intensity of the transmitted beam. The x-ray source and/or detectors rotate or translate around the sample. A series of x-ray attenuation measurements are made and numerically reconstructed to give the spatial distribution of x-ray attenuation coefficients within the sample.

The resolution of the image depends on the thickness of the x-ray beam, the number of detectors in the scanner, and the size of the pixel array used to reconstruct the image. X-ray beam thickness varies from about 2 millimeters to 10 millimeters. CT enables detection and evaluation of internal geological structures such as bedding planes, fractures and nodules, lithological changes, and bulk density. Combination of sequential x-y scans along the core axis can provide slab images or three-dimensional images of the core.

3.4.2.5 Advantages

X-ray techniques provide quantified, objective representations of the core. These representations can provide similar advantages as described for core photographs in 3.4.1.2 without the need for exposing a surface of the core.

3.4.2.6 Limitations

Resolution of the images is less than that provided by photographs. X-ray attenuation may vary with mineralogy, depending on the energy of the x-ray beam. Some applications are still in the development stage. Further development

of the relationships between rocks and x-ray attenuation may extend these methods to other areas of core analysis.

3.4.3 Nuclear Magnetic Resonance (NMR)

3.4.3.1 Principle

NMR imaging is used to provide a reconstructed image of fluids within a core sample. NMR measurements are based on the observation that when the proper radio frequency excitation energy is applied to an ensemble of nuclei, a portion of nuclei at a lower energy level can be promoted to a higher energy level. When the excitation is removed, the rate at which excited nuclei return to the lower level can be detected and measured. The excitation energy is supplied by an oscillating magnetic field in resonance with the nuclei. The nuclei response is measured through a tuned receiver coil. The location of the excited nuclei can be determined by the application of magnetic field gradients during the excitation and detection processes.

The primary nucleus used in core analysis applications is ^{1}H. Other nuclei that can be imaged include, but are not limited to, ^{2}H, ^{31}P, ^{23}Na, ^{13}C.

3.4.3.2 Advantages

Magnetic resonance images are noninvasive and provide an image showing fluid locations within a sample. The advantages described in 3.4.2.5 apply provided the sample is contained within a suitable non-metallic holder.

3.4.3.3 Limitations

This technique is not descriptive of the core but of the liquids within the core. The technique requires a high density of resonant nuclei for an adequate signal. Hence, low porosity cores will give weak signals. Parmagnetic or ferromagnetic minerals may inhibit or degrade the measured signal and can distort the image.

3.5 CORE SAMPLING AND CORE PREPARATION (BASIC CORE ANALYSIS)

3.5.1 Principle

The sampling procedure for basic core analysis is determined by the type of information required. Sampling will generally take into account one or more of the following:

a. Lithological distribution.

b. Porosity and permeability variations within the lithological units.

c. Distribution of hydrocarbons.

3.5.1.1 Plug Samples

Referred to as plugs, should be removed from sections of whole core oriented either vertically or horizontally with respect to the whole core axis or with respect to the normal of the bedding planes. These plugs will provide data on matrix properties. See 3.5.2.4 for procedures for removal of core plugs.

3.5.1.2 Full Diameter Samples

Full diameter samples (sections of whole core), in addition to plug samples, should be taken in the following types of zones or where there are significant large scale heterogeneities that are different from the matrix properties. These can occur in, but are not limited to:

a. Vuggy carbonates—Vugs can bias the measurement of porosity within a sample. Samples should be taken to obtain a representation of the relative proportion of vugs-to-rock in the reservoir.
b. Fractured reservoirs—Fractured zones may be difficult to sample due to the fragile nature of the rock. However, natural fractures can have a significant positive impact on reservoir permeability and should be sampled where possible or appropriate.
c. Conglomerates—Within a conglomerate, if the pebble size is large with respect to the sample size, sample size can greatly bias rock property measurements. To obtain a representative sample of a unit of conglomerate it is necessary that sample size be sufficient to include all pebble sizes.

3.5.2 Sample Cutting, Trimming, and Mounting

3.5.2.1 Principle

The core should be cut and trimmed to provide regularly shaped samples, most commonly right cylinders. Unconsolidated, disintegrating, and very friable samples may have to be mounted prior to testing. Regardless of the degree of lithification, sample handling should be kept to a minimum.

3.5.2.2 Apparatus and Supplies

The following equipment and supplies are commonly used in core sample cutting and trimming operations:

a. Large slab saw with a diamond blade.
b. Trim saw with a diamond blade.
c. Drill press with diamond-edged core drills, capable of drilling cylindrical samples.
d. Dressing wheel or saw for squaring plug ends.
e. Fluid pumps for delivering various coolants (brine, oil, air, water, liquid N_2) to the cutting surfaces.
f. Indelible marking medium such as India ink.
g. Sleeves of lead, aluminum, or heat shrinkable plastic for mounting soft, unconsolidated, or very fragile samples.

The following information should be available prior to cutting, trimming, or mounting operations:

a. The total number of samples required.

b. Size and orientation needed.

c. Exact depth locations for samples and how they are to be labeled.

d. Fluids to use for cutting samples (cores containing clays or shale laminations sensitive to fresh water will deteriorate unless proper drilling fluids are used).

e. Available core images.

f. If required, materials for the preferred method of preservation for both plugs and core from which the plugs are cut.

3.5.2.3 Precautions

The following precautions should be enforced during sample cutting, trimming, and mounting operations:

a. Saw and drill press operators should wear protective earplugs, goggles, gloves, and other necessary safety equipment.

b. Lubricant/coolant flow should be sufficient to cool the drill bit or saw blade and remove the cuttings without eroding the sample.

3.5.2.4 Procedures

The following procedures for preparing various type core samples should be followed:

a. Plug samples:

1. Drill plugs at specified points using the appropriate size bit. Care should be taken to drill straight plugs. If too much pressure is applied during the drilling operation, the bit will flex causing the plug to deform.

2. Trim plugs to the required length making sure that the ends are parallel. Keep and label trimmed ends.

3. Label, preserve, and/or store samples, as required.

b. Full diameter samples:

1. Cut sections of the core selected for analysis slightly longer than required to allow for finish grinding operations.

2. Remove barbs and smooth chipped edges by dressing the ends. Cylinder ends should be as close to parallel as possible.

3. Clearly mark the samples and preserve or store as required.

c. Unconsolidated samples—There are two distinct methods of preparing unconsolidated cores. The procedures depend on whether the core is stabilized by freezing, epoxy impregnation, or both or is unstabilized. Stabilized cores are generally drilled while unstabilized cores are plunge cut.

1. Stabilized cores—Frozen cores are obtained using the practices listed for plug samples, with appropriate modifications to avoid thawing of the core:

(a) Pre-cool the core with dry ice and drill with liquid N_2 coolant.

(b) Drill plugs at specified points using the appropriate size bit. Care should be taken to drill straight plugs. If too much pressure is applied during the drilling operation, the bit will flex causing the plug to deform. Ensure that there is adequate liquid nitrogen flow so that the sample is kept frozen and the cuttings are being removed.

(c) Trim plugs to the required length using a liquid N_2 cooled saw blade, making sure that the ends are parallel. Keep and label trimmed ends.

(d) Mount plugs in a suitable pre-weighed sleeve with pre-weighed and properly sized end screens.

(e) Label, preserve, and/or store samples as required.

Epoxy-stabilized cores can be frozen and drilled using the procedures described in a-e above or by the procedures described for plug samples (see 3.5.2.4.a).

2. Unstabilized cores—Unfrozen cores can be cut using a plunge cut technique. The cutter should be slowly pushed in the core and removed. The cut plug should be gently removed from the cutter and inserted directly into a preweighed sleeve. The plug ends are then trimmed.

3.6 CORE CLEANING

3.6.1 Introduction

Prior to most laboratory measurements of porosity and permeability, the original fluids must be completely removed from the core sample. This is generally accomplished through flushing, flowing, or contacting with various solvents to extract hydrocarbons, water, and brine.

3.6.2 Apparatus and Supplies

Various techniques and apparatus for cleaning are described in the procedures below and in Section 4.

Some solvents used for hydrocarbon extraction purposes are listed in Table 3-1. Listed solvents are those most frequently used for extracting samples for routine analysis. Some are preferred for specific applications; e.g., chloroform has been found to be excellent for many North American crudes and toluene has been found useful for asphaltic crudes. Prior to cleaning samples with unknown oil properties, a subsample should be tested with various solvents for cleaning efficiency.

Since residual salt crystals affect the measured porosity and permeability, core samples containing a formation water with high salinity may require additional extraction to remove

salt. Salt can be removed with methyl alcohol or other solvents in which salt is soluble.

The various solvents used for extracting core samples can be reclaimed by well-known physical and chemical methods. Such recovery techniques can make practical the use of an expensive solvent.

Table 3-1—Selected Solvents and Their Use

Solvent	Boiling Point, °C	Solubility
Acetone	56.5	oil, water, salt
Chloroform/methanol azeotrope (65/35)	53.5	oil, water, salt
Cyclohexane	81.4	oil
Ethylene Chloride	83.5	oil, limited water
Hexane	49.7–68.7	oil
Methanol	64.7	water, salt
Methylene chloride	40.1	oil, limited water
Naphtha	160.0	oil
Tetrachloroethylene	121.0	oil
Tetrahydrofuran	65.0	oil, water, salt
Toluene	110.6	oil
Trichloroethylene	87.0	oil, limited water
Xylene	138.0 –144.4	oil

3.6.3 Precautions

The following precautions should be enforced during core sample cleaning operations:

a. When using solvents, it is the responsibility of the user to establish appropriate safety and health practices prior to use and to comply with all applicable regulatory requirements regarding use and disposal of materials.

b. The solvent selected should not attack, alter, or destroy the structure of the sample.

c. Chloroform may hydrolyze during extraction, forming hydrochloric acid as a product.

d. Not all solvents are compatible with all extraction equipment configurations. Possible solvent/equipment reactions should be considered in selecting equipment and solvent for use in core cleaning operations.

e. Closed-type electrical heaters should be used whenever flammable solvents are used. Safety precautions such as adequate ventilation of the laboratory and ready accessibility of fire extinguisher equipment and safety showers should be observed.

f. Extraction should be conducted under ventilating hoods equipped with forced-draft ventilation.

g. Temperature effects on the samples should be considered. See 3.7 for recommendations regarding drying of core samples.

3.6.4 Procedures

3.6.4.1 Solvent Flushing by Direct Pressure

Extraction of hydrocarbons and salt from reservoir rocks can be achieved by injecting one or more solvents into the core sample under pressure and at room temperature. The pressure used should be dependent on the sample permeability and may range from 10 to 1,000 psi. The core samples may be held in a rubber sleeve under overburden pressure or in a suitable core-holding device that will permit the flow of solvent through the matrix of the sample. The volume of solvent required to completely remove hydrocarbons in the core sample is dependent on the hydrocarbons present in the sample and the solvent used. The core is considered clean when the effluent is clean. In some instances, more than one solvent may be required to remove heavy, asphaltic-type crude oils.

3.6.4.2 Flushing by Centrifuging

A centrifuge with a specially designed head is used to spray warm, clean solvent (from a still) against the core samples. The centrifugal force causes solvent to flow through samples displacing and extracting the oil (and water). The speed of rotation should be varied from a few hundred to several thousand revolutions per minute (rpm), depending on the permeability and degree of consolidation of the core. Most common solvents can be used. The apparatus and procedure are described in Conley and Burrows.[1]

3.6.4.3 Gas-Driven Solvent Extraction (U.S. Patent 2,617,719)

In this procedure, a core is subjected to repeated cycles of internal-dissolved or solution-gas drive until the core is cleaned of hydrocarbons. The remaining solvent and water are removed by means of a drying oven.

[1]Conley, F. R., and Burrows, D. B., "A Centrifuge Core Cleaner," *Journal of Petroleum Technology,* VIII, 61, October 1956.

When a core from an oil-bearing formation is brought to the surface and depressurized, the gas dissolved in the oil comes out of solution and displaces some of the oil and water out of the core. This results in some gas-filled pore space at atmospheric pressure. The gas-filled space in the core can be almost completely filled with solvent by surrounding the core with a suitable solvent containing a dissolved gas and applying sufficient hydraulic pressure. Under this condition, the solvent mixes with oil in the core and subsequent depressuring to atmospheric pressure removes some of the residual oil.

Carbon dioxide gas is excellent for this purpose because of low fire or explosion hazards and high solubility in most solvents. Some of the solvents that can be used are naphtha, toluene, or mixtures of solvents. With certain types of crude oil, cleaning time may be reduced if the core chamber is heated by a water bath, steam bath, or by electric heaters. One successful application of this method for routine cleaning of cores uses carbon dioxide and toluene at 200 psig, with a hydraulic pressure of 1,000 psig. Cycles of approximately 30 minutes are used. The apparatus and procedure are described by Stewart.[2]

3.6.4.4 Distillation Extraction Method

A Soxhlet extractor and suitable solvent or solvents should be used to dissolve and extract oil and brine. Extraction can be arranged in a manifold so that the oil-and water-laden solvent siphons from each extractor into a common still from which fresh solvent is continuously distilled, condensed, and again distributed to all the extractors.

The cleanliness of the sample is best determined from the color of the solvent that siphons periodically from the extractor. Extraction should be continued until the extract remains clear. Non-luminescence of extract under fluorescent light is a good criterion for determining complete extraction of the oil for a given solvent. It should be noted that the complete extraction of certain oils from core samples may require more than one solvent, and the fact that one solvent is clear after contact with the sample does not necessarily mean that oil has been completely removed from the sample. Details of one apparatus and procedure may be found in 4.3.

3.6.4.5 Liquefied Gas Extraction

Liquefied gas extraction uses a pressurized Soxhlet extractor and a condensed, low-boiling-point, polar solvent. The process is a distillation extraction procedure that uses pressurized solvent to clean the core. Solvent is regenerated through low temperature distillation. Because the extraction is conducted at or below room temperature, it can be performed on heat-sensitive core such as those containing gypsum.

[2]Stewart, Charles R., U. S. Patent Number 2,617,719, "Cleaning Porous Media," November 11, 1952.

3.6.5 Advantages

Cleaning a core removes original fluids from the core, preparing it for further tests that do not require those fluids.

3.6.6 Limitations

Individual test conditions may require a particular technique for optimal results. Some techniques may be more applicable to particular rock types or cleaning requirements.

3.7 DRYING

Conventional core samples can be dried by the methods listed in Table 3-2:

Table 3-2—Core Sample Drying Methods

Rock Type	Method	Temperature, °C
Sandstone (low clay content)	Conventional oven	116
	Vacuum oven	90
Sandstone (high clay content)	Humidity oven, 40% relative humidity	63
Carbonate	Conventional oven	116
	Vacuum oven	90
Gypsum-bearing	Humidity oven, 40% relative humidity	60
Shale or other high clay rock	Humidity oven, 40% relative humidity	60
	Conventional vacuum	

Each core sample should be dried until the weight becomes constant. Drying times may vary substantially, but are generally in excess of four hours.

3.7.1 Precautions

Some precautions that should be observed in drying samples for routine core measurements are:

a. Samples containing clays must not be dehydrated during preparation. Care must be exercised in drying these samples. In some cases, temperatures lower than those indicated in Table 3-2 should be used to prevent the dehydration of clays.
b. Samples containing gypsum will require specific procedures during preparation. Extreme care should be exercised to avoid both loss of water and change of the crystalline structure.
c. Samples must be protected from erosion by the drip of clean solvent when utilizing the distillation extraction technique.
d. The extraction technique should not physically damage the core.
e. The usual criterion for sample cleanliness is a clean extract. However, many solvents are not complete solvents for all types of oils and a clean extract may reflect oil solubility and not complete extraction.

f. Samples containing heavy asphaltic oils may require the cycling of more than one solvent.
g. More effective core cleaning can often be achieved by a combination of solvents.
h. Allow solvent laden samples to vent in a fume hood before placing in a closed drying oven.

3.8 SAMPLE PRESERVATION

Preservation of samples in the laboratory will depend on the length of time between tests and the type of tests to be done. Any storage or preservation technique should ensure that structural integrity is maintained and that unwanted drying, evaporation, and oxidation are avoided.

Laboratory samples can be preserved using the techniques described in 2.5. Freezing of samples in the laboratory should follow the precautions outlined in 2.4.4, 2.4.5, and 2.5.2.2.

SECTION 4—FLUID SATURATION METHODS

CONTENTS

Page

4 FLUID SATURATION METHODS 4-1
4.1 Introduction 4-1
4.2 Retort Method at Atmospheric Pressure 4-1
4.3 Distillation Extraction Method (Dean Stark) 4-7
4.4 Solvent Flushing 4-23
4.5 Scanning Methods 4-25
4.6 Coal Analysis 4-26
4.7 Oil Shales 4-27
4.8 Gypsum-Bearing Cores 4-28
4.9 Historical 4-31
4.10 References 4-31
4.11 Bibliography 4-31

Figures
4-1 Stainless Steel Oven Retort and Water Bath—Atmospheric Pressure 4-3
4-2 Retort Oil Correction Curve 4-4
4-3 Retort Cup—Sidewall Sample 4-6
4-4 Dean-Stark Apparatus for Volumetric Determination of Water 4-8
4-5 Dean-Stark Extraction Apparatus for Gravimetric Determination of Water . . . 4-8
4-6 Gas Collection/Pressure Depletion System. 4-14
4-7 Plug and Donut Sample For Filtrate Invasion Study. 4-21

Tables
4-1 Rock Type and Recommended Fluid Saturation Testing Method(s) 4-1
4-2 Measurement Capabilities of Scanning Saturation Techniques 4-26

4 Fluid Saturation Methods

4.1 INTRODUCTION

This section documents the specialized techniques developed for analysis of core samples with different sample physical characteristics and sizes, obtained by various coring methods. Table 4-1 lists the principal techniques for determining core fluid saturations.

Paragraphs 4.2 through 4.5 describe the seven principal saturation analysis methods listed in Table 4-1 for full-diameter cores and plugs/sidewall cores. Plugs are defined as samples drilled or shaped from cores recovered with any of several full-diameter coring devices. Sidewall cores are defined as cores that are obtained after the wellbore has been created by the drill bit or some type of full-hole coring device.

Paragraphs 4.6 through 4.8 address fluid saturation analysis methods for coal, oil shale, and gypsum-bearing samples. Oil shale and coal samples may be obtained with full-diameter coring devices. However, oil shale and coal may also be quarried from surface or shallow formations, and require non-routine sampling procedures different from those typically used for reservoir core. The reporting format of fluid saturations for these latter materials differs from typical reservoir rocks in that the saturations may be expressed as weight percent or gallons per ton instead of percent of pore volume.

Scanning methods that are capable of measuring fluid saturations by non-destructive techniques are included in 4.5. Additionally, a historical section (see 4.9) is included in which some previously acceptable methods for fluid saturation determination are described, but are not recommended.

A difficult, if not impossible, formation evaluation task is recovering a core having the same saturation and distribution of fluids as occurred in the formation prior to coring. Changes in fluid content and distribution frequently take place during the coring, retrieval, preservation, and transportation phases. Laboratory handling, sampling, and testing processes can bring about further alterations. These issues are not addressed in detail in this section, but the reader should be aware of the many complications that influence the final fluid saturation values furnished in a laboratory core analysis report. Fluid saturation analysis may alter the wetting of the core and thus affect further analyses on the core material.

Table 4-1—Rock Type and Recommended Fluid Saturation Testing Method(s)

Rock Type	Recommended Testing Methods
Consolidated clastics, carbonates	a,b,c,d,e,f
Unconsolidated (light oil)	c,d,e
Unconsolidated (heavy oil)	c, c(*),e
Vuggy carbonates	b,d,e,f
Fractured	a,b,d
Clay bearing	a,c(*),e
Evaporites	g,e
Low permeability	a,b,c,d,e,f
Coal	h
Shales	a,b,c
Oil shale	a(*)
Diatomite	c,e

Legend:
a = retort at atmospheric pressure.
b = distillation extraction (full diameter).
c = distillation extraction (plug).
d = pressure-retained core method.
e = solvent flushing/Karl Fischer.
f = sponge core barrel method.
g = gypsum-bearing method.
h = coal method.
(*) = modified procedure.

4.2 RETORT METHOD AT ATMOSPHERIC PRESSURE

4.2.1 Basic Procedure

4.2.1.1 Principles of Analysis

Oil and water fluid saturations are obtained by a high-temperature retorting process in which the oil and water contained in a fresh sample of crushed core material are vaporized, condensed, and collected in calibrated glassware. The gas saturation is determined on an adjacent, lithologically similar sample by placing it in a mercury pump and measuring the amount of mercury injected with the water and/or oil in place.

4.2.1.2 Apparatus

The following items describe suitable apparatus used in the basic retort method:

a. Stainless steel retort(s)—See Figure 4-1. Such retorts are designed to hold 100 to 175 grams of crushed core material. Each retort is equipped with a threaded cap, that in turn is equipped with a gasket to prevent leakage of condensable gases. A long, stainless steel condensing tube is connected to the opposite end of the retort. A coarse mesh screen is placed in the bottom of the retort to prevent crushed core material from entering the condensing tube.

b. Oven—See Figure 4-1. Such ovens are capable of holding multiple retorts. Strip-type electrical heating elements are preferred. A temperature controller, thermocouple, and temperature readout device are also required.

c. Water bath—See Figure 4-1. A water bath should be located so that the condensing tubes pass through it, thereby enhancing condensation of the evolved gases.

d. Calibrated glass receiving tube—A common 15-ml centrifuge tube is generally acceptable, although a tube with a larger capacity may be required. The required tube size depends on the amount of retorted material and the total fluids contained within that material. The receiving tube is affixed to the end of the condensing tube with a rubber stopper arrangement.

e. Rock hammer and crusher—A geologist's pick or a masonry hammer may be used. Typical crushers are of the "chipmunk" variety.

f. Diamond saw—A variety of commercial core saws, equipped with diamond saw blades, may be used.

g. Mercury pump—Typical mercury pumps are equipped with a cell, lid, and valve arrangement. Typical cells can accommodate a sample with a bulk volume of 10 to 15 cm^3. The mercury pump is equipped with a vernier caliper graduated in 0.1 cm^3 increments. A pressure gauge capable of indicating pressures to 1,000 psi (6.895 MPa) is connected to the system.

h. Centrifuge—A hand-crank centrifuge capable of holding four of the 15-ml centrifuge tubes is suitable. Other centrifuges commensurate with the tube size and centrifugal forces required may be used.

4.2.1.3 Procedures/Precautions

The basic procedure is normally conducted on samples taken from full-diameter cores. Typical analyses are done on a foot-by-foot basis, except some lithologies such as shale, anhydrite, gypsum, and heavy clays are usually described and skipped in this type of analysis. A representative sample approximately two inches in length is broken from each foot using a geologist's or similar type hammer. This sample is broken lengthwise into two halves. One half is designated for the fluid saturation sample and the other half is used to obtain a plug for the determination of permeability and/or Boyle's Law porosity (see 5.3.2.1.1 and 5.3.2.2.1). Alternatively, a 1 to 2 inch (25.4 to 50.8 millimeter) piece may be broken adjacent to the piece selected for fluid saturation testing and the plug drilled from it. The advantage of this latter sampling method is that it can afford a larger and longer plug, thereby reducing the potential for drilling fluid contamination.

The sample (10 to 15 grams) for the gas space measurement should be chipped or shaped to a convenient size, i.e., dimensions that allow it to be placed in the cell of a mercury pump. In addition, the sample should be well rounded to permit mercury conformance. The weight of the sample is recorded and the bulk volume of this sample is determined by mercury displacement for later use in the calculation of a summation-of-fluids porosity (see 5.3.2.2.2 and 4.2.1.4). The cell valve is closed and the pressure increased to 750 to 1,000 psi (5.171 to 6.895 MPa) depending upon the permeability and/or porosity of the sample. The amount of mercury injected (properly corrected by pump calibration procedures, see 4.2.1.8) is recorded.

The sample for the water and oil measurement should be crushed into approximately $^1/_4$-inch (6.4-centimeter) pieces. The crushed core material is screened using a 3 or 4 Tyler mesh screen [approximately 0.25 inch (6.4 millimeter)] to remove any fines created in the crushing process. An amount of this material (usually 100 grams to 175 grams) is weighed to the nearest 0.01 gram and poured into the retort. The retort lid is secured and the retort, along with other similarly prepared retorts, is placed in the oven. The oven temperature is initially maintained at 350°F (177°C) (Hensel, 1982[1]) until all samples cease to give up water. The water volumes are recorded and the oven temperature is then raised to a temperature between 1,000 and 1,200°F (538 and 649°C). The fluid collection tubes are monitored and when all samples cease to give off fluids the process is considered complete. The retorting time usually ranges from 20 to 45 minutes. The total water and oil volumes are recorded.

The initial oven setting of 350°F (177°C) is selected to remove pore water, absorbed water, interlayer clay water (e.g., smectite), and waters of hydration (e.g., $CaSO_4$), but not hydroxyl clay water. However, this method is not appropriate for samples containing gypsum or large amounts of montmorillonite. Gypsum bearing core analysis procedures are described in 4.8 as a special core analysis procedure. Although samples containing montmorillonite can pose problems in determining accurate fluid saturation, heating to 350°F (177°C) is documented (Brown, 1961[2]; Mackenzie, 1970[3]) as the lowest temperature at which hydroxyl waters are desorbed. Although the clay hydroxyl water weight loss at this temperature may be as small as 5 percent, it may impact the final water and oil saturation values. This method is superior to other retorting techniques, e.g., the plateau method (Hensel, 1982[1]). The plateau method suggests that the plotting of water collected versus time data, while continuous heat is applied, will yield plateaus whereby a proper time for reading only pore water can be determined for each formation being tested.

The final retorting temperature of 1,000 to 1,200°F (538 to 649°C) may yield incorrect oil volumes. Some core material may contain solid hydrocarbons, e.g., kerogen, gilsonite, etc. that break down at these temperatures. The analyst should take mineral composition into consideration and may elect to make arbitrary and judicious corrections for the oil collected. In extreme cases, the retort procedure should not be used and other methods, e.g., distillation extraction (Dean-Stark) (see 4.3) procedures should be employed.

Other sources of error in the measurement of oil volumes stem from: (a) mechanical hold-up in condensing tubes, (b) leaking retort seals, and (c) coking and cracking of certain

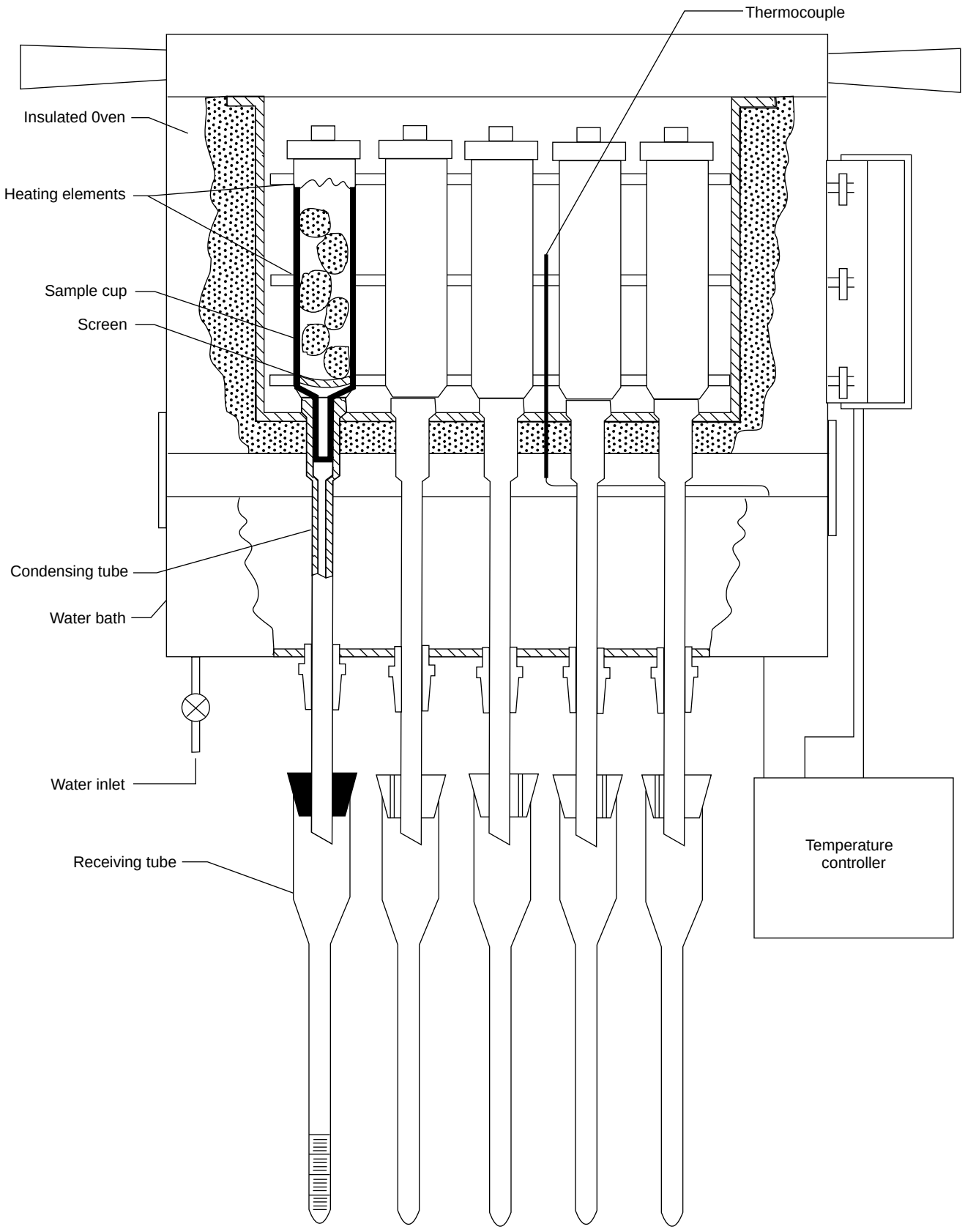


Figure 4-1—Stainless Steel Oven Retort and Water Bath—Atmospheric Pressure

oils. Oil volume correction curves (See Figure 4-2) must be employed routinely.

The "total" water collected after the complete process may be of value in determining if "pore" water may have been mechanically retained in the condensing tube. The analyst may note a calculated grain density for a sample that is anomalous to the general trend. A comparison between the initial (pore) water volume and the final (total) water volume indicates a difference greater than expected based on similar water volume differences from adjacent samples. Laboratory personnel may then elect to make judicious corrections to the initial water volumes so that reasonable data may be obtained. The reporting of obviously anomalous data is not in the best interest of the end user. Such corrected data should be flagged and a footnote inserted.

Crushed core material is vulnerable to fluid loss due to evaporation, particularly of water, due to the high surface area of the crushed core. Exposure time for the crushed core material should be minimized.

Note: Since handling mercury is required for this fluid saturation method all existing safety and health standards should be followed. Consult relevant environmental regulations and local laws.

4.2.1.4 Calculations

The corrected oil (See Figure 4-2) and pore water collected for each sample along with the gas volume may be expressed as a percentage of bulk volume as follows:

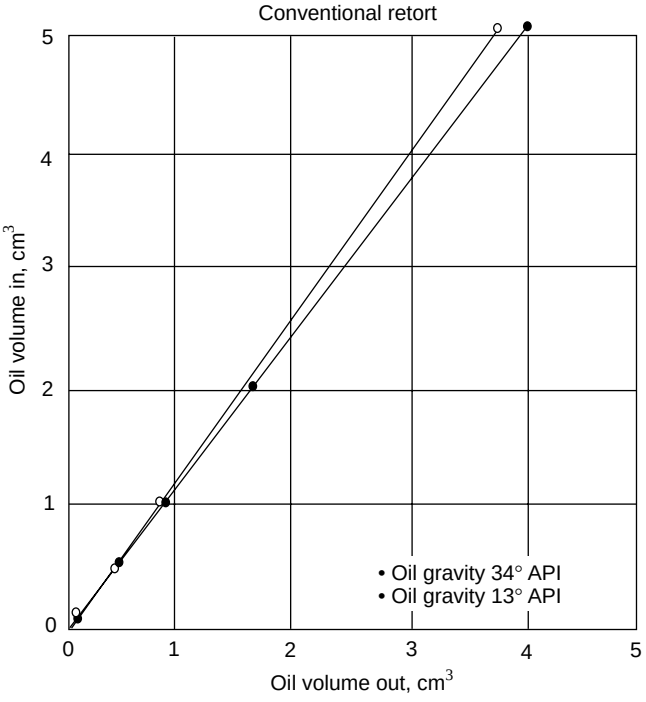


Figure 4-2—Retort Oil Correction Curve

$$NDHg = WtHg/BvHg$$

$$BvRet = WtRet/NDHg$$

$$GB = (HgInj \times 100)/BvHg$$

$$OB = (OilVol \times 100)/BvRet$$

$$WB = (WatVol \times 100)/BvRet$$

$$GS = (GB \times 100)/(GB + OB + WB)$$

$$OS = (OB \times 100)/(GB + OB + WB)$$

$$WS = (WB \times 100)/(GB + OB + WB)$$

Where:

NDHg = natural density of mercury pump sample, g/cm^3.
WtHg = initial weight of mercury pump sample, g.
BvHg = bulk volume of the mercury pump sample, cm^3.
WtRet = weight of the retort sample, g.
HgInj = volume of injected mercury corrected by pump calibration factors, cm^3.
OilVol = volume of recovered oil corrected with oil calibration curves, cm^3.
WatVol = volume of initial water recovered, cm^3.
BvRet = bulk volume of the crushed retort sample, cm^3.
GB = gas saturation as a percentage of bulk volume.
OB = oil saturation as a percentage of bulk volume.
WB = water saturation as a percentage of bulk volume.
GS = gas saturation as a percentage of pore volume.
OS = oil saturation as a percentage of pore volume.
WS = water saturation as a percentage of pore volume.

Grain density may be calculated from the data obtained during the testing procedure (Hensel, 1982[1]). These grain density data are of primary value as a means of quality control to ensure that erroneous readings have not been taken, that all fluids have been determined properly, and no calculation errors have been made. This is not the most accurate means of determining the true grain density of the rock material; however, the values obtained should be reasonably representative of the lithology being tested. Thus, the grain density calculation may be considered as a qualitative data evaluation tool.

4.2.1.5 Advantages

Advantages of the basic retort method include:

a. Fluids are collected from relatively large samples ensuring better representation of the lithology and increasing the potential precision of all measurements.
b. The analytical process is rapid and provides required saturation data within hours. Many samples can be evaluated in a short time and in a cost-effective manner, if sufficient retorts and ovens are available.
c. Fluid volumes are measured directly as opposed to other analytical techniques that rely on combinations of fluid volumes and total weight loss.

d. Grain loss, commonly associated with friable sandstones and some carbonates, does not affect the fluid saturation data.

4.2.1.6 Limitations

Limitations of the basic retort method include:

a. Water saturation (and porosity) may be too high if samples contain large amounts of montmorillonite or gypsum that break down at high temperature. This in turn will result in oil saturations that are too low since the volume of oil would be expressed as a percentage of a pore volume that is too high.

b. Oil saturation (and porosity) may be too high if the sample (e.g., some shales) contains solid hydrocarbons that break down at high temperature. This in turn will result in water saturations that are too low since the pore water is expressed as a percentage of a pore volume that is too high.

c. Oil volume correction curves are required and it may not be feasible to obtain the corrections for the oil in question. It may be necessary to use generalized correction curves, based on reported or assumed API gravities.

d. A second piece of core material is required to determine bulk volume, natural density, and gas-filled pore volume. This sample must be lithologically similar to the sample that is crushed for the oil and water data.

e. The distilled liquids may form emulsions.

4.2.1.7 Accuracy/Precision

Accuracy/precision of the basic retort method is:

a. By using an oil volume correction curve, the accuracy of the oil value obtained is within ±5%, and reproducibility is within ±2.5% of measured volumes.

b. The accuracy of the water volumes obtained by this method is ±2.5% of measured volumes.

4.2.1.8 Calibration

Calibration issues for the basic retort method include:

a. Graduated glassware should be checked for accuracy by using a microburette with water. The cleanliness of the glassware could impact readings due to a lack of complete wetting and indistinct meniscus.

b. The mercury pump used for bulk volumes should be calibrated with steel blanks of known volumes. Additionally a pump/system factor should be determined at the pressure used to determine the gas-filled pore volume. Correction curves and/or equations should be used in both instances.

c. All balances should undergo routine maintenance.

d. Retorts should be regularly subjected to vacuum tests to ensure the seals are performing properly.

4.2.2 Percussion Sidewall Cores

4.2.2.1 Principles of Analysis

The method is a modification of the basic retort procedure (see 4.2.1). The main difference is that gas, oil, and water volume measurements are made on the same sample as opposed to the basic procedure where adjacent core pieces are required. Smaller samples are involved in the sidewall procedure and therefore the equipment is scaled down to provide comparable accuracy. This method may be applied to rotary sidewall cores, but damages the samples for further testing.

4.2.2.2 Apparatus

Suitable apparatus for retorting of sidewall cores include:

a. Stainless steel retorts—See Figure 4-3. The overall design is similar to the retorts used in the basic procedure, i.e., the retort is composed of a cup section that holds the sample, a gasket/lid arrangement, and a long condensing tube. The cup section is greatly reduced in size. A bottle cap may be substituted for a screw-on lid, such cap being affixed with a standard capping device. The condensing tube is shorter and its outside and inside diameters smaller than the tubes used for the large retorts.

b. Oven—The sidewall oven is composed of the same components as the oven used in the basic procedure (see 4.2.1.2, b). The sidewall oven is tailored to accommodate multiple sidewall retorts.

c. Calibrated glass receiving tubes—The 15-ml centrifuge tube is not recommended, rather the receiving tube should be designed to accommodate very small fluid volumes (e.g., a maximum of 5 ml). The graduations should be such that readings/interpolations to the nearest 0.01 ml are possible.

d. Mercury pump—The same mercury pump used for the Basic Procedure (see 4.2.1.2, g) may be used, however a small sample chamber will enhance the bulk volume and gas space measurements.

e. An apparatus capable of piercing the jar lid is optional (see 4.2.2.3).

f. A hot wire gas detector capable of a qualitative measurement of combustible gases is optional (see 4.2.2.3).

g. A gas chromatograph is optional (see 4.2.2.3).

4.2.2.3 Procedures/Precautions

4.2.2.3.1 Procedures

Samples should be received in the laboratory sealed in glass jars with screw-top lids. The samples should be arranged in descending depth order. Prior to opening the jar many laboratories employ a procedure that involves puncturing the jar lid and making a measurement of the contained gases. This procedure will not be discussed in detail in this section, however this optional procedure measures combustible gases with

a hot wire detection device and some laboratories determine the head space gas composition by chromatography.

The sample recovery (sample length), while it is still in the jar, is noted by calibration marks on the side of the jar. Each sample is then removed from the jar and is cleaned of drilling fluid solids by using a single edge razor blade or utility knife. A brief description of the sample is recorded including rock type, grain or crystalline size, siltiness, shaliness, fossils, color, intensity, and distribution of fluorescence, and other megascopic lithological features. The sample is weighed to the nearest 0.01 gram.

Some laboratories may divide the prepared sample so as to provide a portion for a permeability and/or other measurements, e.g., particle size distribution, x-ray diffraction, etc. Obviously this is limited to initial samples of large size, e.g., 2 inches (50.8 millimeters) in length. Even so the fluid saturation data are compromised by testing a smaller sample. Most laboratories employ an empirical approach to sidewall permeability data. Neither of these practices will be discussed in this section.

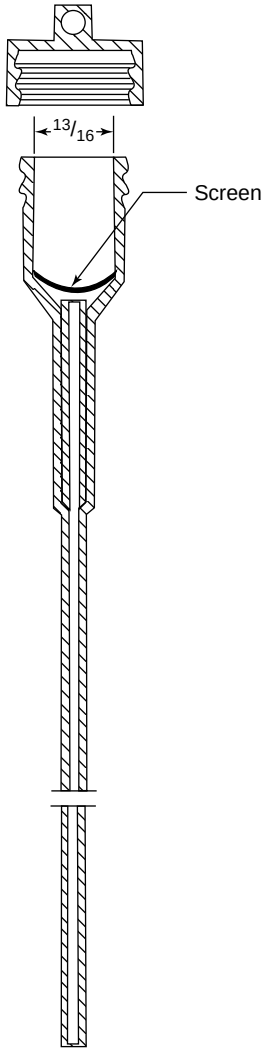


Figure 4-3—Retort Cup—Sidewall Sample

The sample is placed in the cell of a precalibrated mercury pump, and bulk volume is determined by mercury displacement (see 5.2.1). With the sample immersed in mercury, a valve is closed and the pressure raised to 750 psi (5.171 MPa). In tight (low permeability) samples, the pressure is raised to 1,000 psi (6.895 MPa). This procedure measures the gas-filled pore volume assuming that the compressibility of the water and/or oil present in the other pore spaces is minimal.

The sample is removed from the cell and broken into smaller pieces to observe the mercury penetration pattern. The penetration pattern is helpful in predicting probable production.

The retorting procedure described in 4.2.1.3 is followed, including the recording of initial (pore) and total water collected and the use of oil volume correction curves (see Figure 4-2).

4.2.2.3.2 Precautions

Sidewall core material is subject to fluid loss due to evaporation so exposure time to room conditions should be minimized.

Note: Since handling mercury is required with this fluid saturation method all existing safety and health standards should be followed. Consult relevant environmental regulations and local laws.

4.2.2.4 Calculations

The oil, pore water, and gas-filled pore volume may be expressed as percentage of bulk volume as follows:

$$GB = (HgInj \times 100)/BV$$

$$OB = (OilVol \times 100)/BV$$

$$WB = (WatVol \times 100)/BV$$

$$GS = (GB \times 100)/(GB + OB + WB)$$

$$OS = (OB \times 100)/(GB + OB + WB)$$

$$WS = (WB \times 100)/(GB + OB + WB)$$

Where:

BV = bulk volume of the sample, cm^3.
$HgInj$ = volume of injected mercury corrected by pump calibration factors, cm^3.
$OilVol$ = volume of recovered oil corrected with oil calibration curves, cm^3.
$WatVol$ = volume of initial recovered water, cm^3.
GB = gas saturation as a percentage of bulk volume.
OB = oil saturation as a percentage of bulk volume.
WB = water saturation as a percentage of bulk volume.
GS = gas saturation as a percentage of pore volume.
OS = oil saturation as a percentage of pore volume.
WS = water saturation as a percentage of pore volume.

Grain density may be calculated from the data obtained during the testing by slight modifications to the method proposed by Hensel, 1982.[1]

The bulk density, sometimes used in various laboratories, may be calculated by dividing the initial (whole) sample weight by the bulk volume.

4.2.2.5 Advantages

Advantages of the sidewall retort procedure include:

a. Gas, oil, and water measurements are made using one sample.
b. The method is relatively rapid.
c. This is a "direct" measurement method where gas, oil, and water measurements are made independently.

4.2.2.6 Limitations

Limitations of the sidewall retort procedure include:

a. The distilled liquids may form emulsions.
b. Oil and water calibration is required.
c. Volumes of oil <0.1 ml are difficult to determine accurately.
d. For very friable or unconsolidated sands the measured gas-filled pore volume may be high due to sample expansion when overburden pressure is released.
e. Samples must be handled carefully to minimize loss of liquids, especially water. In a low humidity atmosphere, water loss may be substantial.
f. Porosity and fluid saturation values can be in error if gypsum or hydratable clays (e.g., montmorillonite) are present and precautions are not taken in recording only "pore" water.
g. Calibration of all equipment is critical because samples are typically small and may contain equally small amounts of oil and water. Minute differences in recorded fluid volumes may result in significant ranges of saturation and porosity values.

4.2.2.7 Accuracy/Precision

Accuracy/precision for the sidewall retort procedure is:

a. The method's accuracy is strongly dependent on sample size, especially when determining oil volumes.
b. Analytical discretion should be used on testing samples with an initial weight of less than 3 grams.
c. By using oil volume correction curves, the precision of the oil value obtained is within ±5 percent of the measured volumes.
d. The precision of the water volumes is within ±3 percent of the measured volumes.

4.2.2.8 Calibration

Refer to 4.2.1.8 of the basic retort procedure.

4.3 DISTILLATION EXTRACTION METHOD (DEAN STARK)

4.3.1 Plug Samples

4.3.1.1 Principles of Analysis

This procedure is appropriate for plug samples and for rotary sidewall cores. The distillation extraction (Dean-Stark) method of determining fluid saturation depends upon the distillation of the water fraction, and the solvent extraction of the oil fraction from the sample. The sample is weighed and the water fraction is vaporized by boiling solvent. The water is condensed and collected in a calibrated receiver. Vaporized solvent also condenses, soaks the sample, and extracts the oil. The sample is oven dried and weighed. The oil content is determined by gravimetric difference.

4.3.1.2 Apparatus and Reagents

The following apparatus is suitable for this method and is illustrated in Figure 4-4. The apparatus should be set up in a fume hood or room suitable for exhausting solvent vapors.

a. General—The apparatus consists of an electric mantle or heating device with thermostatic controls. The distillation/extraction unit for one sample consists of a boiling flask, thimble, trap or calibrated sidearm, and condenser.
b. Flask—The flask is wide-mouthed, long-necked, and may contain indentations at the base of the neck to support the extraction thimble.
c. Trap—The trap or sidearm has a graduated section marked in 0.1 ml divisions. The graduated trap can be made smaller or larger to accommodate samples with very small or very large water volumes. Attached to the graduated section is a glass tube or arm that is bent at a right angle with a glass joint on the end. The glass joint has a drip tip molded on it and the opening in the tip should be designed so that the dripping solvent is directed in the center of the flask to ensure soaking of the core plug below. A modification to the trap (Figure 4-5) enables the water to be determined gravimetrically.
d. Condenser—The condenser is a water-cooled, reflux, glass-tube type, with a condenser jacket approximately 11.8 inches (300 millimeters) long and an inner tube (see Figure 4-4). The bottom is fitted with a drip tip and the inside tube should be vertical to reduce the difficulty of removing water from the surfaces of the condenser and trap.
e. Desiccant holder—A glass tube desiccant holder is fitted to the top of the condenser when samples are being extracted and a rubber stopper is used when the apparatus is idle.
f. Extraction thimbles—A glass thimble with a fritted glass bottom is recommended for holding a plug sample. Alternatively, a cellulose thimble may be used. However, types of thimble materials other than glass may cause errors in weighing because of absorption of atmospheric water. Also, thimbles made of glass allow viewing of the plug to ensure

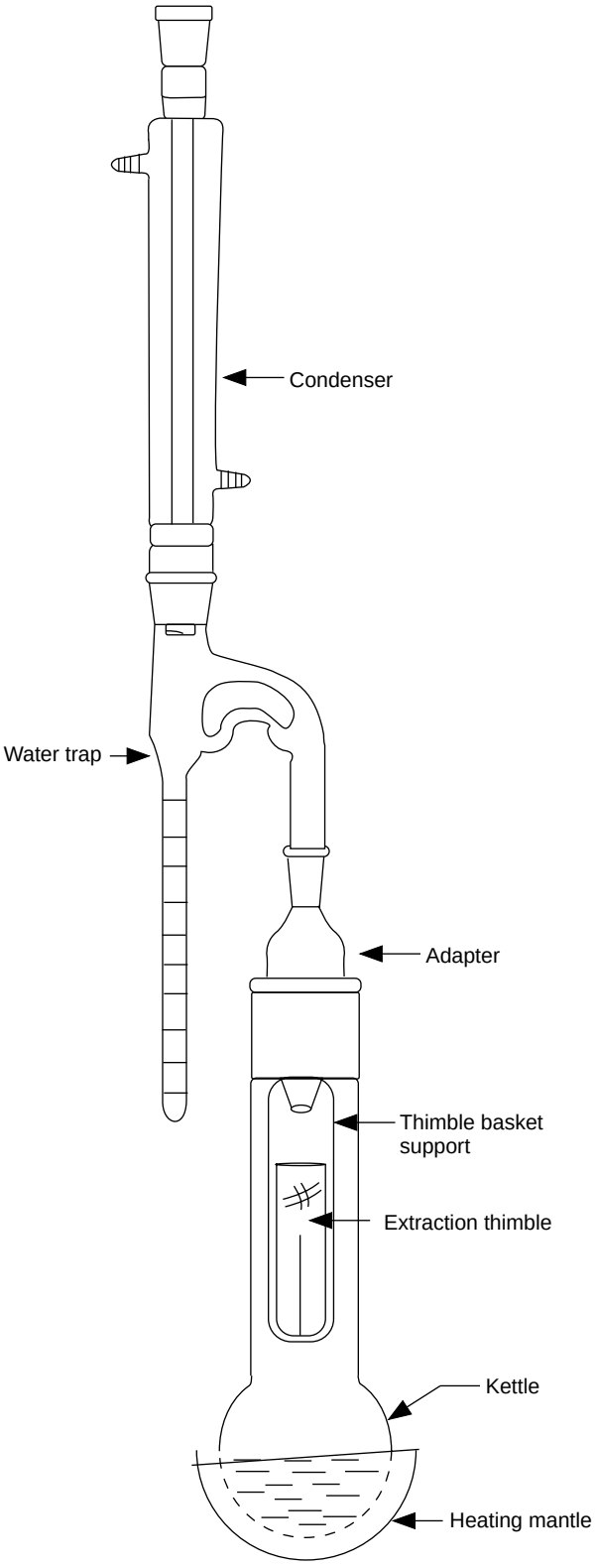


Figure 4-4—Dean-Stark Apparatus for Volumetric Determination of Water

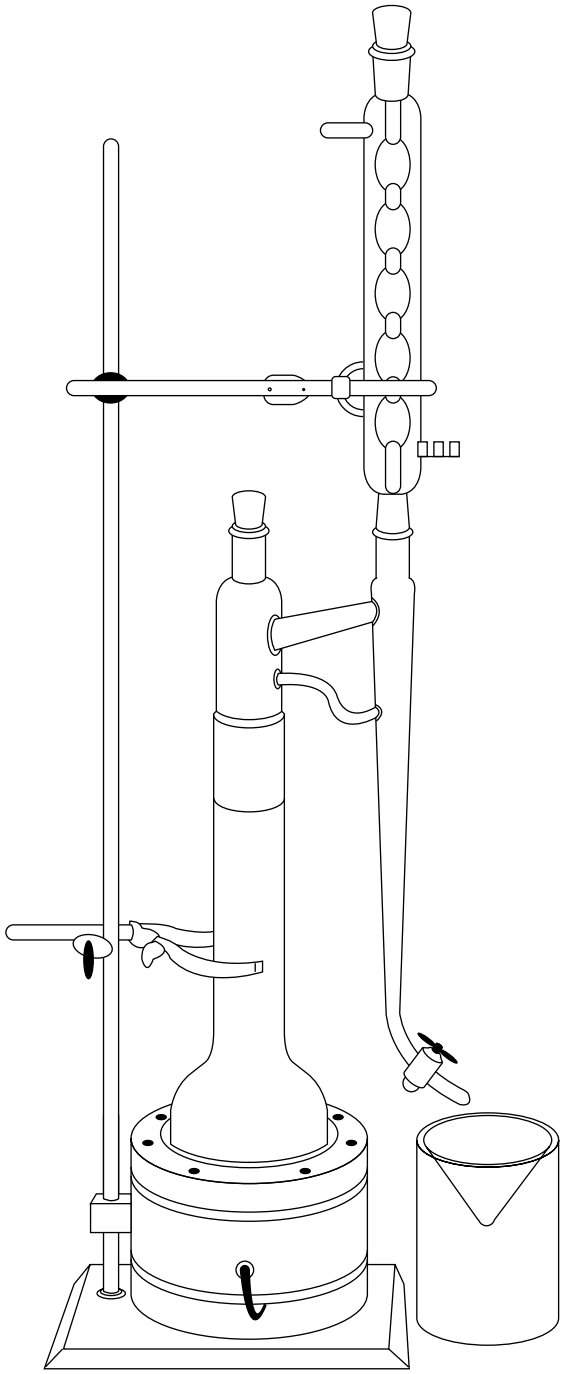


Figure 4-5—Dean-Stark Extraction Apparatus for Gravimetric Determination of Water

that the solvent is dripping and soaking the sample for thorough extraction. A small piece of glass wool may be used to cover the core plug to prevent erosion by the solvent reflux from the condenser.

g. Extraction cup—Cups fitted with siphons can be used to hold the thimbles and allow alternating immersion/drainage cycles. This can increase the efficiency of the extraction process.

h. Drying oven—A convection or vacuum oven may be used having a temperature control of ±2°C. An explosion-proof model is preferred, but non-explosion proof models can be used. For samples containing large amounts of montmorillonite and other clays, a humidity oven can be used to help preserve a single or double water layer to better approximate the expected hydration state of the clays in the reservoir. (Humidity levels during drying procedures are important in maintaining proper waters of hydration.)

i. Boiling chips—Glass beads or small chips of alundum material may be placed in the bottom of the boiling flask. These aid in reducing the tendency of the solvent to "bump" as it becomes more saturated with oil. If bumping or super heating occurs it creates an undesirable boiling process, contaminating the sample and extending the time required for the initial phase of the distillation extraction process.

j. Solvent—Toluene (reagent grade) or other suitable solvent.

4.3.1.3 Procedures/Precautions

4.3.1.3.1 Procedures

The samples are prepared using a fluid that is compatible with the fluid used to cut the whole core or drilled sidewall cores in the well. This ensures the preservation of residual oil or water saturation depending on the use of oil or water-based fluid. In the case of frozen oil sands samples, liquid nitrogen should be used. The excess fluid remaining on the sample after the trimming of the ends should be wiped off using a material that will remove the surface fluid but will not pull fluid from the interior in a sponge-like manner. This process is best described as a squeegee action. The sample's surface should appear dry (with a dull luster) and not shiny with excess fluid. The thimble/plug sample should be weighed to the nearest milligram (0.001 gram) on an analytical balance. The process should be accomplished quickly to minimize evaporation of the fluids in the sample. After final weighing, the samples and/or thimbles should be placed in the apparatus immediately, or should be stored in a container to prevent evaporation until they are placed in the apparatus. This storage period should be kept to a minimum. The necessity for weighing each component is to ensure complete accountability for weight loss. When extraction thimbles are used they should be at room temperature and thoroughly dry.

The samples are extracted with a solvent that does not donate to or absorb any of the collected water. Solvent conditioning can be provided by adding at least 1 percent water by volume to the solvent and pre-boiling the solvent until the water concentration is stable. Some laboratories add 15 percent water by volume to the solvent. Water should be in excess of the amount required to bring common solvents into equilibrium when the water is boiled away.

The desiccant trap ensures that atmospheric moisture (humidity) does not affect the water collected.

Water can adhere to the condenser and to the side of the trap. This fluid may cause appreciable error, particularly for a plug sample of low porosity. The adhered water is dislodged with a stiff solvent stream from a laboratory squirt bottle or a small gauge wire with a very small loop at the end. The wire is used to physically dislodge water particles that are not removed by the stream of solvent. Detergent is sometimes used to flatten the solvent/water interface for improved accuracy; but the use of detergent is not generally recommended because the effects of three phase fluid interaction are unpredictable and the wettability of the sample will be altered.

The distillation/extraction process continues for a minimum of 48 hours. Water levels should be monitored daily and the process stopped only when there is no change in volume of water recovered in 24 hours. Longer times may be required depending on the size of the plug sample and its permeability. This is to ensure that the solvent used has extracted all the oil possible from the plug material. Where heavy oil (low-gravity, high-asphaltene) is involved, another solvent type (see 3.1) is required to thoroughly clean the plug. A complete record of the volumes of water collected should be noted. The rate of extraction and efficiency may be increased by using extraction cups equipped with siphons. Alternative methods that speed extraction involve using the described apparatus until the amount of water collected is at a stable end point, and then doing one of the following with the samples and the thimbles:

a. Place the samples in a vapor phase unit to complete the oil extraction.

b. Place the samples in a CO_2-toluene pressure core cleaner to complete the oil extraction. This should be restricted to competent, low-permeability samples that will not be physically altered by this process.

c. Place the sample in a Soxhlet extractor for alternating immersion and drainage type oil extraction.

d. Place the sample in a flow-through oil extraction apparatus.

e. Alternate between solvent types (e.g., toluene and methanol).

The extraction efficiency is evaluated by treating the sample with chlorothene under an ultraviolet light source to determine if there is any remaining oil, that fluoresces, or by measuring the grain density of the sample. If the grain density is lower than anticipated for the rock type, the sample may need further extraction.

The sample/thimble should then be dried to a stable weight. Samples with high saturations of flammable solvents should be dried in an explosion-proof convection or vacuum oven, unless the excess solvent is allowed to evaporate before being put into the oven. This avoids a potential explosion or fire situation. When the sample/thimble is dried, it should be allowed to cool to room temperature in a sealed container such as a desiccator and the weighed. An oven that adds moisture to the sample should not be used for this step. However, when porosity and permeability will be measured, samples containing large amounts of montmorillonite and other clays may need to be dried in a humidity oven to preserve the hydrated state found in the reservoir. (The humidity level in the oven is important in maintaining proper waters of hydration for measuring permeability and porosity.)

Errors occur by not accounting for a chipped thimble, the core losing small particles, and/or the loss on the sample surface of precipitated salt from concentrated brines. In the case where heavy salt precipitation occurs, the salt is removed from the sample by extraction with methanol or similar solvent. The sample is then dried and weighed. The weight of water collected in the trap is subtracted from the total liquid weight loss to determine the oil weight extracted from the plug sample.

Proper water flow in the condenser will maintain a condenser temperature sufficiently cool so that the vapors condense in the bottom third of the condenser column.

4.3.1.3.2 Precautions

Local regulations regarding the safe use of reagents should be followed. In general, however, the following safety considerations should be noted:

a. Oil may contain compounds exhibiting carcinogenic properties. It may be flammable.

b. Toluene is moderately toxic by skin absorption and inhalation. It possesses irritant and anesthetic properties and is highly flammable.

c. Many other solvents, while effective for the oil removal process, may be dangerous and toxic. Consult relevant environmental regulations and local laws governing the use of these solvents.

d. Solvent vapors should condense in the lower third of the water-cooled heat exchanger on top of the water trap.

e. The analyst should be aware of the change in solvent boiling point with altitude or the change in the water boiling point due to salt in solution. The solvent's boiling point should be checked to ensure an adequate temperature for water distillation. When KCl drilling fluids are used, the filtrate water will have salt concentration on the order of 300,000 ppm, and, consequently, boil at a much higher temperature than fresher water. The use of orthoxylene is suggested as a substitute for toluene in these cases.

4.3.1.4 Calculations

The following calculations are appropriate for this method:

$$\text{Weight \% Water (Gravimetric)} = \frac{(\text{Weight of Water}) \times 100}{(\text{Initial Sample Weight})} \quad (1)$$

or

$$\text{Weight \% Water (Volumetric)} = \frac{(\text{Volume of Water}) \times (\text{Density of Water}) \times 100)}{\text{Initial Sample Weight}}$$

$$\text{Weight \% Solids (Gravimetric)} = \frac{\text{Dry Weight of Sample} \times 100}{\text{Initial Sample Weight}} \quad (2)$$

$$\text{Weight \% Oil (Gravimetric)} = \frac{(\text{Initial Weight} - \text{Dry Weight} - \text{Weight of Water}) \times 100}{\text{Initial Weight of Sample}} \quad (3)$$

The saturations are normally expressed as percentages of the sample pore space. Therefore, the sample porosity, water density, and oil density are required. If the connate water is a highly concentrated salt solution, the water density must be corrected for the salt in solution.

The following calculations apply:

$$\text{\% Water} = \frac{\text{Volume of Water} \times 100}{\text{Pore Volume}} \quad (4)$$

$$\text{\% Oil} = \frac{(\text{Weight of Oil})/(\text{Density of Oil}) \times 100}{\text{Pore Volume}} \quad (5)$$

The liquid content of the sample is reported to the nearest 0.1 percent of the pore space; e.g., 22.1 percent oil and 43.7 percent water.

Knowing the brine salinity and density, one can calculate volume of brine that was in the core from the volume of distilled water recovered as follows:

$$Vbr = [(Vw)(\rho_w)/\rho_b]\,[1{,}000{,}000/(1{,}000{,}000 - Cs)] \quad (6)$$

Where:

Vbr = volume of brine corresponding to the volume of distilled water collected from the plug, cm^3.

Vw = volume of distilled water collected from the plug (e.g., Dean-Stark), cm^3.

ρ_w = density of distilled water, g/cm^3.

ρ_b = density of lease brine having a concentration C_s of salt, g/cm^3.

C_s = concentration of dissolved salts in lease brine, =

$$\frac{(1{,}000{,}000)(\text{Weight of Salt})}{(\text{Weight of Brine})}\text{ppm}$$

4.3.1.5 Advantages

Advantages of this method include:

a. Water volume determinations are generally very accurate.

b. Typically, the sample is not damaged and can be used for further testing. However, the wettability may be altered and certain clays (e.g., montmorillonite) or gypsum may also be subject to change.

c. Relatively low temperatures [212°F (100°C)] are used; hence, little if any of the clay hydroxyl waters are removed.

d. The procedure is simple and requires little attention during distillation.

4.3.1.6 Limitations

Limitations of this method include:

a. Inaccuracies arise in the water determination due to the following:

1. Atmospheric water condenses in the condenser when atmospheric humidity is high. Desiccant tubes can be used to avoid the problem.

2. Water evaporates from the sample at room temperature when it is not immediately set up in the extractor with the condenser water circulating.

3. Water droplets stick to unclean glass in the sidearm or condenser.

4. Salt may precipitate inside the sample from connate brines (saline interstitial water). This can result in significant changes in porosity and/or permeability. Salt can be removed from the sample with methanol extraction.

5. Correction for the higher density of salt water is required when the total solids concentration exceeds 20,000 ppm (see 4.3.1.4, Equation 5).

6. Incomplete drying of solvents.

7. Loss of water due to the joints of the extraction flask not being vapor tight, or from too high an extraction temperature, or from insufficient water flow in the condenser.

8. The density of air must be considered for buoyancy only when the sample is weighed to the nearest 0.1 mg.

9. Extraction time may be insufficient.

10. Water saturation may be too high if samples contain large amounts of gypsum (see 4.8) or montmorillonite clays (water of hydration). Permeability and porosity values can also be altered if waters of hydration that are present in the reservoir are removed during extraction and drying procedures (humidity oven drying may be preferred).

11. If the true oil density is not known, error is introduced into the calculation of oil saturation because an oil density value must be assumed.

b. Oil volumes are not found directly and may be inaccurate due to the following:

1. Additional water collected or lost from the sample as mentioned above.

2. Loss of solids.

3. Incomplete cleaning of oil.

4. Drying at a higher temperature than the extraction temperature may remove additional water of hydration and overstate the oil volume.

c. Rock wettability may be altered.

d. The clay fabric may be altered, that may result in inaccurate permeability measurements.

e. There is no check on the analysis accuracy.

4.3.1.7 Accuracy/Precision

There are no documented standards and the accuracy of the methods cannot be assessed. However, with the calibration procedures noted in 4.3.1.8, water volume reproducibility can be evaluated. Similar procedures assess oil volume accuracy. On relatively small samples or samples containing high gas saturation with residual volumes of oil and water, the percent error for liquid saturations may be ±50 percent of values measured. Percent error will be significantly less as liquid volumes increase.

4.3.1.8 Calibration

The water measurement accuracy should be checked regularly to be sure no bias affects the results.

a. For gravimetric determination—Known weights of water added to the extractors are plotted against the weights of water recovered by extraction under conditions identical to those used in extracting an oil sand sample. The water correction factors may change due to the efficiency of the condensers used in the apparatus. Typical values are listed below:

$$\text{Corrected Weight Water} = (\text{Weight Water} \times a) + b$$

Where: a and b, respectively, are the slope and intercept of the calibration equation, that is, $a = 1.003, \pm 0.001$; $b = 0.090, \pm 0.009$.

b. For volumetric determination—A calibrated buret is used to deliver known volumes of water into the water trap. The traps should have a maximum scale error of 0.02 ml. Volume correction factors can be calculated and applied, if necessary.

4.3.2 Full Diameter Cores

4.3.2.1 Principles of Analysis

The distillation extraction method of determining fluid saturations for full diameter samples is governed by the same principles and procedures as those for plug samples (see 4.3.1.1). Differences in the procedure and apparatus are discussed below.

4.3.2.2 Apparatus

The following apparatus are appropriate for this method:

a. Flask—The flask used to accommodate the full diameter core sample must be larger than the plug analysis flask with a larger solvent volume for oil removal.

b. Trap—The trap or sidearm has to be large enough to accommodate the volumes of water from large samples. The graduated section of the trap should be marked in 0.1 ml divisions.

c. Extraction core protector—Extraction thimbles are not generally used for full diameter samples. Instead a stocking material made from unbleached cotton can be used to prevent the loss of fines from the full diameter core. Thimbles normally used in plug core analysis are not manufactured in sizes that fit the full diameter core.

4.3.2.3 Procedures

The procedures are the same as those for plug samples with the exception that sample weights should be made to the nearest 0.1 gram. Also, the cleaning time of 48 hours may have to be extended in order to fully clean the larger volume samples. Longer times and supplemental cleaning are frequently required.

4.3.2.4 Calculations

See equations in 4.3.1.4.

4.3.2.5 Advantages

Refer to 4.3.1.5. There are additional advantages when performing this test on full diameter cores, as follows:

a. A high precision analytical balance is not required as the weights need only to be determined to the nearest 0.1 gram.

b. Grain loss during the handling and testing procedures is not as critical as with plugs, but still should be minimized.

c. Collected water volumes are quite large as compared to those associated with plug analyses, therefore the water readings need to be recorded only to the nearest 0.1 ml.

d. As the oil volumes are determined by gravimetric difference the final values are not impacted to the same degree by small measurement errors as those determined on plugs, except at very low oil saturations.

4.3.2.6 Limitations

There are additional limitations when analyzing full diameter cores as follows:

a. Larger, more costly equipment is required, e.g., glassware, ovens, cut-off saws.

b. Laboratory space requirements are greater.

c. Large volumes of solvents are used thereby requiring a higher investment in same. Storage and/or disposal logistics are more complex.

d. Longer distillation and core cleaning times are required thereby decreasing data reporting timeliness.

e. Health and safety standards should be carefully monitored and observed due to the large volumes of fumes and solvents that can be involved.

4.3.2.7 Accuracy/Precision

Refer to 4.3.1.7.

4.3.2.8 Calibration

Refer to 4.3.1.8.

4.3.3 Pressure-Retained Core Analysis

4.3.3.1 Introduction

The objective of pressure-retained core analysis is to provide fluid saturation data on cores for which fluid expulsion has been minimized during core barrel retrieval by preventing pressure depletion from bottom hole to surface conditions. A ball valve seals the core barrel and prevents the natural pressure drop as the barrel is brought to the surface. Additionally, the barrel is designed to allow preset pressure maintenance during retrieval with a pressure supply that compensates for pressure loss due to a lower surface temperature.

Flushing of the core with drilling fluid filtrate before the core enters the core barrel is minimized with special, low-water-loss drilling fluid, a high coring penetration rate, a low drilling fluid overbalance, and a special core bit design. In addition, prior to coring, the inner barrel is generally filled with a low-invasion gel material. This material minimizes further drilling fluid filtrate invasion via imbibition by displacing drilling fluid from the core surface as the core enters the barrel.

Fluid saturations are the amounts of oil and water present in the core at laboratory conditions. Alteration of original fluid saturations by flushing can only be qualitatively defined. Any drilling fluid filtrate invasion alters saturations from in situ values unless the in situ saturations are at waterflood residuals. In under-pressured reservoirs, filtrate invasion is minimized by using foam as the drilling fluid. With the foregoing precautions, saturations under these conditions can reflect saturations near in situ values.

Rig site handling procedures are addressed in 2.2.5.

4.3.3.2 Principles of Analysis

The cores are kept frozen with dry ice until the laboratory analysis is initiated. This reduces pore pressure, freezes water, immobilizes oil, and traps gas that does not freeze or liquefy at dry ice temperature.

As the core is allowed to thaw in the gas collection cells, gas comes out of solution and expels both oil and water that are captured in a receiving tube at the bottom the cell. Gas is collected in the void space within the cell. This fluid volume does not necessarily reflect the amount of fluid produced by pressure depletion of a conventional core since both different pressure and different temperature conditions exist in the reservoir.

Since oil volumes are determined from the distillation extraction and gas driven solvent extraction steps of the analysis, grain loss must be kept to a minimum for maximum accuracy.

If no free gas is present in the cored zone and the core is captured above the bubble point of the oil, the total liquid saturation should be equal to the pore space after a formation volume factor is applied to the oil. Saturations should not vary more than ±5 percent.

Filtrate invasion analysis allows assessment of the fluid saturation alteration from in situ values. If available, relative permeability fractional flow curves will aid in this assessment. Detailed discussion of invasion principles is provided in 4.3.7.

4.3.3.3 Apparatus

The following equipment is necessary for the analysis of pressure-retained core. These items are considered exclusive to the analysis of pressure-retained cores. Standard core analysis items used in the analysis are not included.

a. Core storage boxes—Boxes used for storage of the frozen core until analysis is initiated. The boxes should be of sufficient size to hold 5-foot (1.52-meter) sections of core in its inner barrel and enough dry ice to maintain the core in a frozen state. These boxes should be insulated to minimize dry ice sublimation.

b. Dry ice—Used to maintain the core in a frozen state until analysis is initiated.

c. Liquid nitrogen—Used for milling of core barrels and during sample preparation such as core cleaning, drilling plugs, and full diameter sample facing.

d. Milling machine—Used to cut diametrically opposed grooves down the length of the inner barrel to facilitate frozen core removal.

e. Two-liter dewar flask—Used for periodic dipping of samples into liquid nitrogen during the cleaning process to aid in cleaning and to keep the core frozen.

f. Drilling fluid or low invasion gel removal tools—Various tools including carpet knife, insulated gloves, small hammer, etc.

g. Gas collection cells—Cell equipped with a pressure gauge to determine gas volumes bled from the core and core barrel, and to allow for chromatographic gas analysis.

h. Gas collection cylinders (250 cm^3)—Used for sampling of cell gas for chromatographic analysis.

i. Centrifuge—Used for centrifuging gas collection cell receiving tubes to obtain good oil/water/solid separation.

j. Heat lamp—Used for gas mixing in the gas collection cell prior to sampling.

k. Portable vacuum pump—Used for pulling a vacuum on the gas collection cell and 250 cm^3 cylinders prior to gas collection.

l. Stainless steel tubes [3-inch (76.2-millimeter) diameter with screen bottoms]—The core samples are placed in these tubes, thereby preventing grain loss from samples during the gas collection and distillation extraction steps of analysis.

m. Surgical stocking material—Used to encapsulate core samples during CO^2 extraction process to minimize grain loss.

4.3.3.4 Procedures

4.3.3.4.1 Core Preparation Procedures

Core preparation procedures include:

a. Core samples, encased in steel tubing and frozen in chests of dry ice, are brought from the wellsite to the laboratory.

b. Each length of tubing-encased frozen core is placed into a dry ice filled trough attached to a milling machine. Two diametrically opposed grooves are milled down the length of the steel tubing to a depth slightly less than the wall thickness of the tubing. Liquid nitrogen is directed at the point of milling to ensure a tubing and core temperature at or below the dry ice temperature.

c. The tubing is separated into two halves and the frozen core is removed.

d. The low invasion gel that is frozen on the core is removed by scraping and brushing. The core sections are periodically dipped in liquid nitrogen to ensure that the core remains frozen.

e. Cores are visually examined for lithological characteristics and samples are selected for analysis.

f. The ends of each selected core segment are faced with a diamond saw using liquid nitrogen as a coolant. The faced frozen core segments are wrapped in plastic wrap and aluminum foil and stored under dry ice to await laboratory tests. The plastic wrap and foil protect the core from sublimation damage.

g. Care while milling open the core barrel ensures safe core handling. Placing a heavy mesh net over the core barrel during the milling process prevents analyst injury should the core barrel separate due to excessive internal pressure caused by drilling fluid expansion during freezing. Natural gas pockets where core is missing in the barrel may ignite in air. The carbon dioxide and nitrogen environment surrounding the core barrel during milling should prevent gas ignition.

4.3.3.4.2 Gas Collection Procedures

Gas collections procedures include:

a. The faced frozen core sample is placed in a thin-walled, metal cylinder with a fine mesh screen in the bottom, weighed quickly, and placed in the gas collection system (see Figure 4-6).

b. The system is immediately assembled and evacuated for 45 seconds to remove air without pulling gas from the sample. The frozen core is then allowed to thaw to room temperature.

c. Water and oil expelled by the evolving gas are collected in a graduated receiving tube attached to the gas collection cell.

d. The evolved gas is collected in the gas collection cell. The system is equipped with a pressure gauge to allow pressure monitoring inside the cell. If the gas collection cell pressure exceeds 0 psig, an attached and previously evacuated secondary cell is then opened to allow collection of additional evolved gas.

e. Barometric pressure, room temperature, system pressure, and produced liquid volumes are recorded periodically. Thawing of the core is considered to be completed when consecutive readings indicate system equilibrium.

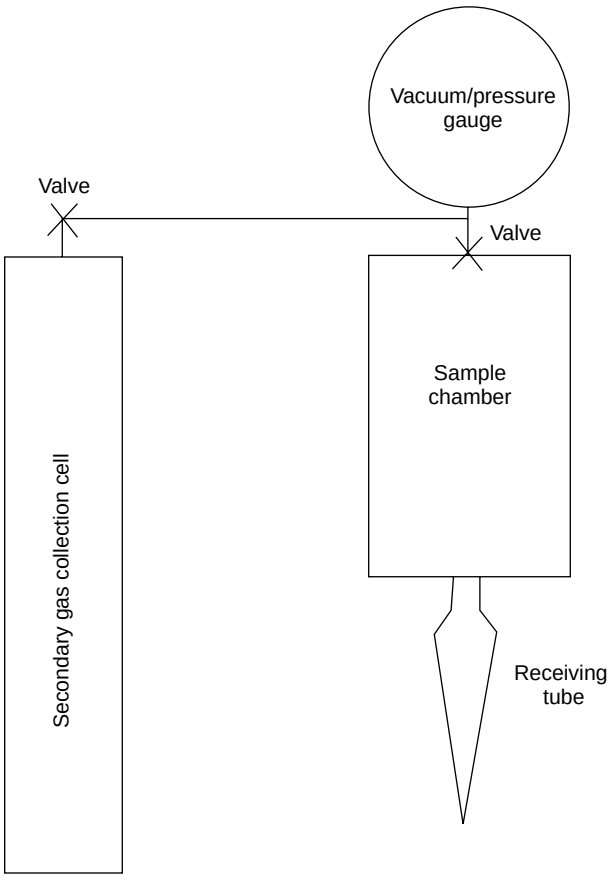


Figure 4-6—Gas Collection/Pressure Depletion System

f. Evolved gas samples may be collected in more than one cell. Gas samples are collected separately from the primary collection cell and, if used, the secondary gas collection cell. The gas samples are analyzed to determine gas gravity and component mole percent. Volumes of oil and water collected are measured. The chloride and bromide content of the water and the produced oil gravity are determined.

g. The sample and its cylinder are removed from the cell, weighed, and placed in a previously prepared distillation extraction apparatus.

4.3.3.4.3 Distillation Extraction (Dean-Stark) Procedures

Distillation extraction procedures include:

a. The sample is placed in the distillation extraction apparatus (see 4.3.1.2), the system is assembled, and heat is applied to distill the remaining water in the core and to extract the remaining oil.

b. When the distillation is complete, as determined by consecutive water readings, the water volume is recorded. The sample and thimble are then removed and placed in a vacuum oven at 240°F (116°C) to remove the extraction solvent. When drying is completed, the sample and thimble are removed from the oven, allowed to cool in the presence of a desiccant, and then reweighed.

c. The volume of additional oil extracted is determined gravimetrically, using an oil density determined from the oil produced in the gas collection step. The volume of water distilled is corrected to reflect the equivalent volume of water having the same salinity as the water recovered during the gas collection phase. If the water expelled during the gas collection is contaminated with drilling fluid filtrate, the water recovered during the gas collection phase may not have the same salinity as less contaminated formation water at the center of the core.

d. The core sample with all loose grains is then encased in a surgical stocking material to minimize grain loss prior to additional extraction procedures.

4.3.3.4.4 Gas-Driven Solvent Extraction and Saturation

Gas-driven solvent extraction and saturation procedures include:

a. The cores are weighed in the stocking material and placed in the gas-driven solvent extractor where they are subjected to further cleaning using carbon dioxide-charged toluene heated to 180°F (82°C). This phase is designed to remove any oil remaining. The weight loss resulting from this extraction process is the weight of additional oil removed that is converted into an oil volume using the previously calculated oil density. In actual practice, the total weight loss from distillation

extraction and gas-driven solvent extraction minus the weight of water recovered is used to determine oil volume.

b. The samples are then placed into a convection oven (or vacuum oven) and dried at 240°F (116°C) until weights stabilize. For samples containing large amounts of montmorillonite and other clays, a humidity oven can be used to help preserve a single or double water layer to better approximate expected clay/water interactions in the reservoir. (Humidity levels during drying are important in maintaining proper waters of hydration.)

c. Full diameter porosities along with horizontal air permeabilities are measured on each core segment. In the cases where more than one sample is combined in a foot of analysis, these values are weighted by representative portions of a foot and averaged to arrive at a mean value.

d. Liquid saturations at stock tank conditions are calculated using the measured total pore volume of all of the core segments comprising the sample and the total oil and water contents recovered from these segments. The volume of gas collected from the samples is then determined from the known volume of the gas collection system, corrected for the grain and tare volumes as well as the total liquid and salt contents. This gas volume is then further corrected to standard conditions.

4.3.3.5 Calculations

The following calculations are used for this method:

a. Gas volume at standard temperature and pressure and gas weight from gas collection:

$$\text{Gas Volume} = \frac{\text{Gas Collection Cell Volume} \times \text{Cell Pressure} \times 520 \times (1 - \text{Air Fraction})}{14.7 \times (460 + \text{Temperature})}$$

$$\text{Gas Weight} = \text{Gas Volume} \times 0.0012232 \times \text{Gas Gravity}$$

Where:

Gas collection cell volume is in ml.
Cell pressure is in psia.
Temperature is in degrees F.
Gas weight is in g.
Gas volume is in ml at STP.
Gas gravity = (density gas, g/ml)/(density of air, g/ml).
Density of air at STP = 0.0012232g/ml.
STP is standard temperature (0°C) and pressure (1 atmosphere).

The (1 – air fraction) term is a correction factor. Evacuation of the gas collection cells removes all but about 5 percent of the air in the system and small leaks may develop at the seal lid allowing air to be pulled into the system. Analysis of a gas sample drawn from the collection cell determines the amount of air present.

The gas weight is used in checking the material balance of the total weight loss as a result of extracting gas, oil, and water from the sample with the calculated weight loss.

b. Saturations:

$$\text{Oil Saturation} = \frac{(\text{gcc Oil Vol} + \text{Dist Ext Oil Vol} + \text{CO2 Ext Oil Vol}) \times 100}{\text{Pore Volume}}$$

$$\text{Water Saturation} = \frac{(\text{gcc Water Vol} + \text{Dist Ext Water Vol}) \times 100}{\text{Pore Volume}}$$

Where:

gcc Water Vol = gas collection water volume, cm^3.
gcc Oil Vol = gas collection oil volume, cm^3.
Oil saturation is given as percent pore volume.
Water saturation is given as percent pore volume.
All oil and water volumes are in ml.

c. Water volume from distillation extraction—See 4.3.2.

d. Pore volume and grain density correction—When salts are not leached from the core samples, corrections in pore volume and grain density are made based on water salinity determined from the water produced during gas collection and pressure depletion. Alternatively, methanol can be used to leach salt from the core.

$$\text{Pore Volume (corrected)} = \text{Pore Volume} + \frac{\text{Water Volume} \times \text{Salinity NaCl}}{C}$$

Where:

Pore volume is in ml.
Water volume from distillation extraction is in ml.
Salinity NaCl is in grams per ml of distilled water.
C = 2.165 g/ml (density of NaCl).

$$\text{Grain Density (corrected)} = \frac{\text{Sample Weight} - (\text{Water Volume} \times \text{Salinity NaCl})}{\text{Bulk Volume} - \text{Pore Volume (corrected)}}$$

Where:

Grain density is in g/ml.
Water volume from distillation extraction is in ml.
Salinity NaCl is in g/ml.
Bulk volume is in ml.
Pore volume is in ml.

4.3.3.6 Advantages/Limitations

Advantages/limitations of this method are:

a. Pressure-retained coring is an expensive, meticulous, and time-consuming operation. It requires a straight and clean hole containing a specially formulated drilling fluid or foam.

b. A laboratory including the necessary special equipment and experienced supervisors should be located as close as

possible to the well location since it may be difficult to safely transport a frozen core over great distances.

c. Pressure-retained core analysis provides fluid saturations more indicative of in situ saturation values than those values obtained by conventional core analysis. The ability to prevent fluid expulsion during core retrieval (pressure depletion) prevents saturation alteration during core retrieval. Refer to advantages/limitations for distillation extraction cleaning (see 4.3.1.5 and 4.3.1.6) to understand the impact on saturation determination.

d. Minimizing pore space flushing during coring is another factor that must be considered in order to obtain near in situ saturations. Pressure-retained coring procedures are designed to prevent alteration from depletion, but not from flushing. As a result, core saturations, from cores normally recovered in water base drilling fluids, are indicative of in situ oil saturation values only if filtrate flushing is minimal (see 4.3.7). If a large amount of flushing occurs, then the measured saturations are more indicative of residual values. This is important for the evaluation of tertiary recovery projects.

e. Flushing is minimized by minimizing drilling fluid overbalance during coring, through use of low fluid loss drilling fluid, and by penetrating the formation as rapidly as possible while maintaining good core recovery. In certain situations, i.e., depleted bottom-hole pressures, a stabilized-foam drilling fluid minimizes flushing. However, if the foam breaks down during coring, the core is subjected to a high surfactant fluid loss and appreciable flushing occurs.

f. The ability to capture oil and water in the core provides a distinct advantage over conventional and sponge core analysis. By capturing all water that existed in the core at the completion of coring, it is possible to determine the filtrate volume that displaced gas, oil, or water during coring operations. By knowing the measured saturations and the filtrate amount, saturation alteration during coring is qualitatively assessed by using the formation relative permeability fractional flow characteristics.

In comparison, conventional and sponge cores allow determination of only the residual core filtrate after coring because fluids are expelled from the core during retrieval. However, since most filtrate is located in the core's periphery, the measurement of filtrate is erroneously low. In a conventional core, the oil saturation is also reduced during pressure depletion, although this oil may be retained by the sponge in a sponge core and analyzed. If oil saturations are near residual, the k_g/k_o is such that usually very little oil is expelled by gas drive.

g. Since the gas is retained in the pressure-retained core, it is possible to measure gas volume as well as gas composition from a pressure-retained core. This is important when evaluating sweep efficiencies from gas injection. Residual saturations in highly-swept zones allow assessment of various petrophysical rock properties at residual saturations after gas injection.

h. Gas-oil ratios, when averaged over an interval, have agreed favorably with production measurements.

i. Oil gravity can be determined on a foot-by-foot basis throughout the cored interval to determine if oil gravity changes with depth. Gravity changes must be appreciable to be observed by this method.

j. The diameter of pressure cores is presently 2.5 to 3.75 inches (64.5 to 95.3 millimeters). The smaller diameter core increases the surface area to volume ratio of the sample. The effect of drilling fluid filtrate invasion and saturation alteration is greater than in larger-diameter core. As in any core analysis measurement, the degree of accuracy is decreased as sample size is reduced; therefore small measurement errors usually will have less impact on pressure-retained core analyses as the core diameter is increased.

4.3.3.7 Accuracy/Precision

Accuracy/precision of this method is:

a. Fluid saturations—Accuracy limits of fluid determination from each phase of analysis are as follows:

1. Gas collection: fluid volumes are ±0.5 ml; water volumes (distillation extraction) are ±0.5 ml.
2. Gas driven solvent extraction: oil volume is ±0.1 g or ±0.1 ml.

b. Gas volume—Gas volume is determined by Boyle's Law. Resulting gas volume is ±2% of actual volume. The variables in calculation of gas volumes have an accuracy as follows:

Cell volume	=	±5 ml.
Cell temperature	=	±0.5°F.
Cell pressure	=	±0.2 psia.
Air fraction	=	±0.5 mol %.

4.3.3.8 Calibration

Calibration issues for this method include:

a. Receiving tubes on gas collection cells and distillation extraction apparatus should be calibrated using deionized water on a scale reading to 0.01 grams. Weights at each interval are converted to volumes using the water density at the appropriate temperature. Volumes should be determined for each 10 ml reading on the receiving tubes. Interpolation is used to determine true volumes between the calibrated volumes.

b. Gas collection cell volumes should be calibrated using an accurate gasometer or meter. Each cell is evacuated and the volume determined. Repeatability of measured volumes shall be within 5 ml.

c. Balances should be checked at regular intervals for accuracy.

4.3.4 Sponge Core Analysis

4.3.4.1 Principles of Analysis

In sponge coring, the core enters a half-inch thick polyurethane or cellulose/polyurethane sponge sleeve inside an aluminum liner. As the core is brought to the surface, expanding gases displace crude oil that is captured by an oil-wet sponge sleeve (or water that is captured by a water-wet sponge sleeve).

The oil-wet sponge sleeve consists of polyurethane sponge with about 70 percent porosity that is easily compressed to almost no porosity. The sponge is saturated with brine prior to going into the well. If the formation brine properties are known, the brine used to saturate the sponge should match the salinity and density of the water in the formation to be cored. Any oil that comes out of the core will displace water in the sponge and will spread out in a thin layer over the walls of the sponge pores.

The water-wet sponge consists of a cellulose fiber mixed in with the polyurethane. The sponge is saturated with dry mineral oil prior to going into a well cored with an oil-based drilling fluid. The objective is to capture any water escaping the core as it is brought to the surface.

4.3.4.2 Apparatus

4.3.4.2.1 Equipment

High speed table saw with at least a 10-inch (25.4-centimeter) diameter carbide blade and 1.5 horsepower motor to open the aluminum core barrel.

4.3.4.2.1.1 Oil-Wet Sponge Analysis Equipment

The following additional equipment is required for oil-wet sponge analysis.

a. Container to hold sponge (large Soxhlet, metal can, etc.).
b. Spectrometer for determining amount of crude in solution, e.g.:
   1. Visible spectrometer (detects intensity of crude color).
   2. Ultraviolet-fluorescence spectrometer (detects aromatics).
   3. Near-infrared (NIR) spectrometer (detects C-H bonds, asphaltenes).
   4. Nuclear magnetic resonance (NMR) spectrometer (detects the C-H shift).
c. Analytical balance for preparing crude extract standards used in calibrating spectroscopic response.
d. Gas chromatograph (optional).

4.3.4.2.1.2 Water-Wet Sponge Analysis

In addition to 4.3.4.2.l, water-wet sponge analysis requires a standard distillation extraction apparatus (e.g., Dean-Stark) for determining water volume recovered (see 4.3.2).

4.3.4.2.2 Reagents

4.3.4.2.2.1 Oil-Wet Sponge Analysis

Any good crude-oil solvent that has:

a. No visible color, if using visible spectrometer.
b. No aromatics, if using UV-fluorescence spectrometer.
c. Preferably no C-H bonds, if using NIR or NMR (e.g., hydrogen-free chlorofluorocarbons, or chlorocarbons).

4.3.4.2.2.2 Water-Wet Sponge Analysis

Any standard distillation extraction solvent, e.g., toluene.

4.3.4.3 Procedure/Precautions

4.3.4.3.1 Rig Site Handling

At the rig site, care should be taken not to jar the ends of the sponge core liner, as this can cause the rock core to shift in the barrel resulting in a mismatch of the sponge and rock core. Although the sponge core should be kept cool, freezing it with the rock core still inside carries some risk of inducing fractures in the rock core.

For certain formations, such as fractured or vuggy carbonates, core can break and wedge into the sponge causing it to bunch together. The larger-diameter sponge core is less susceptible to this problem.

Immediately after retrieval, the cores may be cut into sections (typically about 5 feet) and stored in transport containers filled with the same fluid used to saturate the sponge.

4.3.4.3.2 Oil-Wet Sponge Analysis

Because the sponge is presaturated with water and is highly compressible, any analysis of the amount of oil in the sponge should measure oil directly, and not infer oil volume from a measurement of the water in the sponge and the porosity (as is done in the distillation extraction method for measuring oil saturations of rocks).

Because the oil in the sponge is spread out in a thin layer over the oil-wet sponge, mechanical compression of the sponge is not a good method for trying to recover the captured oil.

A large-bore Nuclear Magnetic Resonance instrument, such as an NMR imager, can be used to directly determine the amount of oil in the sponge, provided the sponge sleeve is removed from the electrically-conducting aluminum liner. The direct NMR method will become more attractive as the measurement cost declines and NMR availability increases.

As of 1993, the least expensive, most widely applicable method, and the method recommended here, is solvent dissolution of the captured crude followed by a spectroscopic determination of the amount of crude in solution. Since sponge may be subject to attack by solvents during the solvent dissolution step, one must either select solvents that are gentle

on sponge or be sure that the subsequent analysis is unaffected by dissolved sponge components mixed in with the dissolved crude. For spectroscopic determination, a calibration curve should be developed by injecting known weights or volumes of lease crude into pieces of sponge, then solvent-extracting the sponge and determining the spectroscopic response of the extracts. Sponge samples should be taken from the actual lot of sponge used in the sponge coring operation. If different lots of sponge are used during coring, a new calibration curve should be developed for each new sponge lot.

4.3.4.3.3 Water-Wet Sponge Analysis

The water-wet sponge is hygroscopic, so care must be taken to prevent absorption of moisture from the atmosphere because this could impact the final analysis for the water saturation. Standard distillation extraction analysis can be performed on water-wet sponge to determine water content.

4.3.4.3.4 Separation of Sponge Sleeve and Aluminum Liner From Rock Core

With a table saw, make two cuts 180 degrees apart down the length (long axis) of the aluminum liner that jackets the sponge sleeve. Set the depth of cut to 0.25 inches (6.35 millimeters) so that the saw blade cuts only through the aluminum liner and not the sponge sleeve. Also, make the cut between a pair of aluminum centralizer vanes (the vanes that hold the rock core centered within the aluminum liner) instead of on top of a vane.

Next, cut through the sponge sleeve with a knife and remove each section of whole core from the liner for trimming. Note any evidence that the core has shifted relative to the liner. Mark the liner so it can be cut to the same length as the corresponding section of trimmed core and label the matching pieces of whole core and liner.

Complete the process of cutting and sealing the rock and sponge samples within 30 minutes to minimize exposure to the atmosphere. This minimizes evaporation of core fluids, oxidation of liquid crude, and the resulting errors in quantifying the amount of crude. If the sponge is not immediately analyzed, carefully preserve it until it is analyzed (see 2.2.6 for preservation methods).

The corrected whole-core oil (or water) saturation is obtained by adding the volume of oil (or water) in each piece of trimmed core to the volume of oil (or water) in the matching piece of sponge sleeve and dividing by the total whole-core pore volume.

4.3.4.3.5 Solvent Extraction and Spectroscopic Analysis (Oil-Wet Sponge)

Analysis of oil-wet sponge is as follows:

a. Prepare sponge samples with known amounts of lease crude and extract sponge with chosen solvent. Calibrate the spectroscopic response using these extracts.

b. Place sponge core crude into solution by any of the following:

1. Soxhlet extraction.
2. Submerged soak and vigorous shaking.
3. Submerged soak and disaggregation of sponge in blender.

For options (2) and (3) it will be necessary to separate the sponge sleeve from the aluminum liner.

4.3.4.4 Calculations

4.3.4.4.1 Oil-Wet Sponge Analysis

Calculations for oil-wet sponge analysis are as follows:

a. Determine the spectroscopic response of an aliquot of the solvent extract.
b. Knowing the total amount of solvent extract and using a calibration performed with prepared standards, determine the total amount of crude that was originally imbibed by sponge.

$$V_{tc} = W_{tc} / \rho_c = (R_a / R_{pwf}) (W_{ts} / \rho_c)$$

Where:

R_{pwf} = Response of spectrometer per weight fraction of sponge-imbibed lease crude in the final solvent extract. This response calibration is done using prepared standards consisting of solvent extracts of sponge samples (of the current lot) that have been injected with known amounts of lease crude. (For example, absorbance or fluorescence of solvent extract per weight fraction of sponge-imbibed crude when using a given sample cell at a given wavelength of light.) Units of R_{pwf} must be the same as the units of R_a.

R_a = Response of spectrometer for an aliquot of the solvent extract recovered from a piece of sponge. Response is obtained under the same conditions (e.g., using same sample cell, wavelength, etc.) that were used when calibrating the spectrometer response with prepared standards. Units of R_a must be the same as the units of R_{pwf}.

W_{ts} = Weight of the total crude solution recovered from a piece of sponge core. Weight is measured before the small aliquot is removed, g.

W_{tc} = Total weight of crude that was in a piece of sponge core, g.

V_{tc} = Total volume of crude that was in a piece of sponge core, cm^3.

ρ_c = Density of lease crude, g/cm^3.

4.3.4.4.2 Water-Wet Sponge Analysis

Knowing the brine salinity and density, the volume of brine that was in the sponge can be calculated from the volume of distilled water recovered from the sponge as follows:

$$V_{br} = [(V_w)(\rho_w)/\rho_b]\ [1{,}000{,}000/(1{,}000{,}000 - C_s)]$$

Where:

V_{br} = Volume of brine corresponding to the volume of distilled water collected from a piece of sponge, cm^3.
V_w = Volume of distilled water collected from a piece of sponge (e.g., Dean-Stark), cm^3.
ρ_w = Density of distilled water, g/cm^3.
ρ_b = Density of lease brine having a concentration, C_s, of salt, g/cm^3.
C_s = Concentration of dissolved salts in lease brine = (1,000,000) (weight of salt)/(weight of brine), ppm.

4.3.4.5 Advantages/Limitations

Sponge core is a less expensive alternative to pressure-retained coring (as described in 4.3.3) and is operationally simpler. The amount of oil (or water) captured in the sponge is added to the amount of oil (or water) remaining in the core to obtain more accurate values of oil (or water) saturation.

Unlike pressure core, any hydrocarbon gases that are released are not retained, and so are not available for analysis. Also, with sponge core, a choice must be made between determining corrected values of the core's oil saturation or determining corrected values of the core's water saturation. Both corrected saturations cannot be determined using the same sponge core.

4.3.4.6 Accuracy/Precision

For extraction and spectroscopic analysis combined, the oil volume from oil-wet sponge core analysis is within 5 percent of the actual volume of oil or 0.2 ml of oil, whichever is larger. The water volume from water-wet sponge core analysis is within 0.1 ml of the actual volume of water from which the corresponding volume of brine can be calculated if the brine salinity and density are known. The water volume is determined more accurately than the oil volume because the water is determined by a direct measurement of volume. In contrast, the oil sample is highly diluted in solvent and only an aliquot of the dilute solution is analyzed spectroscopically.

4.3.4.7 Calibration

Refer to 4.3.1.8. Standard calibration procedures should be followed for all commercial equipment.

4.3.5 Productive Shale

4.3.5.1 Principles of Analysis

This paragraph covers the special considerations for measuring fluid saturations of the pore space in "oil productive shale"—rocks composed of clay-to-silt-sized particles and potentially oil productive. Analysis of samples from this type formation may need special handling due to their low permeability, possible presence of tightly bound or structural water, and potential for solid organics. A retort process is not recommended for samples containing solid organics. Oil will be generated from the solid organics at temperatures used in this process yielding high oil volumes. A distillation process should be used to determine fluid saturation data.

The principle is the same as in 4.3. This method should be used if solid organics present are not to be included as oil volume. If shorter analysis times are needed, removal of hydrocarbons can be expedited by crushing the sample. The crushed sample will also expedite equilibrium time for grain volume measurements.

4.3.5.2 Apparatus

The apparatus for distillation extraction is the same as in 4.3.1.2. For sample-crushing, a mortar and pestle are needed.

4.3.5.3 Procedures/Precautions

The procedures are the same as in 4.3.1.3. If samples are to be crushed to expedite hydrocarbon removal or grain volume determinations, then bulk volumes should be measured and samples weighed prior to crushing. Crush sample using mortar and pestle and reweigh. If there is weight loss during crushing, a correction to either rock or fluid volumes must be made. If weight loss is high, the sample should be discarded and another sample tested. Careful control of sample loss throughout the procedure is critical to accurate oil volume measurement.

4.3.5.4 Calculations

Calculations should be performed as stipulated in 4.3.1.4.

4.3.5.5 Advantages

Advantages of this method include:

a. All measurements can be made on the same sample.
b. Solid organics are not represented in oil volumes.
c. Accurate grain densities can be measured.

4.3.5.6 Limitations

Limitations of this method include:

a. Oil volumes are sensitive to drying techniques.
b. Long extraction times are needed due to the low permeability of this type of rock material.
c. Sample loss is critical in determination of oil content.

4.3.5.7 Accuracy/Precision

Proper distillation and drying techniques should permit oil and water content determination to within 2 percent of the values measured. Special care is necessary when drying samples because of the rocks' ability to rehydrate quickly, which will cause erroneously low oil volumes.

4.3.5.8 Calibration

Calibration techniques should be performed as outlined in 4.3.1.8.

4.3.6 Tar (Oil) Sand Analysis

4.3.6.1 Principles of Analysis

The distillation extraction method can be used to determine the fluid saturations of unconsolidated tar (oil) sands. However, certain modifications to the procedure and data presentation are required. The required changes are described in the following paragraphs.

4.3.6.2 Apparatus

The same basic equipment as described in 4.3.1.2 may be used in this procedure.

4.3.6.3 Procedures/Precautions

The unconsolidated core may be frozen prior to obtaining a sample. The plug should be drilled using liquid nitrogen as a lubricant. The frozen plug is then sleeved, usually with heat shrinkable polytetraflouroethylene (PTFE), in order to keep the plug intact when the bitumen is removed. The sample is then placed in the distillation extraction apparatus (see Figures 4-4 and 4-5) with toluene as the solvent. In some instances, the core may not be frozen, or if the sample is obtained during the quarry process of a tar sand deposit, the test sample may be analyzed on a volume/weight basis. In such instances, the test specimen is transferred to an appropriate extraction thimble such as those discussed in 4.3.1.2.(f).

Avoid violent boiling by supplying a minimum heat rate to boil the solvent. Change the toluene, as required, to prevent tar build up and subsequent "bumping" or super heating. This latter condition, should it occur, can slow down the distillation/extraction process or possibly negate the complete test.

If the bitumen has an initial boiling point greater than 392°F (200°C), the oil content can be determined directly. For direct determination, the toluene-bitumen bottoms are transferred into a volumetric flask and toluene is added until the total volume equals the volumetric flask's volume. An aliquot is taken from the flask and placed on glass fiber filter paper. The toluene is evaporated and the bitumen is weighed. The total bitumen weight is then calculated. This method is known as the Modified Dean-Stark (distillation extraction) Method or AOSI-3573.[4]

4.3.6.4 Calculations

The liquid contents of the sample are usually reported as weight percent as opposed to percent pore volume.

$$\text{Water Content} = [(V_w \times \rho_w)/\text{Sample Weight}] \times 100$$

$$\text{Oil Content} = \frac{[(\text{Sample Weight}) - (V_w \times \rho_w) - \text{Dry Weight}] \times 100}{\text{Sample Weight}}$$

Where:

Water Content = weight percent of the sample weight.
Oil Content = weight percent of the sample weight.
V_w = volume of water recovered, ml.
ρ_w = density of water, g/cc.
Sample Weight = original (net) sample weight, gm; i.e., excludes sleeve weight.
Dry Weight of cleaned and dried sample, g.

Note: For practical purposes the density of the water may be assumed to be 1.00 g/cc.

(For ACOSA Method calculations, see 4.10, Reference 4.)

4.3.6.5 Advantages/Limitations

Advantages/limitations for this method include:

a. The liquid content values are determined on one sample.
b. The method is fairly rapid.
c. The calculations are not complex.
d. If the sample has a high tar (oil) content, it may be necessary to stop the distillation process so that the dirty toluene in the boiling flask can be replaced with clean toluene.

4.3.6.6 Accuracy/Precision

The weight percent values should be reported to the nearest 0.1 percent. Although there are no industry standards, the weight percent values should fall in the range of ±0.5 percent of the calculated value. This level of accuracy should be acceptable for most applications.

4.3.6.7 Calibration

Refer to 4.3.1.8.

4.3.7 Filtrate Invasion Analysis (Pressure Cores)

4.3.7.1 Principles of Analysis

The objective of filtrate invasion analysis is to quantify the amount of drilling fluid filtrate that invades the core during the coring operation. This is accomplished by adding a tracer(s) to the drilling fluid system in known quantities and measuring this tracer(s) content in the core. The degree of this tracer(s) concentration/activity/dilution allows determination of the drilling fluid filtrate amount contained in the core. Knowledge of the drilling fluid filtrate amount in the core allows a qualitative assessment of the saturation alteration that occurs by flushing during coring and calculation of the salinity of the formation water (see 7.7).

Filtrate invasion studies may be planned for other than pressure cores using a variety of water-based and oil-based coring fluids. Procedures for such studies will not be addressed in this publication. The wide range of core types

along with a similar broad spectrum of coring fluids and potential tracer materials precludes the recommendation of standard procedures for these other techniques.

Use of relative permeability fractional flow characteristics of the formation enhances the assessment of the fluid saturation alteration. The use of low-invasion profile coring bits minimizes drilling fluid filtrate invasion and saturation changes due to flushing.

The drilling fluid system is tagged with a tracer material. For water-base drilling fluid, tritiated water is recommended and is the basis of the following procedural descriptions. For oil base drilling fluid, tritiated hexadecane has been used.

The drilling fluid system is circulated sufficiently to allow even mixing of the tracer. A "rule of thumb" is to circulate the drilling fluid "bottoms up" three times. Periodically during coring, a drilling fluid sample is taken for use as a base reference for tracer concentration/activity in the drilling fluid for filtrate concentration calculations in the core.

A vertical plug is drilled through selected intervals resulting in "plug" and "donut" samples (see Figure 4-7). Water is extracted from these samples and a determination of the tritium activity is made by liquid scintillation analyses. The decrease in tracer concentration/activity in comparison with the drilling fluid filtrate is equal to the dilution of the drilling fluid filtrate by the formation water. The amount of drilling fluid filtrate invasion can be determined for both the exterior of the core and the interior of the core as well as the total drilling fluid filtrate invasion.

To minimize the extent to which drilling fluid filtrate migrates into the interior of the core by diffusion or imbibition, the samples should be cut at the well site as soon as possible after core retrieval. If this is not feasible, then the inner core barrel should be promptly frozen and preserved. Gidman and Conner, 1992[5] have reported that core freezing causes some fluid migration that may lead to incorrect assessment of the amount of drilling fluid filtrate invasion.

4.3.7.2 Apparatus

The following apparatus are used:

a. Drill press capable of cutting samples using liquid nitrogen as the bit lubricant.
b. Distillation extraction apparatus in 4.3.1.2 for plug analysis.
c. Drilling fluid filter press for extraction of the coring fluid filtrate.

4.3.7.3 Procedures/Precautions

Procedures and precautions include:

a. Use established safety procedures.
b. Designate selected intervals for the filtrate invasion study. Generally a sample is taken approximately every 4 feet (1.22 meters) throughout the cored section.
c. Care should be taken to avoid evaporation of water during handling of the core, plugs, and donuts.
d. The selected full diameter core intervals should be faced into right cylinders approximately 2 to 3 inches (50.8 to 76.2 millimeters) long. A vertical plug is drilled from the center of each sample to provide plug and donut portions of the sample (see Figure 4-7).

There are other methods to determine the spatial extent of drilling fluid filtrate invasion, such as cutting a plug perpendicular to the axis of the whole core. This plug is then sliced into many segments and each segment is analyzed for drilling fluid tracer.

e. The plug and donut samples are weighed and placed into separate Dean-Stark distillation extraction apparatuses to distill the water from the core and to extract the oil. Thimbles should be used to minimize grain loss. The water removed from each sample is measured, bottled, and marked. Since tritiated water has nearly the same chemical and physical properties as water, it is not separated from the ordinary water during the distillation process.
f. The samples are dried in a convection oven at 240°F (117°C) until weights are stable. The dry weight of each sample is recorded.

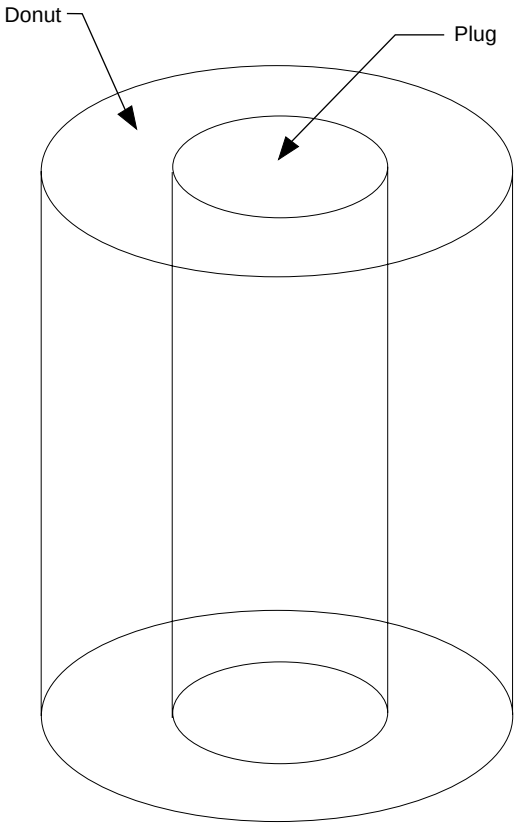


Figure 4-7—Plug and Donut Sample For Filtrate Invasion Study

g. Porosity is determined from measurements on the plug sample. The donut is assumed to have the same porosity as the corresponding plug. The pore volume of the companion donut is determined by the ratio of weight of the plug to the weight of the donut.

h. Water saturation is calculated for the plug and donut samples.

i. The amount of drilling fluid filtrate in the water extracted from each plug and donut sample pair is determined. The ratio of drilling fluid filtrate to total water in each sample is calculated and expressed as a percent of each sample's pore volume.

j. The analyst should observe appropriate safety measures when using tritiated water.

4.3.7.4 Calculations

The following calculations are appropriate for this method:

a. Water saturation in the plug and donut:

$$S_w\,(\text{plug}) = [S_{wt}\,(\text{plug})/P_v\,(\text{plug})] \times 100$$

$$S_w(\text{donut}) = [S_{wt}(\text{donut})/(P_v(\text{plug}) \times (\text{donut wt}/\text{plug wt})] \times 100$$

Where:

S_w (plug) = water saturation of the plug given as a percent of pore volume.

S_w (donut) = water saturation of the donut given as a percent of pore volume.

S_{wt} (plug) = total water volume of the plug, ml.

S_{wt} (donut) = total water volume of the donut, ml.

P_v (plug) = pore volume of the plug, ml.

The plug and donut weights are given in g.

b. Filtrate invasion in the plug and donut:

$$\text{Filtrate (drilling fluid)} = \frac{\text{Tritium Activity (sample)} \times 100}{\text{Tritium Activity (drilling fluid)}}$$

$$\text{Filtrate } (PV) = \text{Filtrate (drilling fluid)} \times S_w/100$$

Where:

Filtrate (drilling fluid) = drilling fluid filtrate as a percent of total water.

Filtrate (PV) = pore volume, percent.

S_w = water saturation, percent pore volume.

Tritium activity is in pCi/ml.

4.3.7.5 Advantages/Limitations

Advantages and limitation of this method include:

a. The advantage of determining coring fluid filtrate invasion on pressure-retained core as opposed to either conventional or sponge core is that all the water phase is retained within the core during retrieval since the core barrel pressure is maintained at original coring pressure. Thus, the quantity of drilling fluid filtrate contained in the core segments at the completion of the coring operations prior to surfacing can be quantitatively determined.

Fluids are expelled from conventional or sponge cores as the core barrel is retrieved from downhole to the surface. Since conventional cores contain a larger percent of drilling fluid filtrate in the periphery of the core, this filtrate is the first aqueous fluid to be expelled as the pressure drops during core barrel retrieval. The volume of drilling fluid filtrate is reduced by this fluid expulsion, and the amount of drilling fluid filtrate measured from these types of cores will be only the residual value.

Either a water-retaining or an oil-retaining sponge liner can be selected. The water expelled by gas evolution is recovered with a water-wet sponge and can be analyzed for drilling fluid filtrate tracer. The core fluids can also be analyzed for tracers by Dean-Stark extraction and analysis of the recovered waters.

b. In situ saturation may not be qualitatively determined even with knowledge of the amount of drilling fluid filtrate contained in the core segments. The drilling fluid filtrate displaces both oil and water. The amount of each phase displaced depends on the in situ saturations, fluid properties, and formation properties. If in situ, the formation is at irreducible water saturation, then the filtrate displaces mostly oil or gas. Conversely, if in situ, the formation is near residual oil saturation then mostly water or gas is displaced. Between these two extremes, oil and water displacement depend on the respective relative permeabilities. Relative permeability fractional flow curves should be used in assessment of the fluid saturation alteration during coring operations.

c. Tritiated water is the recommended drilling fluid filtrate tracer because it is can be recovered by Dean-Stark extraction of water from the core material. If ionic tracers are used instead of, or in addition to tritiated water, a selection criterion is that they should not be chemically or biologically altered by the reservoir rock. Anionic tracers used with varying degrees of success in water-based drilling fluid include bromide, iodide, and nitrate salts. Many reservoirs waters contain bromide and iodide in significant amounts, so accurate analysis of the reservoir water composition is important in selecting the appropriate tracer. The nitrate ion tracer requires a drilling fluid bactericide to prevent microbiological reduction from nitrate to nitrite.

Ionic tracers must be recovered from the cores by a method of water extraction, that requires considerably more time and

effort than extraction of tritiated water. The procedures are provided in 7.7. Ionic tracers provide semi-quantitative water invasion results.

d. Since the plug and donut samples are placed into the Dean-Stark distillation extraction apparatus in a frozen state, gas is prevented from escaping from the samples. Weight loss from gas release during extraction is included in the weight loss attributed to oil extraction. Oil is determined gravimetrically. If sufficient gas is present, a significant error may be introduced into the oil saturation determination. Thus, only water saturation is reported from filtrate invasion studies.

4.3.7.6 Accuracy/Precision

The precision of the measurements of the extent of drilling fluid filtrate invasion into the core is within about 5 to 20 percent. The simultaneous use of more than one drilling fluid tracer adds confidence in the reported filtrate invasion. There is no information on the accuracy of the method, since there is no accepted reference value for the extent of drilling fluid filtrate invasion downhole.

4.3.7.7 Calibration

Calibration of the liquid scintillation equipment, anionic tracer detection equipment, and ion chromatograph is essential for consistent results.

4.4 SOLVENT FLUSHING

4.4.1 Karl Fischer Titration

4.4.1.1 Principles of Analysis

Core plug samples are cleaned by dynamic miscible displacement with an appropriate sequence of solvents and the water content of the produced effluents analyzed by Karl Fischer titration (refer to ASTM D1364-90[6] and ASTM D4377-88[7]).

4.4.1.2 Apparatus

Apparatus appropriate for this method includes:

a. General—The apparatus includes a core flow system for the solvent injections and a Karl Fischer titrimeter for the effluent analysis. The core flow system is composed of a core holder assembly, solvent delivery device, and related hardware. The Karl Fischer analysis requires a titrimeter, appropriate Karl Fischer reagents, analytical balance, and syringes.

b. Core holder assembly—Hassler or hydrostatic core holder, sleeves, end-pieces, and a source of pressure appropriate for the expected confining and flow pressures.

c. Solvent delivery device—A constant rate high pressure liquid chromatographic (HPLC) pump or a gas-driven transfer system may be utilized. The pump may be a syringe or a continuous-flow type system. The constant pressure method requires a high pressure gas bottle, regulator, and a vessel such as a floating piston accumulator.

d. Related hardware—Consists of corrosion-resistant tubing, valves, and fittings to connect the core holder and solvent delivery system.

e. Karl Fischer titrimeter—Volumetric or coulomatic titrimeter.

f. Analytical balances—Covering range from 0.1 milligram to 1000 g.

g. Syringes—0.1–10 ml capacity, plastic or glass.

h. Materials and reagents:

1. Karl Fischer reagents: Utilize reagents appropriate for choice of titrimeter.
2. Toluene, reagent grade.
3. Methanol, Karl Fischer grade.
4. Glass vials, 25 ml capacity with caps lined with polytetrafluoroethylene (PTFE).
5. Glassware, stoppered vessels of approximately 1000 ml capacity, 10 ml receiving tubes.
6. Silver nitrate, 0.1 Normal.
7. Check solution, 0.1 weight percent water in methanol standard.

4.4.1.3 Procedures/Precautions

4.4.1.3.1 Procedures

This method is suitable for determining water saturation in whole core and plug-sized samples and can be used as an alternative to the distillation extraction (Dean-Stark) method.

a. The sample is weighed to the nearest 0.1 gram (assuming a plug-size sample), and loaded into the core holder. The desired confining stress is applied and any produced fluids are collected in a receiving tube and the volumes are recorded.

b. An alternating methanol-toluene solvent injection sequence is performed, beginning with the solvent that is miscible with the mobile phase, i.e., methanol if the brine is mobile or toluene if the oil is mobile.

c. All storage vials, syringes, or collection vessels should be weighed or tared prior to use.

d. A glass vial is filled with the initial injectant reagent and sealed. The solvent is then injected into the core sample at rates or pressures suitable for the core material, and the effluent is collected in a stoppered vessel. Care should be taken to minimize exposure of the solvent to atmospheric humidity.

1. For methanol injection, the effluent is sampled periodically and tested for the presence of salts using silver nitrate. Injection is complete when a drop of silver nitrate fails to form precipitates in the effluent aliquot.
2. For toluene, injection is continued until (a) the effluent appears clear, or (b) refractive index of the effluent equals the injectant.

3. The Karl Fischer titration is performed as soon as possible to minimize changes in water content due to absorption of moisture. (refer to Steps g–i).

e. Step d is repeated using the alternating solvent.

f. Step d is repeated using the initial solvent.

g. The storage vials and effluent vessels are reweighed and the weights of the solvents calculated.

h. Using a syringe, a portion of the injectant solution is analyzed for water content by Karl Fischer titration according to the manufacturers' recommended procedures. The syringe is reweighed to ascertain the exact weight of solvent analyzed. Making sure the effluent sample is uniform, water content is determined as described above. Results are reported in weight percent or ppm equivalent.

i. The sample is unloaded, dried using appropriate techniques, weighed, and the pore volume determined by helium injection.

4.4.1.3.2 Precautions

Precautions for Karl Fischer titration include:

a. Material safety data sheets should be consulted for proper handling procedures for all required chemicals, including the Karl Fischer reagents.

b. Methanol is highly susceptible to water absorption from atmospheric humidity as well as from untreated glassware. Since it is extremely difficult to quantify these variables, exposure to air should be minimized, and the Karl Fischer titrations performed as quickly as possible.

c. The Karl Fischer titration requires only a small amount of solvent; therefore, the analyzed sample must be representative of the entire solution to yield accurate results.

4.4.1.4 Calculations

The following calculations should be performed:

a. Water Saturation:

$$S_{wes} = W_{es} \times (S_{we} - S_{wi})$$

$$S_{ws} = \text{Sum } S_{wes}$$

$$S_{wb1} = S_{ws} / [(1 - A) \times \rho_b]$$

$$S_{wb2} = (S_{wb1} \times 100)/PV$$

Where:

ρ_b = density of brine, g/ml.
S_{wes} = water content of effluent solvent, g.
W_{es} = weight of effluent solvent, g.
S_{we} = effluent water content, g/g.
S_{wi} = injectant water content, g/g.
S_{ws} = sample water content, g.
Sum S_{wes} = summation of the water contents of the effluent solvents, g.
S_{wb1} = sample brine content, ml.
S_{wb2} = sample brine saturation, percent of the pore volume.
A = salt content of the brine, grams of salt per gram of brine.
PV = pore volume of the sample, ml.

b. Oil Saturation:

If the sample was fully saturated, oil saturation may be determined by volumetric difference:

$$S_o = 100 - S_{wb2}$$

If the sample contained an initial gas saturation, the oil saturation must be determined by gravimetric difference:

$$W_o = W_i - W_d - [S_{ws} / (1 - A)]$$

$$V_o = W_o / \rho_o$$

$$S_o = \frac{V_o \times 100}{PV}$$

Where:

S_o = oil saturation as a percent of the pore volume (PV).
S_{wb2} = sample brine saturation, percent of the pore volume.
W_o = weight of oil, g.
W_i = initial weight of the sample, g.
W_d = weight of the dry sample, g.
S_{ws} = sample water content, g.
V_o = volume of oil, ml.
ρ_o = density of oil, g/ml.
A = salt content of the brine, grams of salt per gram of brine.
PV = pore volume of the sample, ml.

c. Data reporting—Fluid saturations are reported to the nearest 0.1 percent pore space.

4.4.1.5 Advantages

Advantages of this method include:

a. All saturation levels can be determined.
b. The Karl Fischer titration is very precise.
c. Damage to sensitive minerals is minimized.
d. The procedure removes salts from the sample.
e. Saturations can be determined in special core tests in which stress hysteresis effects prohibit unloading the sample.

4.4.1.6 Limitations

Limitations of this method include:

a. Methanol readily absorbs moisture from the environment.
b. Accuracy of the method is dependent on solvent handling and storage techniques.
c. The analyzed sample must be representative of the bulk effluent.

d. The technique is more complex and expensive than the distillation extraction method.
e. Oil saturations are determined indirectly. Oil saturation by gravimetric difference assumes no grain loss of the sample.
f. This technique is not appropriate for samples that contain halite, sulfur, or other minerals that are soluble in methanol.

4.4.1.7 Accuracy/Precision

Precision of automated Karl Fischer titration units is ±0.5 percent of the measured value. However, the accuracy of the technique is limited to the ability to handle and store the solvents without altering the water content.

4.4.1.8 Calibration

Instrument operation is checked by analyzing standard solutions of known water content. Spiked samples should yield percent recoveries between 90 and 110 percent. Duplicate analyses are performed on 10 percent of the total samples analyzed and should have a relative percent difference less than 2 percent.

4.5 SCANNING METHODS

4.5.1 Introduction

Several laboratory techniques for scanning measurements of water, oil, and gas saturations in core samples have been reported. These techniques include: (a) linear x-ray absorption, (b) microwave absorption, (c) computer assisted (axial) tomography (CT), (d) linear gamma-ray absorption, and (e) nuclear magnetic resonance (NMR). The linear x-ray, gamma-ray, microwave, and CT methods may be considered as emerging technologies for saturation measurement. Other scanning techniques that have been used for saturation determinations are radio wave resonance and neutron attenuation radiography. The neutron radiography has a good potential for determination of fluid saturation because neutrons are attenuated much more by fluids (proton) than by rocks. Thus, neutron tomography may not require any addition of tagging agents to the resident fluids for saturation determination. These techniques are not widely used for core analysis.

4.5.2 Principles

The x-ray, CT, and gamma-ray techniques measure the absorption of high energy electromagnetic radiation by fluids that have been tagged with high absorption agents (usually high atomic weight elements) to determine fluid saturations. The microwave absorption technique is based on the absorption of microwave energy by water molecules. NMR is based on the detection of the proton (H1), carbon (C13), sodium (Na23), phosphorous (P31), and fluorine (F19) containing fluids by magnetic fields alternating at radio frequencies while the sample is located in a large constant magnetic field.

4.5.3 Apparatus

Apparatus for these techniques are generally sophisticated and expensive. X-ray and CT equipment manufactured for medical use are often adequate for saturation measurements. Medical NMR equipment is not often suitable for saturation measurements. Microwave and gamma-ray absorption apparatus have usually been specially built.

4.5.4 Procedures/Precautions

Several parameters must be considered for accurate saturation determination when scanning methods are used, such as the power level setting, tagging material and its concentration, tagging fluid-rock and fluid-fluid interaction (refer to the Bibliography for the many procedures and precautions for the individual techniques).

4.5.5 Calculations

Because all techniques (except NMR) generate signals from both the solid matrix and the fluids, measurements at a minimum of two known saturations (normally 0 and 100 percent) are required to establish a calibration line. The unknown saturations for the oil-brine system are then calculated based on the following equation:

$$S_w = (U_{sat} - U_o)/(U_w - U_o)$$

Where:

S_w = unknown brine saturation, as a fraction.
U_{sat} = attenuation of core at the unknown saturation (S_w).
U_w = attenuation of core at 100% brine saturation.
U_o = attenuation of core at 100% oil saturation.
(The units of U_{sat}, U_w, and U_o must all be the same.)

4.5.6 Advantages

An important advantage of these methods is their ability to provide information on the spatial distribution of the fluid saturation. Another advantage of these techniques is that the measurements are made noninvasively and nondestructively. Extraction of fluids is not required prior to flow tests on preserved cores (that can be difficult in full-sized or tight cores). Saturations can be monitored without terminating a laboratory experiment.

An advantage of CT and NMR imaging techniques is that they provide the saturation distribution of fluids in all three dimensions.

An advantage of the microwave and NMR techniques is their capability to determine water saturation in the core without addition of any tagging agents. The microwave and NMR techniques can be used for saturation determination in standard core analyses because they do not require any tagging agents and calibration curves are established after core cleaning and resaturation processes.

4.5.7 Limitations

A limitation of the x-ray, CT, and gamma-ray techniques is that they can determine saturations in cores only if they are saturated with tagged fluids. Therefore, they may not be suitable for the basic core analyses.

A limitation of the NMR technique is the inability to handle cores containing significant ferromagnetic materials, clays, or gas.

4.5.8 Accuracy/Precision

The accuracy and capability of some of the scanning techniques for saturation measurement are listed in Table 4-2. The accuracies listed in this table represent values achievable only under optimum calibration and measurement methodology.

4.5.9 Calibration

The absorption techniques require measurements of signal intensity at a minimum of two saturation levels to establish a calibration curve to relate instrument response to saturations. Calibration is instrument dependent.

4.6 COAL ANALYSIS

4.6.1 Principles of Analysis

Basic core analysis techniques, such as retort analysis and solvent distillation extraction, are not suitable for the determination of fluid saturations of coal samples. Alternative methods have been developed to measure the fluid saturation of coal. With coal, the fluid of primary concern is water. Water saturation, or more appropriately, moisture content is a fundamental property of coal that must be accurately determined to properly evaluate a coal seam. The method described here for determining the moisture content of coal involves drying water from a crushed sample in a convection oven and taking successive weight measurements until equilibrium is achieved. The weight loss is equivalent to the amount of water driven off, and moisture content is reported as the weight percentage of moisture with respect to the weight of the wet coal sample.

4.6.2 Apparatus

The following apparatus are appropriate for this method:

a. Crusher—Use a jaw, cone, or rotary crusher to reduce the coal to the size retained on a No. 8 (2.38 millimeter) sieve.

b. Drying pans—The pans used should be of sufficient size to accommodate crushed samples spread to a thickness of less than 2.5 centimeters. The sides of the pans should not be greater than 3.8 centimeters in height. The pans should be made of a material that is stable and will not corrode at the temperature used.

c. Drying oven—A forced-draft oven that is capable of maintaining a constant temperature of 225°F, ±5°F (107°C, ±3°C) should be used. The velocity of forced air in the oven should be sufficiently low so as not to disturb the coal sample. An oven with a gas port capable of accommodating a hose from a nitrogen tank is desirable, because flowing nitrogen, rather than air, through the oven can help minimize oxidation of the coal sample.

d. Balance—The balance used to weigh the samples should be sensitive to 0.1 gram and have a capacity large enough to accommodate both the sample and the pan.

e. No. 8 Sieve (2.33 millimeter)—The sieve should be large enough to hold the entire prepared sample while sifting out the larger particles.

f. Glass desiccator—The desiccator should be large enough to hold the prepared sample and sample pan. It is recommended that either no desiccant or fresh color-coded desiccant be used because some desiccants actually give off moisture if not fresh.

4.6.3 Procedures/Precautions

4.6.3.1 Sampling

4.6.3.1.1 Crushing

Use a jaw crusher or other suitable type of crusher or instrument to reduce the coal to the size retained on a No. 8 (2.38 millimeter) sieve. At least 95 percent of the crushed sample should pass through a No. 8 sieve.

Table 4-2—Measurement Capabilities of Scanning Saturation Techniques

Technique	Dimensions	Tagging requirements	Accuracy, saturation units	Spatial resolution, cm	Maximum sample size, in.	Measured fluids
Linear x-ray	2	yes	1	1.0	12	oil, water, gas
Computer Assisted Tomography	3	yes	1	0.2	12	oil, water, gas
Gamma-ray	2	yes	3	0.1	12	oil, water, gas
Microwave	2	no	1	2.0	2	water
Nuclear Magnetic Resonance	3	no	1	0.1	4	oil, water

4.6.3.1.2 Sample Size

The sample used for moisture content determination should have a minimum weight of about 500 grams.

4.6.3.2 Weighing

4.6.3.2.1 Drying Pan

Immediately after sample preparation is complete measure and record the weight of the clean and dry drying pan. Pour the crushed, sifted coal into the drying pan and spread to a thickness of not more than 2.5 cm.

4.6.3.2.2 Sample Initial Weight

Measure and record the weight of the wet coal sample and the drying pan. Place the pan in the oven at a temperature of 225°F, ±5°F (107°C, ±3°C).

4.6.3.2.3 Dry Weight Measurements

After the sample has dried for a period of 1 hour, remove it from the oven and place it in a glass desiccator to cool. The use of desiccant is not recommended. Weigh the sample immediately after cooling and record the weight. Return the sample to the oven. Repeat this weighing procedure at half-hour intervals until the weight loss between successive measurements is less than 0.05 percent of the sample weight. The final measurement taken is considered the dry weight of the sample plus the weight of the drying pan.

4.6.4 Precaution

Do not over-dry the coal sample. Oxidation of the coal occurs if it dries too long in air.

4.6.5 Calculations

Calculate moisture content using the following equation:

$$M = [(W_2 - W_3)/(W_2 - W_1)] \times 100$$

Where:

M = moisture content of coal sample,%.
W_1 = weight of empty pan, g.
W_2 = weight of wet coal plus pan, g.
W_3 = weight of dry coal plus pan, g.

4.6.6 Advantages

The principle advantage of this method is its simplicity. The procedure does not require a large capital investment in specialized equipment. Neither does it require specialized training to perform the measurements. More sophisticated techniques are available to determine the moisture content of coal (refer to 4.10, References 8, 9, and 10). However, in the context of basic core analysis, the technique described here is adequate.

4.6.7 Limitations

Some types of coals have a tendency to oxidize using the technique described here. Moisture content results will be too low if oxidation occurs; oxidation adds weight and will cause the dry weight to be too high. It is recommended that drying times be kept within the guidelines outlined here to minimize the possibility of oxidation. Forcing nitrogen, rather than air, through the oven may also help to reduce the potential for oxidation.

4.6.8 Accuracy

The accuracy of this technique is very good because it depends only upon the accuracy of the balance. Thus, the accuracy for this technique is ± 0.05 percent.

4.6.9 Precision

Results of duplicate moisture content determinations performed on the same sample by the same technician using the same equipment should not differ by more than ±0.3 percent. Results of moisture content measurements performed by different laboratories on adjacent samples should not differ by more than ±0.5 percent.

4.6.10 Calibration

Use of a calibration weight set is recommended to ensure that the balance works properly. Periodic calibration of the balance at regular intervals should also be performed by a trained technician according to factory specifications to ensure measurement accuracy.

4.7 OIL SHALES

4.7.1 Principles of Analysis

Oil shale is defined as rock composed of clay to silt size particles containing various quantities of solid organic material (e.g., kerogen). The solid organics present in these samples are typically liquefied to generate oil. Analysis of this type of formation, with low permeability and potential for solid organics, requires special procedures.

Oil and water saturations are obtained by a high temperature retorting process. No attempt is made to measure gas-filled pore volume. The fluid saturations are reported in gallons per ton.

4.7.2 Apparatus

The apparatus is the same as used for the Retort Method at Atmospheric Pressure (see 4.2.1.2). The fluids are collected in 15 cm^3 centrifuge tubes. Typically, gas is evolved during the retorting process. Should it be desirable to collect/measure these gases, modified glassware may be used consisting of a calibrated liquid receiving section with the upper portion

of the glassware fitted with a sidearm to allow produced gases to be channeled to collection cells or measuring equipment.

4.7.3 Procedures/Precautions

The procedure is similar to the procedures described in 4.2.1.3, with the following exceptions:

a. The representative sample, approximately two inches in length, is not broken in half. Permeability is a not a consideration in oil shale analysis.

b. No attempt is made to screen the crushed sample to exclude fines created during the crushing process.

c. An adjacent sample is not prepared for the mercury pump tests described in 4.2.1.3, because neither the gas-filled pore volume nor the porosity are required.

d. Oil coking and cracking losses are ignored. If significant, a correction for mechanical oil hold-up should be made.

e. The oven is allowed to heat at a rate consistent with the heating elements and number of retorts in place, i.e., no attempt is made to measure pore water at an initial oven temperature setting. The oven temperature is set initially at 1000°F (538°C). When the oven has stabilized at this temperature and all collection tube volumes remain constant, the retorting process is considered complete.

f. Under certain conditions (e.g., samples containing different varieties of kerogen) the volume of oil recovered may vary with the rate of heating.

g. Only the total water collected is used for the final calculation of water content.

h. Retorting operations should be carried out with adequate ventilation (e.g., under fume hoods), as typical oils recovered from kerogens are high in sulfur content and give off noxious vapors.

4.7.4 Calculations

The oil and water contents are calculated as follows:

$$\text{Oil Content} = \frac{\text{Volume of Oil Recovered} \times 239.7}{\text{Sample Weight}}$$

$$\text{Water Content} = \frac{\text{Volume of Water Recovered} \times 239.7}{\text{Sample Weight}}$$

Where:

Oil volume is in milliliters.

Water volume is in milliliters.

Sample weight is in grams.

Oil content is in gallons/ton.

Water content is in gallons/ton.

4.7.5 Advantages

Advantages of this method include:

a. Large volumes of sample can be used.
b. The method is fairly rapid.
c. All measurements are made directly and independently of others.
d. Standard retorting equipment and procedures can be used.
e. Care is not required to differentiate pore water from total water.
f. Oil volume correction curves are not required.
g. Duplicate samples are more easily obtained since a large volume of crushed material may be prepared, and samples obtained, by the “cone and quarter” technique.

4.7.6 Limitations

Limitations of this method include:

a. Oil yields may be dependent on rate of heating.
b. Due to emulsions, there may be difficulty in reading the meniscus in the collection glassware.

4.7.7 Accuracy/Precision

Accuracy/precision of this method is:

a. The accuracy of the oil and water volumes is ±2.5 percent of the measured volumes.
b. The reproducibility should fall in the ±0.5 gal/ton range for low yield samples (e.g., 3 to 10 gal/ton) and ±1.0 gal/ton in high yield samples (e.g., 20 to 40 gal/ton).

4.7.8 Calibration

For glassware calibration procedures, refer to 4.2.1.8.

4.8 GYPSUM-BEARING CORES

4.8.1 Principles of Analysis

Cores containing gypsum ($CaSO_4.2H_2O$) often are from carbonate formations. Such formations are frequently heterogeneous with vugs and/or fractures so that full-diameter core analysis procedures are preferred. However, determination of fluid saturations of gypsum-bearing cores by the distillation extraction method (Dean-Stark) for full diameter cores (see 4.3.2) using toluene as the solvent is not recommended. At the boiling point of toluene, gypsum dehydrates. If the core contains a significant amount of gypsum, erroneously large water volumes and incorrect pore volumes will be measured. The pore volume used to determine saturation will also be in error because of the increase in pore volume due to the dehydration of the gypsum. The volume of oil extracted from the sample is not affected by the presence of gypsum.

There are emerging technologies, e.g., NMR that show promise for the quantification of gypsum and in the future

may be offered as basic services by commercial laboratories. The Karl Fischer titration method (see 4.4.1) can be used to analyze gypsum-bearing cores by determining water content of flushed solvents during cool solvent extraction. However, this method is generally not available at service companies for analysis of a large number of core samples. The qualitative method described in the following paragraph can be performed by commercial laboratories.

The qualitative method presented here is based on adjustment of retort saturations for the dehydration of the gypsum by comparing retort and low temperature porosities and weights on adjacent samples. A plug sample is cleaned by pressure cycling solvents at low temperature so that oil and water are extracted from the sample without removing the chemically-bound water held within the gypsum. Boyle's law porosity is then measured on the cleaned sample. After the Boyle's law porosity is determined, the plug sample is retorted to determine the amount of bound water in the sample. The difference in porosity between the low and high temperature extractions is used to calculate the bulk volume of gypsum contained in the core sample. The volume of water given off in the dehydration of gypsum is approximately 1.27 times the increase in pore volume that results from shrinkage of gypsum (grain density = 2.32 g/cm^3) as it converts to anhydrite (2.89 to 2.98 g/cm^3). Retort saturation measurements are obtained on trimmed ends of the plug sample or on material adjacent to where the plug was drilled. The retort method at atmospheric pressure (see 4.2) removes the fluids in the core, and water of crystallization, from the gypsum. The summation-of-fluids porosity is determined from the retort data. Water saturations determined by the retort method are then adjusted. The adjustment will result in a decrease in water saturation. This adjustment is qualitative because carbonates are noted for being heterogeneous; so the companion samples may not contain the same amount of gypsum.

4.8.2 Apparatus

Appropriate apparatus for this method include:

a. High temperature retorting equipment (see 4.2.1.2).

b. Low temperature cleaning apparatus, e.g., pressure saturator large enough to pressure cycle a solvent through several samples at one time.

c. Boyle's law porosimeter.

4.8.3 Procedures

The core to be analyzed should be laid out on a table and fitted together as in standard practice (see 3.2.4). From each foot of core to be analyzed, drill a plug sample. If 1-inch diameter plugs are used, the samples should be at least one inch long. After the plugs are trimmed to length, they should be weighed. Check fluorescence of the plug and record. The initial visual estimate of hydrocarbon saturation will allow the analyst to better evaluate the sample during the pressure-cleaning phase. The plug should be cleaned by a low temperature method that minimizes gypsum alteration.

The fluid saturation of the trimmed plug ends and/or the remaining material from the segment of core from which the above mentioned plug has been drilled, is determined by the retort method at atmospheric pressure (see 4.2.1.3). Use the maximum amount of core material possible for the best precision. This will yield high temperature fluid saturation data and the summation-of-fluids porosity may be calculated (see 5.3.2.2.2 and 4.2.1).

The drilled plug samples should be cleaned in a pressure vessel using an azeotrope mixture of $^1/_3$ acetone, $^1/_3$ cyclohexane, and $^1/_3$ methanol. The samples are placed in the pressure vessel and totally immersed in the azeotropic mixture. Air or nitrogen pressure is applied to the top of the mixture forcing the azeotrope into the pore spaces of the plug samples. The pressure is cycled to force the mixture in and out of the plugs. After approximately 24 hours, the fluorescence of the samples should be checked to see if extraction is complete. Plugs with oil fluorescence should be returned to the pressure vessel for another cleaning cycle. If there is no visible oil fluorescence, the samples are deemed clean. The samples are placed in a vacuum oven at 120°F (49°C) until weight stabilizes. Samples should stay in the vacuum oven for at least 24 hours to remove the azeotrope mixture from the samples. Low temperatures are used during the cleaning and drying phases of this process to minimize alteration of gypsum in the plug samples. After the plug samples have been cleaned and dried, Boyle's law porosity is determined.

After the Boyle's law porosity is measured on the plug sample, the plug is retorted to remove the water bound in the gypsum. After retorting, the plug is reweighed and the porosity of the plug sample is remeasured by Boyle's law. Comparison of the retort porosity with the low temperature porosity identifies zones with gypsum. When the high temperature porosity is much greater than the low temperature porosity, the gypsum content is very high. When the two porosities agree, the gypsum content is low.

The oil volume extracted by the retort method is not affected by the presence of gypsum. However, the pore volume used to calculate the oil saturation must be corrected for the presence of gypsum. The oil saturation can be calculated from the corrected pore volume and the oil volume.

4.8.4 Precautions

Care should be exercised drying samples taken from the pressure saturator to prevent dehydration of any gypsum present. Monitor vacuum oven heat with a temperature probe.

4.8.5 Calculations

Refer to Retort Method at Atmospheric Pressure (see 4.2) and Summation-of-Fluids Porosity Calculations (see

5.3.2.2.2). Refer to 5.3.2.1.1, "Boyle's Law Porosity Calculations".

$$S_{oc} = V_o / (V_{br} \times \phi_{lt})$$

Where:

V_o = volume of oil obtained from the retorting of the trimmed ends or material adjacent to the plug sample, cm^3.

V_{br} = bulk volume of the retorted material, cm^3.

ϕ_{lt} = porosity of the plug after low temperature extraction, fraction.

S_{oc} = adjusted oil saturation, fraction.

If the gas saturation is assumed to be zero, then the water saturation, S_{wc}, is:

$$S_{wc} = 1 - S_{oc}$$

If the gas saturation is non-zero, the water saturation can be calculated by assuming a grain density for anhydrite:

$$\Delta V_w = (\rho_a / \rho_g) \times (V_{br} \times \Delta\phi)$$

$$\Delta_{wcorr} = V_w - \Delta V_w$$

$$S_{wc} = V_{wcorr} / (V_{br} \times \phi_{lt})$$

Where:

Swc = water saturation adjusted for the gypsum dehydration, fraction.

ΔV_w = volume of water from the dehydration of the gypsum, cm^3.

ρ_a = grain density of anhydrite, g/cm^3.

ρ_g = grain density of gypsum, g/cm^3.

$\Delta\phi$ = difference in porosity between the high and low temperature extractions of the plug sample, fraction.

V_{wcorr} = volume of water adjusted for the gypsum dehydration, cm^3.

V_w = Volume of water obtained from the retorting of the trimmed ends or material adjacent to the plug sample, cm^3.

Alternatively, the saturations can be expressed in terms of sample weights:

$$f_{wat} = W_{wat} / (W_{wat} + W_{oil})$$

$$W_{ewat} = (W_{po} - W_{pr}) \times f_{wat}$$

$$W_{eoil} = (W_{po} - W_{pr}) \times (1 - f_{wat})$$

$$W_{gyp} = W_{pl} - W_{pr}$$

$$V_{wcorr} = (W_{ewat} / \rho_{br}) - (W_{gyp} / \rho_{gw})$$

$$S_{oc} = (W_{eoil} / \rho_{oil}) / V_{plt}$$

$$S_{wc} = V_{wcorr} / V_{plt}$$

Where:

W_{po} = weight of the plug after drilling, g.

W_{pl} = weight of the plug following low temperature extraction, g.

W_{pr} = weight of the plug following retorting, g.

W_{wat} = weight of water determined by the retort extraction of the end pieces or adjacent material, g.

W_{oil} = weight of the oil determined by the retort extraction of the end pieces or adjacent material, g.

f_{wat} = weight fraction of water removed by retort extraction, fraction.

W_{ewat} = estimated water content of the plug sample including the bound gypsum water, g.

W_{eoil} = estimated oil content of the plug sample, g.

W_{gyp} = gypsum water of hydration in the plug sample, g.

ρ_{br} = density of the formation brine, g/cm^3.

ρ_{gw} = density of the gypsum bound water, g/cm^3.

V_{wcorr} = volume of water adjusted for the gypsum dehydration, cm^3.

ρ_{oil} = density of the formation oil, g/cm^3.

V_{plt} = pore volume of the plug sample following low temperature cleaning, cm^3.

S_{oc} = oil saturation calculated for the plug sample accounting for the change in pore volume due to the dehydration of the gypsum, fraction.

S_{wc} = water saturation calculated for the plug accounting for the bound gypsum water and the change in pore volume due to the dehydration of the gypsum, fraction.

4.8.6 Advantages

Advantages of this method include:

a. Damage to gypsum is minimized when the sample is cleaned by pressure cycling. Pore volume measurements used to calculate saturations are more representative if the gypsum is not damaged.

b. Gypsum content can be estimated by porosity comparisons.

4.8.7 Limitations

Limitations of this method include:

a. The low temperature cleaning process can be time consuming depending on the oil gravity and the permeability of the core material.

b. The retort oil and water saturation values are not as precisely determined as for samples that contain no gypsum.

4.8.8 Accuracy/Precision

Accuracy/precision of this method is:

a. If the water of crystallization is preserved during pressure cycling, pore volume determination will have the standard accuracy.
b. The precision of oil and water saturation calculations determined by retort method will be reduced due to the estimation of gypsum content by porosity comparisons.
c. Porosities obtained by the Boyle's Law helium method are more reliable than those obtained by the summation-of-fluids method. When porosity values obtained by Boyle's Law and summation-of-fluids methods are in good agreement, the saturations are assumed to be more reliable than in intervals in which there are large differences between the two porosities.

4.8.9 Calibration

The equipment used to measure porosity by both methods must conform with standards for the appropriate porosity method.

4.9 HISTORICAL

4.9.1 Vacuum Retort Method

The vacuum retort method for obtaining core fluid saturations is a full-diameter technique. This method is used extensively in "hard rock" regions, e.g., West Texas, where production is predominately from carbonate formations. Although several core analysis companies still maintain equipment for this procedure it is not a recommended procedure, having been replaced by the distillation extraction method (see 4.3).

The procedure consists of distilling off the pore fluids at a maximum temperature of 450°F (232°C) while maintaining a partial vacuum on the system. The fluid collection glassware should be immersed in an alcohol/dry ice bath maintained at approximately –75°F (–59°C) to prevent the vapors from being lost through the vacuum pump.

The procedure lends itself to the determination of the pore volume by the summation-of-fluids method. This is possible because the first step consists of saturating the unoccupied or gas-filled pore spaces with fresh water. The unoccupied pore volume is determined by gravimetric difference. An oil volume correction factor is employed and the pore water is determined by difference. The three values comprise the pore volume and the collected fluids can be expressed accordingly. A porosity can be calculated with a bulk volume determined on the same sample used for the distillation process.

One of the prime disadvantages of this method is the condition of the cores after the distillation process. It is not uncommon for the cores to be black from coked oil and no cleaning process will restore the samples to a condition whereby they could be deemed suitable for use in further testing procedures, e.g., a Boyle's Law porosity, capillary pressure test, relative permeability, etc. For this and other reasons, the authoring Subcommittee decided not to record this as a recommended procedure.

4.10 REFERENCES

1. Hensel, Jr., W. M., "An Improved Summation-of-Fluids Porosity Technique," *Society of Petroleum Engineers Journal*, April 1982, pp. 193-201, Society of Petroleum Engineers, Richardson, Texas.
2. Brown, G., *The X-Ray Identification and Crystal Structures of Clay Minerals*, Mineralogical Society (Clay Minerals Group), London, 1961.
3. Mackenzie, R. C., *Differential Thermal Analysis*, Academic Press Inc., New York, 1970.
4. Alberta Oil Sands Index (AOSI)-3573, "Round Robin Study of Analytical Procedures of Various Laboratories on Assay Analysis of Athabasca Tar Sands," 1974, Alberta Oil Sands Technology and Research Authority (AOSTRA) Information Center, Edmonton, Alberta, Canada (telephone 403/427-8382). Published by the Canadian Petroleum Association Oil Sands and Heavy Oil Committee.
5. Gidman, J., and Conner, F. J., "The Effects of Freezing on Tracer Distribution in Cores," presented at EUROCAS III, Advances in Core Evaluation, 1992.
6. ASTM D 1364-90: *Test Method for Water in Volatile Solvents (Fischer Reagent Titration Method)*, American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428.
7. ASTM D 4377-88: *Method for Water in Crude Oils (Karl Fischer) Titration*, American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428.
8. ASTM D2013: *Method for Preparing Coal Samples For Analysis*, American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428.
9. ASTM D2961: *Total Moisture in Coal Reduced to No. 8 (2.38 mm) Top Sieve Size (Limited Purpose Method)*, American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428.
10. ASTM D3302: *Test Method for Total Moisture in Coal*, American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428.

4.11 BIBLIOGRAPHY

a. Baldwin, B. A., and Yamanashi, W. S., "NMR Imaging of Fluid Saturation Distributions in Cores," Society of Core Analysts, 3rd Annual Conference, Dallas, Texas, August 20–21, 1989.
b. Blackband, S.J., Mansfield, P., Barnes, J. R., Clague, A. D., and Rice, S. A., "Discrimination of Crude Oil and Water

in Sand Bore Cores With NMR Imaging," *Society of Petroleum Engineers Formation Evaluation Journal*, Volume 1, No. 1, pp. 31–34, February 1986.

c. Brown, A., and Marriott, F. T., "Use of Tracers To Investigate Drilling-Fluid Invasion and Oil Flushing During Coring," *SPE Reservoir Engineering*, 1988, pp. 1317-1322.

d. Coles, M. E., Muegge, E. L., and Marek, B. F., "Use of Attenuation Standards for CAT Scanning Applications Within Oil and Gas Production Research," Society of Core Analysts Symposium, SCA-9223, *Transactions*, 1992.

e. DiFoggio, R., Calkin, C. L., Ellington., W. E., Setser, G. G., "Improved Method for Extraction and Quantification of Hydrocarbon Content of Sponge Core Liners," Society of Core Analysts, 4th Annual Conference, SCA-9019, Dallas, Texas, August 14–16, 1990.

f. Graue, A., Kolltvelt, K., Llen, J. R., and Skauge, A., "Imaging Fluid Saturation Development in Long-Core Flood Displacements," *Society of Petroleum Engineers Formation Evaluation Journal*, December 1990, pp. 406-412.

g. Levine, J. E., "New Pressure-coring Techniques Accurately Evaluate Reservoir," *Oil and Gas Journal*, January 21, 1980, pp. 63-66.

h. Lovelace, K. B., and Armstrong, F.E., "A Study of Core Invasion by Water-Base Mud Filtrate Using Tracer Techniques," *Drilling and Production Practice* 1961, API, 104-113.

i. Oak, M. J. and Erlich, T., "A New X-ray Absorption for Measurement of Three-phase Relative Permeability," SPE Paper 14420 presented at the Society of Petroleum Engineers Annual Technical Conference, 1985.

j. Owen, L. B., "Rapid, Room-Temperature Quantitative Analysis of Oil in Sponge Core," Society of Core Analysts, 4th Annual Conference, SCA-9021, Dallas, Texas, August 14–16, 1990.

k. Park, A., and Devier, C. A., "Improved Oil Saturation Data Using Sponge Core Barrels," Society of Petroleum Engineers, Production Operation Symposium, SPE-11550, Oklahoma City, 1983.

l. Parson, R. W., "Microwave Attenuation—A New Tool for Monitoring Saturations in Laboratory Flooding Experiments," *Society of Petroleum Engineers Journal*, Volume 15, No. 4, pp. 302–310, 1975.

m. Tibbitts, G. A., Reed, M. G., and McCarter, M., "New Coring System Reduces Filtrate Invasion, Advances in Core Evaluation—Accuracy and Precision in Reserves Estimation," EUROCAS I, London, May 21–23, 1990, pp. 55–77.

n. Vinegar, H. J., Tutunjian, P. N., Edelstein, W. A., and Roemer, P. B., "13C NMR of Whole Cores," SPE Preprint 19590, 64th Annual Technical Conference, San Antonio, Texas, October 8–11, 1989.

o. Vinegar, H. J., Tutunjian, P. N., Crabtree, P. T., Raffaldi, F. J., DiFoggio, R., and Edelstein, W. A., "NMR Spectroscopy of Tight, Gypsum-Bearing Carbonates," *The Log Analyst*, September–October 1991, Volume 32, #5, pp. 527–535.

p. Wellington, S. L., and Vinegar, H. J., "X-ray Computerized Tomography," *Journal of Petroleum Technology*, Volume 39, No. 8, pp. 885–898, August 1987.

SECTION 5—POROSITY DETERMINATION

CONTENTS

Page

5 POROSITY DETERMINATION 5-1
5.1 General 5-1
5.2 Bulk Volume Measurement 5-3
5.3 Pore Volume Measurements 5-9
5.4 Historical Procedures 5-19
5.5 Organic-Rich Rocks 5-20
5.6 References 5-23

Figures
5-1 Total and Effective Pore Volumes as Defined by Core and Log Analysts 5-2
5-2 Archimedes Mercury Immersion Apparatus 5-4
5-3 Volumetric Mercury Displacement Pump 5-6
5-4 Archimedes (Buoyancy) With Fluids Other Than Mercury—Apparatus 5-8
5-5 Double-Cell Boyle's Law Porosimeter 5-11
5-6 Schematic of Isostatic Load Cell for Direct Pore Volume Determination 5-13
5-7 Laboratory Loading Schematic 5-16
5-8 Laboratory and Reservoir Loading Schematic 5-17
5-9 Volumetric Adsorption Measurement Method 5-22

Tables
5-1 Bulk, Grain, and Pore Volume Directory 5-2
5-2 Jacketing Material for Poorly Consolidated/Unconsolidated Core Samples . . . 5-3
5-3 Density of Mercury Versus Temperature 5-5

5 Porosity Determination

5.1 GENERAL

5.1.1 Introductory Comments/Equations

5.1.1.1 Porosity, which is defined as a ratio of the void-space volume to the bulk volume of the material, is an intrinsic property of all reservoir rocks. The amount of void space which can be occupied by hydrocarbons or water in a reservoir must be known for an intelligent estimate of the initial oil/gas in place. The precision with which porosity can be determined is largely a function of the methods used in these measurements. Several logging tools using either electrical, nuclear, density, or sonic methods are used for continuous porosity determination in the wellbore. The measurements obtained from these well logging tools should be calibrated against core porosities determined under simulated subsurface conditions, bearing in mind the volume of rock investigated by logging tools. Effects of core sample bias, sample frequency and sample volume must be taken into consideration. With one exception, all discussions refer to porosity determination at zero to <400 psi (2760 kPa) confining stress. The method to determine porosity at simulated overburden pressure is discussed in 5.3.2.2.1.2.

5.1.1.2 Unless otherwise stated, the analysis methods described are applicable to cylindrical core plugs drilled from consolidated and relatively homogeneous rock. Porosity from core analysis may commonly be arrived at by several different methods (see Table 5-1). Measurement of bulk volume (BV) and grain volume (GV) yields pore volume (PV) by difference, and porosity (ϕ) by:

$$PV = (BV - GV) \tag{1}$$

$$\phi = (BV - GV) / BV$$

Direct measurement of pore volume (PV) and grain volume (GV) yields:

$$\phi = PV / (PV + GV) \tag{2}$$

Direct measurement of pore volume (PV) and bulk volume (BV) yields:

$$\phi = PV / BV \tag{3}$$

5.1.1.3 In the laboratory, usually one of two types of porosity are measured; viz., effective porosity or total porosity. There is a difference between the historical definitions of these porosities and the current definition used by log analysts. This is further complicated by core analysis techniques designed to leave several molecular layers of clay- or mineral-bound water within the pore space (see Figure 5-1).

5.1.1.4 Historically, effective porosity has been defined as a measure of the connected voids. It is derived from either the difference between bulk volume and apparent grain volume determinations, or by a direct measurement of the connected void space. The measured volume of the connected void space may vary with sample preparation and the analytical method used. Total porosity is a measure of the total void space, both connected and isolated, in the rock sample. This can be determined by bulk volume, dry weight, and grain volume measurement on a disaggregated sample.

5.1.1.5 In the field of log interpretation, effective porosity has been defined as interconnected pore volume occupied by free fluids. In this definition, effective porosity does not include the volume of clay-bound or mineral-bound water, and in some analytical approaches, does not include the additional water held within the shale by capillary forces. Also in the field of log interpretation, total porosity is that volume occupied by all fluid (connected and isolated pore space) in the rock, including the volume occupied by the clay-bound water (see Figure 5-1).

5.1.1.6 Core analysis experience indicates that for most reservoir rocks there are few isolated pores, and hence there is very little or no measurable difference in historically defined effective and total porosity (see Figure 5-1). Thus, with these definitions, effective porosity determined from core analyses on a totally dried sample at 210° to 240°F (99° to 116°C) more closely corresponds to the total porosity as defined by the log analysts. In certain circumstances, such as volcanic rocks, there can be a measurable difference between the effective (connected) and total (connected and isolated) porosity from core analysis. In addition, core humidity drying techniques can be tailored to leave some amount of bound water on mineral surfaces.

5.1.1.7 There is no universal agreement between log analysts on the definition of effective porosity (see Figure 5-1). The lowest value for this parameter is obtained if all shale-associated water (adsorbed/anion free water plus capillary-held water within the shale) is excluded as noneffective pore space. A higher value is obtained if only the adsorbed/anion-free water (defined as a function of both salinity and cation exchange capacity or the concentration of clay counterions per unit pore volume, meq/cm^3) is excluded. Core porosities based upon humidity-controlled, dried samples are normally between this higher value and the total porosity. At this time, it is difficult to select laboratory conditions that yield effective porosity on core samples as defined by log analysts.

5.1.1.8 Another facet of porosity measurement, which in fact applies to the entire field of core analysis, is the selection of conventional (small sample) analysis versus full-diameter

analysis. The decision as to which type should be used would ideally be based entirely on the homogeneity of the formation being analyzed. Many sandstones are sufficiently homogeneous so that a small sample can be considered representative of the analysis increment. On the other hand, when the formation is heterogeneous as to pore structure or lithology, such as in vugular or fractured carbonates, full-diameter core techniques are more appropriate. Therefore, the size of the sample required to adequately represent the pore structure and lithology of the core material, as well as the sampling strategy should control the type of analysis used.

5.1.2 Poorly Consolidated/Unconsolidated Core Samples

5.1.2.1 Poorly consolidated/unconsolidated samples present unique requirements. The term "poorly consolidated" encompasses a wide range of core materials, from friable samples to completely unconsolidated samples with no apparent cementation between grains. For the purposes of this discussion, "poorly consolidated" refers to any plug sample that requires mounting in an appropriate jacketing material to withstand the cleaning, preparation, and measurement process.

5.1.2.2 Plug samples are commonly jacketed using a cylinder of metal or polymer about the circumference of the sample. Table 5-2 lists some common jacketing material along with their advantages and disadvantages.

Table 5-1—Bulk, Grain, and Pore Volume Directory

Method Number	Title	Purpose
5.2.1	Archimedes (Buoyancy) Mercury Immersion	Bulk volume measurement
5.2.2	Mercury Displacement	Bulk volume measurement
5.2.3	Caliper	Bulk volume measurement
5.2.4	Archimedes (Buoyancy) with Fluids Other than Mercury	Bulk volume measurement
5.2.5	Bulk Volume by Summation of Grain and Pore Volume	Bulk volume determination
5.3.1	Total Pore Volume from Disaggregated Sample Grain Density	Grain volume measurement
5.3.2.1.1	Boyle's Law Double-Cell Method	Grain volume measurement
5.3.2.2.1	Boyle's Law Single-Cell Method	Direct pore volume measurement
5.3.2.2.2	Summation of Fluids Porosity	Summary bulk and pore volume measurements
5.3.2.2.3	Liquid Saturation Method	Direct pore volume measurement
5.4	Historical Procedures	Bulk, grain and pore volume measurements
5.5	Organic Rich Rocks	Pore volume and adsorbed gas

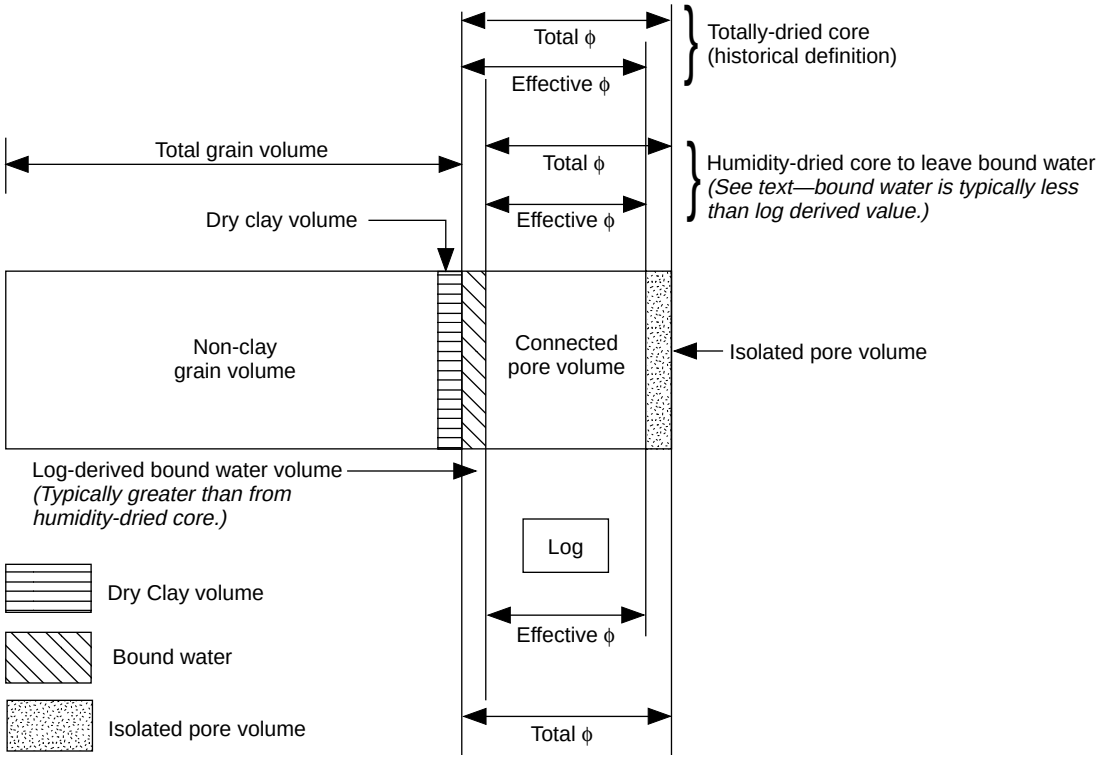


Figure 5-1—Total and Effective Pore Volumes as Defined by Core and Log Analysts

Table 5-2—Jacketing Material for Poorly Consolidated/ Unconsolidated Core Samples

Jacketing Material	Advantages	Disadvantages
Lead Sleeves	Malleable, conforms well to sample	Interacts with mercury and some brines
Teflon® tape	Inert	Difficult to apply, porous and permeable
Heat shrink Teflon tubing	Inert, easy to apply	May not conform well to sample at low confining stress, possible sample alteration from heat and radial stress applied as tubing shrinks
Aluminum sleeve	Malleable, conforms reasonably well to samples	Interacts with some brines and may not conform well at low confining stress
Paint-on epoxy or other coating	Cheap, easy to apply	Interacts with solvents, poor mechanical strength, can imbibe into sample
Tin	Malleable, conforms well to sample	Interacts with mercury and some brines

5.1.2.3 To allow flow, the plug faces are covered with screens or other porous material. The mesh size of the screens must be small enough to prevent grain loss, but large enough to avoid plugging by mobile fines. Screens with a mesh size of 200 or 120 are typically used. Samples with some consolidation require only a single screen. Less consolidated samples may require more sophisticated end treatments. For instance, two screens may be used. One screen is of finer mesh to prevent grain loss, and a second less flexible, coarser screen is added for mechanical strength. The material chosen for the screens must be inert to the solvents and brines used for cleaning and testing. A high quality stainless steel such as 316 or Monel is recommended.

5.1.2.4 The apparent pore and grain volume of the jacketing material and end screens must be determined and accounted for in the porosity measurement. The volume of jacketing material on an individual sample can be determined by applying the Boyle's Law Double-Cell Method for grain volume (see 5.3.2.1.1) or the Archimedes method for bulk volume using either mercury (where suitable, see 5.2.1) or other solvents (see 5.2.4). In practice, it is easier to determine the density of the jacketing material and then determine the weight of jacketing material applied to each sample. Jacket volume can then be computed. This method assumes that the density of the material is constant from piece to piece.

5.1.2.5 The "grain" and "pore" volume of the screens must be determined as well. The grain volume can be determined by taking a number of identical screens and measuring their solid volume in a Boyle's Law Double-Cell apparatus. The value determined is then divided by the number of screens tested and this average value is applied to each sample. The pore volume is more sensitive to the test method applied. For the Boyle's Law method, the pore or void volume in the screens can be determined by mounting a solid non-porous plug in the same manner as the real samples are prepared. The Boyle's Law Single-Cell method will then yield the screens' void volume.

5.1.2.6 Once a sample is packaged with a jacketing material and screens, it can be tested using many of the same techniques applied to consolidated samples. In the following sections, comments about the applicability of each measurement method for jacketed poorly consolidated samples will be made in 5.2.1.3.

5.1.2.7 Since the physical properties of poorly consolidated samples are more sensitive to confining stress than consolidated samples, measurements performed at stresses below in-situ effective reservoir stress will not be as representative of the reservoir rock. Therefore, the recommended method for determining the porosity of poorly consolidated samples is to use the Boyle's Law Single Cell method for direct pore volume measurement at elevated confining stress (see 5.3.2.2.1.2), and the Boyle's Law Double-Cell method for grain volume (see 5.3.2.1.1).

5.2 BULK VOLUME MEASUREMENT (PLUG SIZE)

The bulk volume of a rock sample is required to determine the porosity of that sample. Bulk volume of a core plug can be determined by several methods. Measurement techniques include Archimedes immersion, mercury displacement, and calipering.

Bulk volumes can also be calculated by summing direct measurements of grain volume and pore volume.

The bulk volume of a sample selected for porosity measurement should preferably be at least 10 cm^3. Normally, samples are a right cylinder with diameters from 2.54 cm to 3.81 cm and lengths of at least 2.54 cm and 3.81 cm, respectively. Irregular shapes may be used with proper precautions if samples of regular dimensions cannot be obtained.

5.2.1 Archimedes (Buoyancy) Mercury Immersion

5.2.1.1 Principle

A core plug is immersed in mercury and the volume of mercury displaced by the sample is determined gravimetrically (Archimedes' principle).

5.2.1.2 Advantages

Advantages of this method include:

a. Samples can be used for subsequent tests if no mercury penetration occurs.
b. The method is very accurate if careful technique is used and precise measurements are made.

5.2.1.3 Limitations

Limitations of this method include:

a. Trapping air around the samples will create errors and yield erroneously high bulk volumes.

b. Vuggy samples or extremely high permeability samples will be penetrated by the mercury resulting in low bulk volume values and rendering the samples unsuitable for further testing. Samples with a vugular surface or containing open fractures are not recommended for bulk volume analysis by mercury immersion. However, if it is necessary to analyze such samples using this technique, the sample surface must be sealed by coating or filling the vugs to prevent mercury penetration. This may not be an adequate treatment.

1. Materials such as heat-shrink Teflon tubing can be used for coating the cylindrical surfaces of a sample to cover the vugs. The volume of any coating must be subtracted from the total bulk volume measured, and air gaps on nonvugular surfaces must not exist between the tubing and the rock.
2. The surface vugs can be filled (e.g., with modeling clay, paraffin, or an epoxy) which will allow a direct measurement of the bulk volume when the sample is immersed under the mercury. Usually, filling the vugs is a destructive test and should be the last step in the analysis.
3. Calipering sample length and diameter, and computing bulk volume (see 5.2.3), is a method utilized for vuggy samples. Experience has shown that computed bulk volumes are often too high. Calibration techniques presented in 5.2.3.6 improve data accuracy.

c. The method is unsuitable for poorly consolidated/unconsolidated samples that are mounted in lead sleeves because of lead-mercury amalgamation. Mercury may also be trapped between jacketing materials and the sample; consequently, this is not a recommended procedure for jacketed samples.

5.2.1.4 Accuracy

The measurement of weight can be repeated within ±0.015 grams if air is not trapped when the sample is submerged and the temperature remains constant. Bulk volume measurements can be repeated to within ±0.01 cm^3.

5.2.1.5 Apparatus

The following apparatus are appropriate for this method:

a. Electronic, single-pan, analytical balance accurate to ±0.01 gm.

b. Mercury vessel large enough that plug positioned with the large dimension horizontal can be fully immersed without touching the sides of the container.

c. Mercury to fill the vessel so that the plugs can be fully immersed.

d. Adjustable, pronged fork with reference mark.

e. Thermometer.

5.2.1.6 Procedure

Bulk volume (BV) is measured by the Archimedes Mercury Immersion method with the apparatus shown in Figure 5-2. A cup of mercury is placed on a single-pan electronic balance, and a pronged fork is immersed to the fork reference mark. The mark is located such that the top of the core plug will be submersed 3 to 7 millimeters under the mercury. The balance is then tared. The fork is withdrawn from the mercury, and the plug to be measured is floated lengthwise on the mercury surface. The plug is then forced into the mercury with the fork to the same reference mark. The sample must not touch the side of the mercury vessel. The resulting weight represents the mass of the mercury displaced. Eighteen-gauge

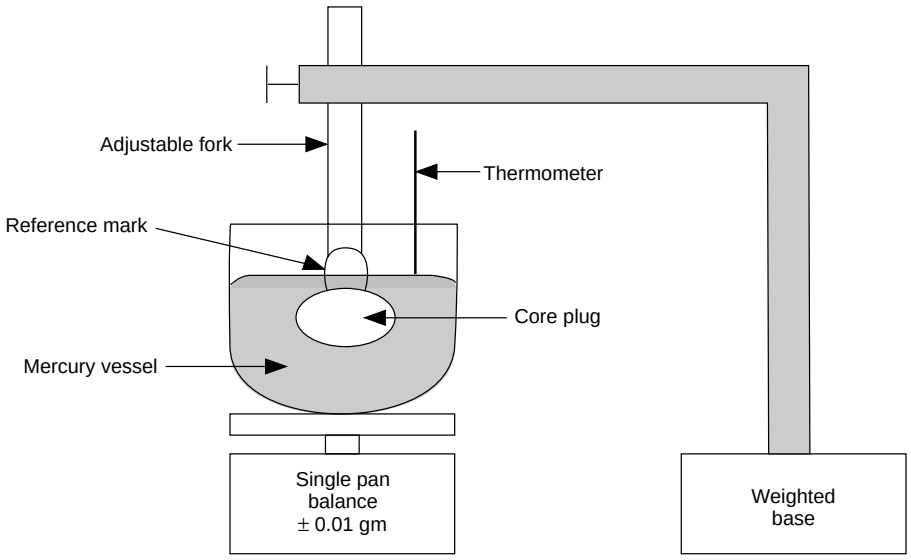


Figure 5-2—Archimedes Mercury Immersion Apparatus

(1.0-millimeter diameter) stainless steel wire is a suitable material for the fork. Heavier gauge wire has been observed to result in unstable weight measurements (changing weights with time) and long balance equilibrium times.

5.2.1.7 Calculations

The bulk volume is calculated using the following equation:

$$BV = \frac{\text{Mass of mercury displaced}}{\text{Density of mercury at measurement temperature}} \quad (5)$$

5.2.1.8 Precautions

A heavy weighted base for supporting the fork assembly is essential to hold the sample firmly in place to ensure accurate weights. It is important to use the correct mercury density for the mercury temperature. A temperature variation of 5°C will induce a systematic error of 0.02 percent in the bulk volume (see Table 5-3).

Table 5-3—Density of Mercury Versus Temperature[a]

°C	Hg Density gm/cm³	°C	Hg Density gm/cm³
18.0	13.5512	27.0	13.5291
19.0	13.5487	28.0	13.5266
20.0	13.5462	29.0	13.5242
21.0	13.5438	30.0	13.5217
22.0	13.5413	31.0	13.5193
23.0	13.5389	32.0	13.5168
24.0	13.5364	33.0	13.5144
25.0	13.5340	34.0	13.5119
26.0	13.5315		

[a]From *Handbook of Chemistry and Physics*, 71st Edition, 1990-1991, Editor-in-Chief: David R. Lide, Ph.D., CRC Press, Boca Raton, Florida.

In order to obtain stable weights, the fork should be of such design as to yield point contact on the sample. If the fork volume is too large relative to the plug volume, erroneous weights will be measured. Unstable weights may indicate an unsuitable fork or mercury invasion. Mercury contamination can be confirmed by comparing sample weights before and after mercury immersion.

5.2.2 Mercury Displacement (Volumetric Displacement Pump)

5.2.2.1 Principle

Sample bulk volume is measured by mercury displacement utilizing a volumetric displacement pump to which a stainless-steel sample chamber is attached.

5.2.2.2 Advantages

Advantages of this method include:

a. This procedure allows very rapid measurements.

b. The technique is employed as part of the Summation of Fluids porosity measurement.

c. Samples are suitable for subsequent tests if no mercury penetration or adsorption occurs.

5.2.2.3 Limitations

Limitations of this method include:

a. Air trapped around the sample will yield erroneously high bulk volumes.

b. Vuggy samples or extremely high permeability samples will be penetrated by the mercury resulting in low bulk volume values and rendering the samples unsuitable for further testing (see 5.2.3).

c. In most mercury displacement pumps, the sample is immersed approximately 50 millimeters beneath the mercury. This mercury height induces a pressure of about 1 psi (6.9 kPa) to the top of the plug. This may make the bulk volumes systematically low because of 1) conformance to microscopic surface roughness or 2) penetration into large pores. The depth of immersion is minimized (3 to 7 millimeters) in the Archimedes Mercury Immersion method (see 5.2.1).

d. The method is unsuitable for unconsolidated samples that are mounted in lead sleeves because of lead-mercury amalgamation. Mercury may also be trapped between jacketing materials and the sample; consequently, this is not a recommended procedure for jacketed samples.

5.2.2.4 Accuracy

The measurement can be reproduced to 0.01 cm³ if the pump has been calibrated and is zeroed for each sample.

5.2.2.5 Apparatus

Figure 5-3 illustrates a high pressure, volumetric mercury displacement pump. The sample chamber will hold samples up to approximately 25 cm³ in volume.

5.2.2.6 Procedure/Calculations

The bulk volume of a regularly or irregularly shaped core sample is obtained by mercury displacement. The chamber, with no sample in place, is filled with mercury to the reference level. The instrument volume read-out is zeroed. The mercury level is then lowered, the sample is inserted and the chamber is again filled to the reference level. The instrument volume read-out is obtained. The difference between this reading and the instrument zero is the bulk volume of the sample.

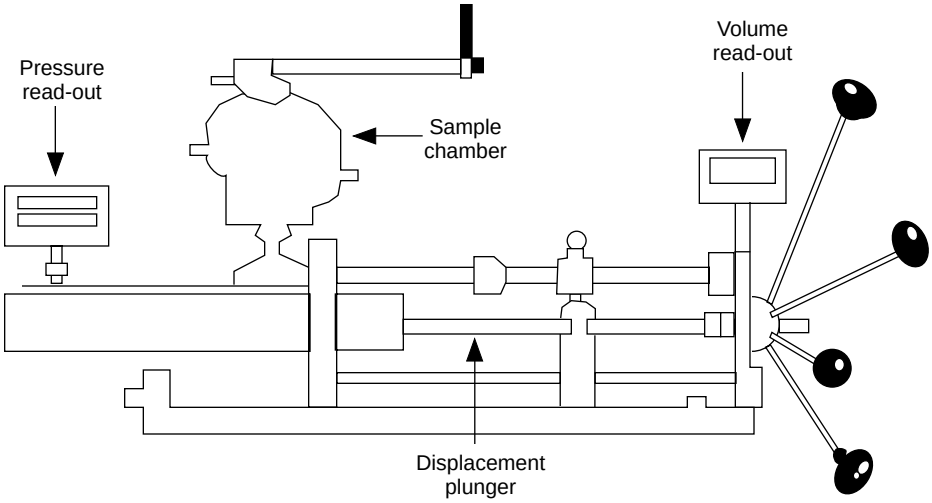


Figure 5-3—Volumetric Mercury Displacement Pump

5.2.2.7 Precautions

The mercury and chamber should be clean and free from surface films, solids, and oils.

5.2.2.8 Calibration

The mercury pump is calibrated with steel billets of known volume.

5.2.3 Caliper

5.2.3.1 Principle

Samples which are right cylinders or other regular shapes may be calipered to obtain bulk volume. A micrometer or vernier caliper, which can be read to the nearest 0.002 cm, can be used to measure length and diameter. A minimum of five measurements is recommended.

Note: Samples with surface vugs and/or open fractures may be analyzed using this approach if bulk volume data are calibrated on samples using the technique discussed in 5.2.3.6.

5.2.3.2 Advantages

Advantages of this method include:

a. The sample may be used for other tests.
b. The procedure is rapid.

5.2.3.3 Limitations

Limitations of this method include:

a. Samples with uneven shapes cannot be measured by this method.
b. Irregularities in sample surfaces may yield erroneously high bulk volumes.
c. Calipered bulk volumes are not generally recommended for porosity methods where $PV = (BV - GV)$. The accuracy of the bulk volume is not as critical where $f = PV / BV$ and the pore volume is determined by direct measurement (see 5.3.2.2). Where rock type (vuggy or fractured) dictates the use of the caliper technique, data should be calibrated as discussed in 5.2.3.6.
d. This method is not preferred for jacketed, poorly consolidated, or unconsolidated samples. If employed, corrections for screen and jacket thickness must be applied to both calipered length and diameter.

5.2.3.4 Accuracy

Length and diameter measurements of actual samples have been shown to be reproducible with a 99 percent confidence level (within 3 standard deviations) to within 0.15 millimeters for length and 0.04 millimeters for diameter. Bulk volumes can be repeated to 0.15 cm^3. See 5.2.3.6 for accuracy improvement.

5.2.3.5 Apparatus

Vernier or digital calipers may be utilized.

5.2.3.6 Procedure

The length and diameter of a cylinder or dimensions of a regularly shaped sample are measured at a minimum of five different positions to define any irregularities in the shape. Small deviations in the shape may be averaged out. Several samples without surface vugs and/or open fractures, whose gross dimensions cover the range of samples to be analyzed, should have their bulk volume determined by an appropriate immersion technique. Their bulk volume should then be determined using the exact caliper technique that will be used for measurement of subsequent samples. Correction factors may then be determined to calibrate calipered bulk volumes to those measured by immersion. These factors are then

applied to subsequent calipered bulk volume measurements to correct them to an equivalent immersion bulk volume.

Less time is required and more readings are taken if digital calipers are used. Compared to vernier calipers, the measurements are not as susceptible to human error since the readings are displayed as digital output. Digital calipers can be interfaced with a computer so the readings will be automatically recorded. About 10 length or diameter readings can be taken in less than 60 seconds.

5.2.3.7 Calculations

The cross-sectional area of the cylindrical sample is calculated from the average diameter and multiplied by the average length to obtain bulk volume. Appropriate formula can be used for noncylindrical samples.

5.2.3.8 Precautions

Precautions for this method include:

a. Diameter measurements should be taken at a minimum of five positions evenly spaced along sample length.
b. Length measurements should be obtained at a minimum of five positions around the sample's periphery.
c. Ensure that calipers are zeroed between successive measurements.
d. When measuring diameters and lengths of samples with surface irregularities, ensure that calipers do not intrude into surface vugs or fractures yielding measurements less than the true dimensions.

5.2.4 Archimedes (Buoyancy) With Fluids Other Than Mercury (e.g., Brine, Refined Oil, or Toluene)

5.2.4.1 Principle

A body placed in a liquid is buoyed up by a force equal to the weight of the displaced liquid.

5.2.4.2 Advantages

Advantages of this method include:

a. Accurate values can be attained if proper technique is used.
b. The sample can be totally saturated with liquid for other tests which may be desired.
c. If the sample is 100-percent saturated with a single fluid prior to the measurement of bulk volume, the pore volume, grain volume, and grain density can be calculated from recorded weights.

5.2.4.3 Limitations

Limitations of this method include:

a. The liquid may be unsuitable for subsequent tests and have to be removed.
b. Cores containing vugs should not be measured by this method.
c. Liquids which may leach the sample or cause swelling of the matrix cannot be used.
d. Vugs or large pores in extremely high permeability samples will be filled while submerged under the liquid, resulting in low bulk volume values (see 5.2.2.3, b.). Filling the vugs prior to measurement will allow a direct measurement of the bulk volume when the submerged weight is taken.
e. This is not a preferred technique for jacketed, poorly consolidated/unconsolidated samples because of the possibility of trapping extraneous fluid volume between the plug surface and the jacket.

5.2.4.4 Accuracy

The measurement of weight can be repeated within ±0.015 grams if air is not trapped when the sample is submerged and the temperature remains constant. The accuracy of bulk volume measurements will vary depending upon the density and volatility of the liquid used in the measurements. Good technique should yield repeatable bulk volume within 0.01 cm^3.

5.2.4.5 Apparatus

An analytical balance accurate to one milligram, fine wire cradle, liquid container, and thermometer are required. Figure 5-4 shows two possible experimental configurations.

5.2.4.6 Procedure

5.2.4.6.1 A sample is saturated with a liquid of known density, such as a nondamaging brine, light refined oil or a high boiling point solvent. The sample can be essentially 100-percent saturated with liquid by evacuating the pore space, introducing the saturant, and following with pressure saturation (see 5.3.2.2.3.6).

5.2.4.6.2 Excess liquid is carefully removed (avoiding grain loss) from the sample and the saturated sample is weighed in air. When removing excess liquid from the sample surface, precautions must be taken to ensure that fluids are not removed from pores exposed at the surface. Materials (such as dry towels) which remove liquid from surface pores due to capillary action should be avoided, as should any mechanical method such as violent shaking. Rolling the sample over a lint free cloth or paper towel dampened with the saturating liquid, or carefully wiping the sample with the dampened cloth or the fingers are acceptable methods for removing excess liquid.

5.2.4.6.3 A beaker is filled with the saturating liquid. A fine wire (maximum diameter of 1.0 millimeter) cradle attached to the stirrup of a balance is first submerged under the liquid to a reference mark, and the balance is tared. The sample is then placed on the cradle, submerged to the reference mark, and a submerged weight of the sample is obtained (see Figure 5-4A).

5.2.4.6.4 Figure 5-4B illustrates an alternate procedure. A beaker is filled with the saturating liquid and placed on a balance. A fine wire cradle is then lowered into the liquid to the reference mark, and the balance is tared. The saturated sample is then placed on the cradle, submerged to the reference mark, and the immersed weight of the sample is obtained.

5.2.4.6.5 A check on the pore volume can be made by taking (a) the difference in the weight of the 100-percent saturated sample in air and the dry weight, and (b) dividing by the saturating liquid density.

Note: It is not essential for the sample to be 100-percent saturated with the liquid for the determination of the bulk volume. The technique provides an accurate bulk volume as long as the sample is not imbibing liquid when the weight is taken while suspended under the liquid. However, if the sample is not 100-percent saturated, the pore volume, grain volume, and grain density calculated from the weights will be incorrect.

5.2.4.7 Calculations

Bulk volume from the apparatus shown in Figure 5-4A equals the initial weight of the saturated sample (or partially saturated sample with stabilized weight) in air minus the weight when submerged, divided by the density of the immersion liquid. Bulk volume from the apparatus shown in Figure 5-4B equals the immersed weight of the saturated sample divided by the density of the immersion fluid.

A

$$BV = \frac{\text{Saturated Weight} - \text{Immersed Weight}}{\text{Density of Immersion Fluid}}$$

B

$$BV = \frac{\text{Immersed Weight}}{\text{Density of Immersion Fluid}} \quad (6)$$

5.2.4.8 Precautions

Precautions for this method include:

a. Care must be taken in the design of the apparatus. The arrangement should be such that both the samples and the wire cradle are totally immersed during measurement, are not supported in any way by the immersion container, and only a length of straight wire (maximum diameter of 1.0 millimeter) penetrates the surface of the liquid. The wire and fluid should be clean.

b. It is essential to use the correct fluid density for the temperature of the liquid at the time of the submerged weight measurement. The density of the fluid should be known to at least the nearest 0.005 gm/cm^3. An error of 0.005 gm/cm^3 results in a porosity bias of 0.5 porosity points (porosity expressed as percent) and a grain density bias of 0.01 gm/cm^3. The density of the fluid can be measured with a pycnometer, an electronic digital density meter, various specific gravity balances, or it can be calculated using a silicon standard available from the National Institute of Standards and Technology.

$$V_{SS} = \frac{W_{SS}}{\rho_{SS}} \quad (7)$$

Where:

V_{SS} = silicon standard volume.
W_{SS} = air weight of silicon standard.
ρ_{SS} = silicon standard density from National Institute of Standards and Technology.

$$\rho_F = \frac{W_{SS} - W_{ISS}}{\rho_{SS}} \quad (8)$$

Where:

ρ_F = density of fluid in which silicon is immersed.
W_{ISS} = immersed weight of silicon standard.

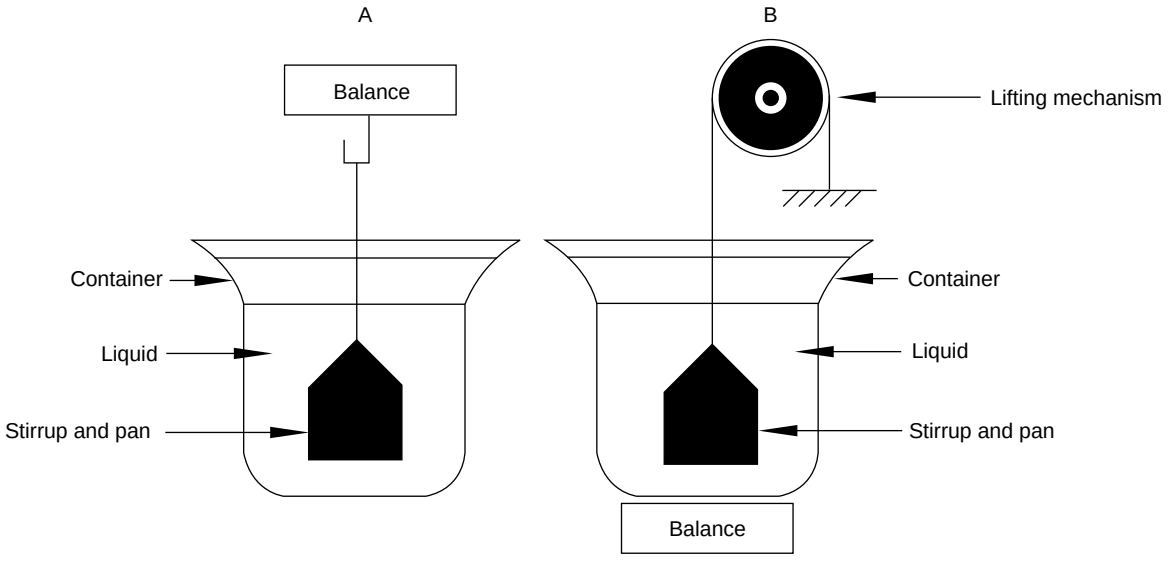


Figure 5-4—Archimedes (Buoyancy) With Fluids Other Than Mercury—Apparatus

c. It is probable that unstable decreasing weights indicate that fluid is imbibing into the sample, which will result in an erroneously low bulk volume.
d. Temperature fluctuations should be minimized as immersion liquid density will vary.
e. Care should be used in wiping the surfaces of the saturated sample so as to avoid grain loss or excess saturant removal. See 5.2.4.6.2 for suitable techniques.

5.2.5 Bulk Volume Calculated By Summing Direct Measurements of Grain Volume (5.3.2.1.1) and Pore Volume (5.3.2.2.1)

5.2.5.1 Calculations

The basic calculation is:

$$BV = GV + PV \quad (9)$$

5.3 PORE VOLUME MEASUREMENTS

Total pore volume is the total void space, both connected and isolated, in a rock sample.

5.3.1 Total Pore Volume From Disaggregated Sample Grain Density

The total pore volume equals the difference between the sample bulk volume and the disaggregated grain volume. Disaggregation is done to expose any isolated pore volume.

5.3.1.1 Dry Method for Grain Density/Grain Volume

5.3.1.1.1 Principle

The bulk volume of a cleaned sample is first determined (see 5.2). The sample is then dried, weighed, and disaggregated. After disaggregation, a weighed portion is placed into a Boyle's Law porosimeter to determine the grain volume (see 5.3.2.1.1). The grain volume of the total sample is calculated using the ratio of the dry weight of the consolidated sample to the dry weight of the disaggregated sample placed in the porosimeter.

5.3.1.1.2 Advantages

The advantages of this method are:

a. Fast.
b. Sample can be used for supplemental measurements requiring disaggregated sample. See Section 7, Supplementary Tests.

5.3.1.1.3 Limitations

The limitations of this method are:

a. Drying required.
b. Not viable for gypsum-bearing rock.
c. Not viable for rocks containing minerals such as halite that are soluble in the cleaning fluid.
d. Technique assumes any isolated pore volume remains isolated during the time the sample is dried.
e. If water fills the isolated pore space and is not removed while drying the sample, the calculated grain volume (*GV*) will be erroneously high.

5.3.1.1.4 Accuracy

Technique is capable of supplying grain density within ±0.01 g/cm^3. No comparative porosity data are available, but technique is expected to supply porosity within ±0.4 porosity units, or better.

5.3.1.1.5 Apparatus

The following apparatus are appropriate for this method:

a. Pulverizer with adjustable tolerance between the grinding plates.
b. U.S. Bureau of Standards 60-mesh sieve.
c. Analytical balance, with accuracy to 0.1 milligram.
d. Boyle's Law porosimeter modified to handle powdered samples. (1) Sample Cup cover (to keep the powdered sample in the cup).

5.3.1.1.6 Procedure

The procedure for this method is as follows:

a. Sample Preparation.
   1. Clean.
   2. Dry at 225°F (107°C).
   3. Cool in Desiccator.
   4. Weigh the sample and record weight (*W*).
   5. Repeat the above (steps 2 through 4) until weight (*W*) has stabilized.
   6. Determine Bulk Volume (*BV*) (see 5.2).
b. Crush the sample and pass the crushed sample through a 60-mesh sieve.
c. Dry a portion of the sample for Grain Volume measurement at 210° to 240°F (99° to 116°C) to a constant weight (W_d).
d. Determine the Grain Volume (GV_d) of the disaggregated sample portion by the Boyle's Law Double-Cell method (see 5.3.2.1.1).
   1. Calibrate the porosimeter with the sample cup cover in place.
   2. Place a known weight (W_d) of crushed and dried sample into the sample cup.
   3. Measure the Grain Volume of the disaggregated and dried sample portion with the cover in place.

5.3.1.1.7 Calculations

The calculations for this method are as follows:

$$PV = BV - GV \quad (10)$$

Where:

PV = total pore volume.
BV = bulk volume of original consolidated sample.
GV = grain volume of sample calculated by equation b.

$$GV = GV_d \times W/W_d \quad (11)$$

Where:

GV_d = measured grain volume of disaggregated sample portion placed in porosimeter.
W = original sample weight.
W_d = weight of dry, disaggregated sample portion placed in porosimeter cup.

$$GD = W_d/GV_d \quad (12)$$

Where:

GD = grain density of sample portion.

5.3.1.1.8 Precautions

Precautions for this method include:

a. The portion of rock selected for grain density measurement must be representative of the total sample.
b. See 5.3.2.1.1.8, Precautions for Boyle's Law Double-Cell (matrix cup) method for grain volume, items a through f.

5.3.1.1.9 Calibration

See 5.3.2.1.1.9, Calibration for Boyle's Law Double Cell (matrix cup) method for grain volume.

5.3.2 Effective Pore Volume of Aggregated Samples

Effective pore volume can be computed by subtracting the measured grain volume from the sample bulk volume, or by direct measurement of sample void volume.

5.3.2.1 Grain Volume Measurement

5.3.2.1.1 Boyle's Law Double-Cell (Matrix Cup) Method for Grain Volume

5.3.2.1.1.1 Principle

Boyle's Law: When the temperature remains constant, the volume of a given mass of ideal gas varies inversely with its absolute pressure.

$$\frac{V_1}{V_2} = \frac{P_2}{P_1} \quad \text{or} \quad P_1V_1 = P_2V_2 \quad (13)$$

Extension of the equation to account for temperature variation and nonideal gas behavior is required for accurate grain volume determination.

$$\frac{P_1V_1}{z_1T_1} = \frac{P_1V_1}{z_2T_2} \quad (14)$$

This latter equation should be utilized with a double-cell device when determining sample grain volume (GV). Gas is admitted into a reference cell of known volume (V_r) at a predetermined reference pressure (100 to 200 psig). The reference cell gas is then vented into a connected chamber of known volume containing a core sample. This results in a lower equilibrium pressure, from which GV is calculated. The GV is subsequently subtracted from bulk volume to yield pore volume and thence porosity.

5.3.2.1.1.2 Advantages

The advantages of this method include:

a. The test sample is not damaged in any way and can be used for other measurements.
b. The operation is quick, simple, and has excellent repeatability.
c. Irregular-shaped samples and vuggy cores can be tested.

5.3.2.1.1.3 Limitations

The limitations of this method include:

a. For good accuracy, extremely careful and frequent calibration is required.
b. Changes in either temperature or barometric pressure must be accounted for in the calculations.
c. The resultant porosity value will be higher than the true porosity value if gas adsorbs on the core surfaces. The use of helium gas minimizes this possibility.
d. This method is suitable for poorly consolidated/unconsolidated jacketed cores if proper techniques are employed. The volume of the jacketing material and end screens must be accurately determined and subtracted from the measured apparent grain volume. The jacketing material and screen solid volume can be determined by 1) direct measurement in a Boyle's Law Double-Cell matrix cup prior to use, or 2) jacketing material volume may be computed from a measured weight, and a previously-determined jacketing sleeve material density.

5.3.2.1.1.4 Accuracy

A well calibrated system will supply grain volume within ±0.2 percent of the true value. This corresponds to approximately ±0.03 cm^3 in one-inch diameter samples approximately 1 inch long and ±0.1 cm^3 in $1^1/_2$-inch diameter samples approximately 2 inches long.

5.3.2.1.1.5 Apparatus

The grain volume is measured in an apparatus consisting of two connected chambers of known volumes. An example of such an apparatus is shown in Figure 5-5.

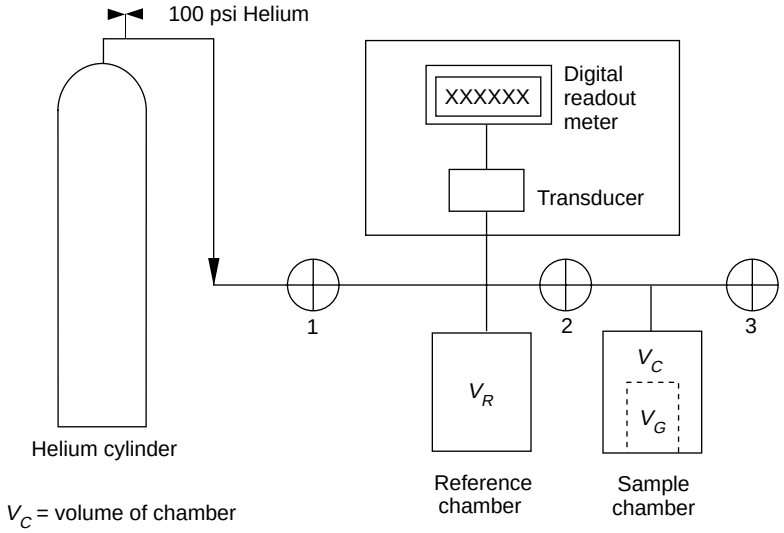


Figure 5-5—Double-Cell Boyle's Law Porosimeter

Note: For accurate measurements, it is important to (a) incorporate a valve having a zero displacement volume (e.g., a ball valve) between the reference volume and the sample chamber, or (b) the valve displacement volume must be known. The ball valve must always be vented to atmospheric pressure before it is turned to the closed position. Used in this manner, the valve bore volume is incorporated into the calibrated chamber volume and measurements are accurate. Otherwise, the valve displacement volume must be measured and accounted for in calculations for accurate measurement of grain volume.

5.3.2.1.1.6 Procedure

The porosimeter is first calibrated, yielding the reference chamber volume (V_r) and the sample chamber volume (V_c). A core plug is then placed in the sample chamber. Helium gas is admitted into the reference chamber at a predetermined pressure, typically 100 to 200 psig (690 to 1380 kPa). About 30 seconds should be allowed for pressure equilibrium and then p_1 (pressure indicated by the transducer digital readout) should be recorded. The gas is then allowed to expand into the sample chamber. The resulting lower pressure (p_2) is measured after the system has reached equilibrium (see 5.3.2.1.1.8, e). The grain volume of the sample is calculated using the gas law equations discussed in 5.3.2.1.1.7.

5.3.2.1.1.7 Calculations

The grain volume of the sample is calculated from the initial reference chamber pressure and the final system pressure by means of the gas law equation. The pore volume is the difference between the bulk volume and the grain volume.

The following grain volume equation is derived by a mass balance of gas within the reference and sample chamber:

$$\frac{P_1 V_r}{z_1 T_{1r}} + \frac{P_a (V_c - V_g)}{z_a T_{1c}} = \frac{P_2 V_r}{z_2 T_{2r}} + \frac{P_2 (V_c - V_g + V_v)}{z_2 T_{2c}} \qquad (15)$$

Where:

P_1 = absolute initial reference volume pressure.
P_2 = absolute expanded pressure.
P_a = absolute atmospheric pressure initially in sample chamber.
z_1 = z-factor of gas at P_1 and T_1.
z_2 = z-factor of gas at P_2 and T_2.
z_a = z-factor of gas at T_1 and atmospheric pressure.
T_{1r} = absolute temperature of reference volume at P_1.
T_{1c} = absolute temperature of sample chamber at P_1.
T_{2r} = absolute temperature of reference volume after P_2. is stabilized.
T_{2c} = absolute temperature of sample chamber after P_2 is stabilized.
V_g = grain volume.
V_c = sample chamber volume.
V_r = reference chamber volume.
V_v = valve displacement volume (from closed to open position).
p_1 = initial reference volume gauge pressure.
p_2 = final system gauge pressure.

If isothermal conditions exist ($T_1 = T_2$) and z values are assumed to equal 1.0, the equation reduces to:

$$V_g = V_c - V_r\left(\frac{P_1 - P_2}{P_2 - P_a}\right) + V_v\left(\frac{P_2}{P_2 - P_a}\right) \qquad (16)$$

If absolute pressures P_1 and P_2 are expressed as equivalent gauge pressures (i.e., $P_1 = (p_1 + P_a)$), and are substituted in the above equation, the following results:

$$V_g = V_c - V_r\left(\frac{p_1}{p_2} - 1\right) + V_v\left(1 + \frac{p_a}{p_2}\right) \quad (17)$$

If a zero displacement volume ball valve is used and the valve is always vented to the atmosphere before closing, V_v is included in the sample chamber volume, V_v = 0.0 and the equation further simplifies to:

$$V_g = V_c - V_r\left(\frac{p_1}{p_2} - 1\right) \quad (18)$$

Note: Calculations ignoring either *z* factors, or valve volume as small as 0.1 cm^3, can result in porosity errors of approximately 0.5 porosity points.

5.3.2.1.1.8 Precautions

Precautions for this method include:

a. The matrix cup must be designed to ensure that it will have the same internal volume each time it is reassembled.
b. The system must be recalibrated if there are changes in temperature or barometric pressure.
c. The core plug must be thoroughly and properly dried. Some samples require special drying techniques.
d. When pore water salinities (either from formation water or mud filtrate) are greater than 100,000 mg/l, samples should be leached with methanol to remove salts. Ignoring the leaching step when pore water salinities exceed 100,000 mg/l can bias porosities low by 0.4 porosity points (typical 20 percent porosity and a water saturation of 50 percent pore space).
e. Equilibrium pressure is essential for accurate grain volumes. Equilibrium is usually attained in 1 to 2 minutes, but low permeability and porosity cores require longer stabilization times (30 minutes to several hours). Longer times yield greater chance for temperature and barometric pressure changes.
f. Extraneous or dead volume (volume not occupied by the sample in the sample chamber) must be kept to a minimum or erroneous grain volumes will be determined. Solid stainless steel cylinders of known volume can be added to fill the sample chamber void volume left by shorter samples. When added, these steel volumes must be subtracted from the calculated grain volume to yield sample grain volume.

Note: If steel cylinders are present in the matrix cup during the calibration to yield V_c and V_r, their volumes should not be subtracted.

g. The pore volume of the sample to be measured should approximate the reference cell volume for best analytical results. Accuracy of the method on small, irregular test samples can be checked by determination of grain density on larger, and then smaller, similarly sized samples of a known grain density (i.e., Ottawa Sand).

5.3.2.1.1.9 Calibration

Porosimeter calibration varies with the instrument. In principle, two or more measurements are made; one is with the sample chamber filled with solid stainless steel cylinders of known volume. Subsequent measurements are made after removing one or more cylinders representing approximately 80, 60, 40, and 20 percent of the sample volume chamber. For best accuracy, sufficient cylinders should be removed to reduce p_1 by one-half.

Helium gas is admitted into the reference chamber at a predetermined pressure, typically 100 to 200 psig (690 to 1,380 kPa). About 30 seconds should be allowed for pressure equilibrium, and then p_1 (reference cell pressure indicated on the transducer digital read-out) is recorded. The gas is then expanded into the sample chamber.

The resulting lower pressure (p_2) is measured after the system has reached equilibrium (approximately 30 seconds). With valve volume (V_v) zero, temperature constant, and V_g known from volume of the steel cylinder in the chamber, V_r and V_c can be calculated from simultaneous solution of the appropriate equation presented in 5.3.2.1.1.7.

When V_v is not equal to zero or temperature varies, the solution becomes more complex, but the principle remains the same.

Note 1: In some systems equations are rearranged, and data are fit by linear regression over the cylinder volume range to yield V_c and V_r.

Note 2: Pressure transducers should be calibrated over 5 psi intervals with a dead weight tester or 0.02 percent full scale (or better) secondary standard pressure transducer. Transducer output can be fit to "true pressure" with a fourth degree polynomial using regression techniques. Check of the calibration can be made by measuring the volume of each steel cylinder and comparing it to the known volume. Agreement should be equal to or less than 0.03 cm^3 to assure porosity within 0.5 porosity points over the range of grain volumes measured.

5.3.2.2 Void Volume Measurement

5.3.2.2.1 Boyle's Law Single Cell Method for Direct Pore Volume

5.3.2.2.1.1 Low Confining Stress

5.3.2.2.1.1.1 Principle

Pore volume is determined in an apparatus consisting of a gas charged reference cell of known volume and initial pressure, which is then vented into a sample's pore volume. The sample is held in a core holder which utilizes an elastomer sleeve and end plugs. These conform closely to the sample when confining pressure is exerted on their external surfaces. The sleeve and end stems in turn exert compressive stress on the core sample. Pore volume is therefore determined directly using Boyle's Law. This is in contrast to the double cell method whereby grain volume is determined and pore volume is calculated by subtracting grain volume from bulk volume.

The core holder may be a Hassler, isostatic, biaxial, or triaxial load cell. Low confining stresses, which are generally 400 psig or less, are employed. In consolidated, hard rocks, the type core holder is of little significance as minimum pore space reduction occurs at low stress. Although these are typi-

cally referred to as "ambient measurements," it is important to note that some finite confining stress is necessary, and stress can have significant effects on unconsolidated or friable rocks.

Helium gas is admitted into the reference cell of known volume (V_r), at a predetermined reference pressure (100 to 200 psi). The reference cell gas is then vented into the sample pore volume. This results in a lower equilibrium pressure, from which the pore volume is calculated.

5.3.2.2.1.1.2 Advantages

Advantages of this method include:

a. The sample, if clean and dry at test initiation, is clean at test conclusion and ready for subsequent measurements.
b. Porosity and permeability can be run in sequence with a single loading of the sample.
c. Operation is quick and simple.
d. Harmful reactions between the rock and saturating fluid are eliminated by use of non-reactive gas.
e. Direct measurement of the pore volume eliminates the sensitivity of the pore volume to errors in the measurement of either *BV* or *GV*, where both *BV* and *GV* are large numbers relative to the pore volume, and $PV = BV - GV$.

5.3.2.2.1.1.3 Limitations

Limitations of this method include:

a. The system must be carefully calibrated for dead volume.
b. Changes in temperature and barometric pressure must be accounted for in the calculations.
c. The sample must be a good quality right cylinder with no surface vugs or chipped corners. (Vugs will yield erroneously low pore volume and non-parallel ends will yield high pore volume.) A porosity error exceeding 1.5 porosity units can result from a single, non-parallel sample face, which results in a reduction of sample length of 1.0 millimeter on one side of an otherwise one-inch diameter by one-inch long right cylindrical plug.
d. The sample must be clean of hydrocarbons and dry, or erroneously low pore volume will be determined.
e. The gas used must not adsorb to active mineral sites such as clays or carbonaceous materials. Adsorption results in erroneously high pore volume, and can be minimized by the use of helium gas.
f. Some low permeability rock (<0.01 md) may require a long time (thirty minutes to several hours) to reach pressure equilibrium. Longer times yield greater chances for temperature and barometric pressure changes.
g. When pore water salinities (either from formation water or mud filtrate) are greater than 100,000 mg/l, samples should be leached with methanol to remove salts. Ignoring the leaching step when pore water salinities exceed 100,000 mg/l can bias porosities low by 0.4 porosity points (typical 20 percent porosity and a water saturation of 50 percent pore space).
h. This method is suitable for jacketed, poorly consolidated/unconsolidated samples if proper precautions are observed. The void volume of the screens or porous plates attached to the plug faces must be determined. This volume must be subtracted from the jacketed sample's measured void volume to yield sample pore volume. A solid non-porous jacketed plug equal to sample diameter may be prepared and tested as a sample. This will yield the screen or porous plate void volume, as well as any volume resulting from overlap of the jacketing material on the sample ends.
i. Porosity is determined at low confining stress, which results in a porosity higher than present in the reservoir.

5.3.2.2.1.1.4 Apparatus

The basic apparatus shown in Figure 5-6 is the same as that illustrated for the double-cell Boyle's Law porosimeter shown in Figure 5-5 (see 5.3.2.1.1.5). The primary difference is the design of the sample chamber, which eliminates volume around the periphery of the sample.

5.3.2.2.1.1.5 Procedures

The porosimeter is first calibrated, yielding the reference chamber volume (V_r) and system dead volume (V_d). A clean, dry core plug is then inserted into an elastomer sleeve. An end stem with diameter equal to that of the plug is placed in contact with each end of the sample. A confining stress of 400 psi or less is applied to the external surface of the elastomer. If the sample is confined in an isostatic core holder, an equal

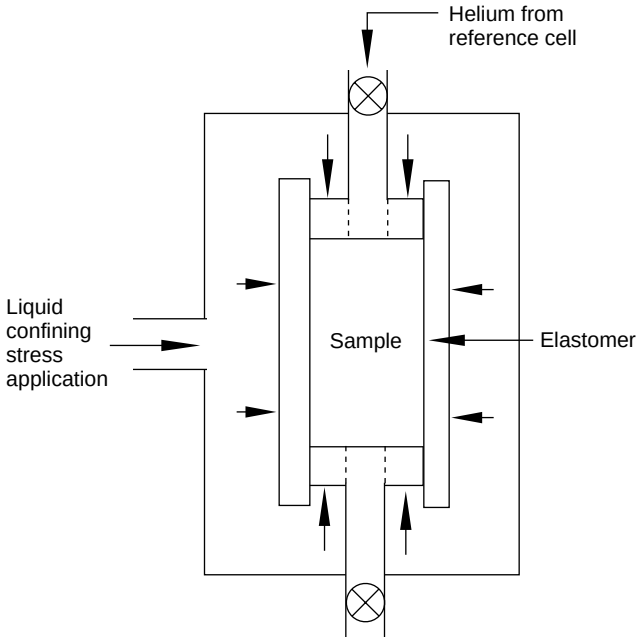


Figure 5-6—Schematic of Isostatic Load Cell for Direct Pore Volume Determination

confining stress is also applied to the outside surface of the end stems (see Figure 5-6).

Helium gas is admitted to the porosimeter reference chamber (V_r) at a predetermined pressure (p_1), typically equal to 100 to 200 psig (690 to 1380 kPa). Pressure is recorded, and the reference chamber is vented into the void volume of the sample. The resulting lower equilibrium pressure (p_2) is recorded. Pore volume of the sample is computed utilizing equations in 5.3.2.2.1.1.6.

5.3.2.2.1.1.6 Calculations

Sample volume is determined by expanding helium from a reference cell at known initial pressure directly into the porous rock. The following pore volume equation is derived by mass balance of gas within the reference cell, system dead volume, valve volume and sample pore volume.

$$\frac{P_1 V_r}{Z_1 T_{1r}} + \frac{P_a(V_p + V_d)}{Z_a T_1} = \frac{P_2(V_r + V_p + V_d + V_v)}{Z_2 T_2} \qquad (19)$$

Where:

P_1 = absolute initial reference volume pressure.
P_2 = absolute expanded pressure.
P_a = absolute atmospheric pressure initially in sample.
Z_1 = gas deviation factor at P_1 and T_1.
Z_2 = gas deviation factor at P_2 and T_2.
Z_a = gas deviation factor at P_a and T_1.
T_{1r} = absolute temperature of reference volume at P_1.
T_1 = absolute temperature of sample pore volume at P_a.
T_2 = absolute temperature of reference volume and sample after P_2 is stabilized.
V_r = reference chamber volume.
V_p = sample pore volume.
V_v = valve displacement volume (from closed to open position).
V_d = system dead volume.

If isothermal conditions exist, ($T_1 = T_2 = T_{1r} = T_1$) and:

$$\frac{P_1 V_r}{Z_1} + \frac{P_a(V_p + V_d)}{Z_a} = \frac{P_2(V_r + V_p + V_d + V_v)}{Z_2} \qquad (20)$$

grouping terms yields:

$$V_p = \frac{V_r\left(\frac{P_1 Z_2}{P_2 Z_1} - 1\right) - V_v}{1 - \frac{P_a Z_2}{P_2 Z_a}} - V_d \qquad (21)$$

Note: This equation is valid if no change in pore volume occurs as the sample pore pressure increases from P_a to P_2, i.e., (a) when the net effective confining stress remains constant, or (b) when it is not constant, but pore volume change with net confining stress change is negligible.

5.3.2.2.1.1.7 Precautions

Precautions for this method include:

a. Confining pressure on the external surface of the elastomer sleeve should be applied by liquid and not gas. Gas may diffuse into and through the sleeve with extended testing times.
b. Durometer of the elastomer sleeve must be low enough (suggest 50 durometer maximum) so that the sleeve seals on the metal end stems and properly conforms to the sample surface at the low confining stress utilized.
c. Samples with ends that deviate slightly from parallel can sometimes be successfully tested by utilization of two deformable, rubber discs. Each disc should be soft, equal to end butt diameter, and contain a drilled center hole. A single disc should be inserted between each metal end stem and the sample face. The disc will compress and conform to fill the void space between sample and end stem.

Note: This is recommended only when sample ends cannot be squared. The procedure is not a substitute for good quality samples.

5.3.2.2.1.1.8 Calibration

Equipment calibration utilizes (a) billets of known volume which are added or removed from a special chamber introduced only for calibration, or (b) a series of steel plugs precision bored to yield a variety of void volumes that cover the range of pore volumes to be measured. The reference volume is charged to initial pressure and vented into the special chamber or into the known void volume of the confined calibration plug. The measurement is repeated with multiple calibration billets or plugs. The pore volume equations can be solved simultaneously to yield the reference cell volume V_r and the dead volume. These values then become a constant for subsequent measurements of pore volume.

Dead volume (V_d) of the system can also be determined by utilizing a solid steel billet as a sample within the core holder or by placing the end stems adjacent in the elastomer sleeve and applying the same low confining stress to be used for sample measurement. The charged reference cell (V_r) is vented to the steel billet or to the adjacent end stems, after which the pore volume equation is used to calculate V_d. This value also remains constant as long as no changes are made to the system. Steel check plugs of known void volume can be measured as a final check of system operation and proper V_r and V_d values. These check plugs should also be utilized as daily confirmation of proper equipment performance.

5.3.2.2.1.1.9 Accuracy/Precision

A properly calibrated system will yield pore volumes within ±0.03 cm^3 in perfect cylinders. Measurements on actual core samples indicate scatter of approximately 0.1 cm^3 on samples of 50 cm^3 bulk volume, yielding porosity deviation of ±0.2 porosity units from the true value.

5.3.2.2.1.2 Elevated Confining Stress

5.3.2.2.1.2.1 Principle

The principle is similar to that discussed under low confining stress; however, in this measurement, porosity is determined at elevated stress. These laboratory data in turn are used to estimate reservoir porosity at in-situ reservoir stress conditions. Unlike low confining stress measurements, knowledge of the (a) elevated confining stress magnitude, and (b) method of application (isostatic stress, triaxial stress, biaxial stress, or uniaxial strain) must be known to scale results to reservoir conditions (see Figures 5-7 and 5-8). Historically, most routine pore volume measurements at elevated stress have been made with an isostatic (equal in all directions) confining stress, because this is an easily applied, relatively inexpensive method. Data are subsequently presented as porosity versus effective (confining pressure minus pore pressure) isostatic stress. In some reservoirs, a more complex and time consuming pore volume compressibility should be measured to more accurately determine porosity under reservoir conditions. These compressibility measurements typically require that liquid be present in the pore space to model liquid-rock interaction effects, if any,[1] and are beyond the scope of this document (see references in 5.6).

5.3.2.2.1.2.2 Advantages

Advantages of this method include:

a. Advantages are similar to those for low confining stress (see 5.3.2.2.1.1.2).

b. Measurements at elevated confining stress more closely represent original reservoir porosity than measurements at zero or low confining stress.

5.3.2.2.1.2.3 Limitations

Limitations of this method include:

a. Limitations are similar to items a through h for low confining stress (see 5.3.2.2.1.1.3).

b. Penetration of the elastomer sleeve into the depressions between sand grains or surface vugs on the sample surface as the confining stress increases may indicate pore volume reduction, even if no sample compression actually occurs. This penetration seldom exceeds 0.2 porosity units for non-vuggy sandstones (e.g., 10.0 percent to 9.8 percent), and while present, normally has no significant impact on porosity values. For vugular samples, this error can be significant. To reduce this error, a mylar or metal inner sleeve may be used. Surface effects can be minimized by increasing sample radius (r), as the ratio of surface area to bulk volume equals $2/r$ and decreases as sample radius increases.

c. The initial confining stress must be high enough to seal the elastomer sleeve to the sample and sample holder end butts, and to press the sample holder end butts tightly against the sample face. Sealing of the elastomer to the sample surface is dependent on the elastomer stiffness and sample roughness. Sealing stress must also be high enough to prevent bypassing of gas if permeability is to be measured in conjunction with porosity. Consequently, two opposing effects on porosity can occur. Poor conformance of the sample holder end butts with the sample, which yields porosity values that are too high, may be offset by sample pore volume compression. Use of good quality, right cylindrical samples with parallel ends minimizes conformance difficulties.

d. The resultant porosity at any elevated stress may be dependent on the rate of stress application, on the stabilization time and on the stress history.[2]

e. Reservoir effective stress must be known or estimated in order to properly utilize the laboratory-determined elevated-stress porosity data. Actual reservoir stresses causing pore volume reduction are usually not isostatic. The maximum principal stress in most reservoirs is vertical. This vertical effective stress equals the weight per unit area of overburden rock minus the reservoir pressure. Overburden stress can be computed from the depth and a downhole density log, or estimated assuming rock density equals 1.0 psi/ft.[3] Reservoir pressure can be measured, or in normally pressured areas, is assumed to be depth times the water gradient, which is approximately 0.45 to 0.5 psi/ft.

Note: Offshore reservoirs have a component of overburden stress resulting from water depth and density, as well as overburden rock.

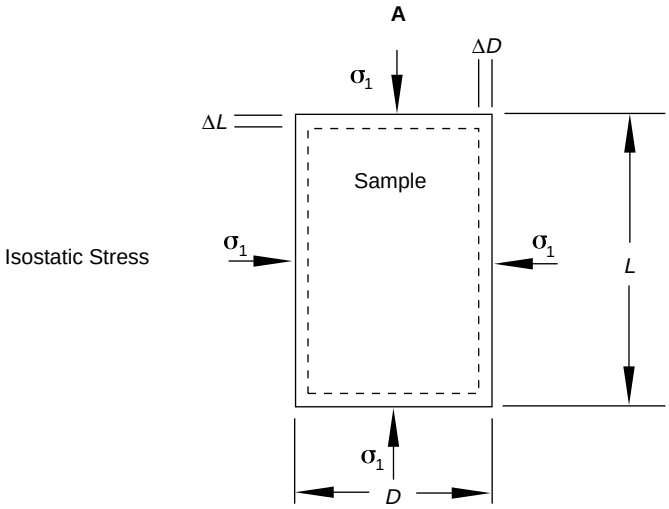
Typically, horizontal stresses are some fraction of the vertical stress, and can be estimated from hydraulic fracture treatments or formation integrity (leakoff) tests.[4,5] The mean reservoir effective stress is an average of vertical and horizontal effective stresses. Consequently, the mean reservoir effective stress is typically less than an isostatic stress equal to the vertical effective stress.

f. The presence of water within the pore space has been shown to weaken some rocks, thereby increasing pore volume reduction as confining stress is applied.[1] Since routine measurements, as discussed here, are on cleaned and dried rock samples, while the reservoir contains interstitial water, some uncertainty is introduced. While this effect is minimal in hard, well-cemented rocks, the importance of this effect can be quantified by conducting specialized tests on samples that contain water.

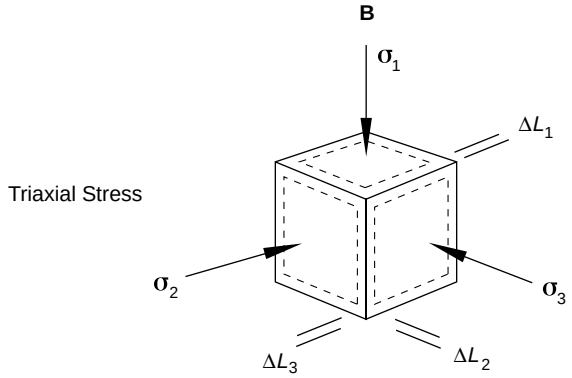
g. Friable or poorly-consolidated cores can exhibit significant compression at low confinement stresses.

5.3.2.2.1.2.4 Apparatus

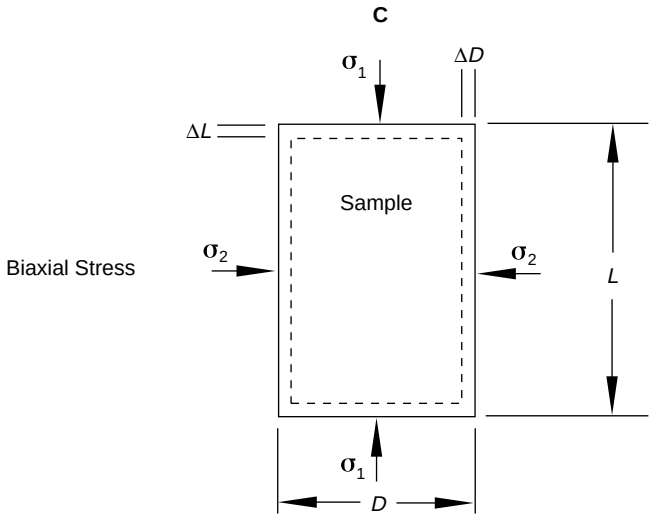
The apparatus is similar to that for low confining stress. However, high confining stress measurements require a sleeve material made of a tough elastomer that can withstand forces imposed during confining stress application, and yet will conform to the sample at low stress.



Under isostatic stress loading, equal stress is applied to the sample in all directions, and sample strain can occur on all axes. Excessive porosity reduction typically occurs when the imposed isostatic stress is equal to the vertical reservoir stress (i.e., the overburden stress).

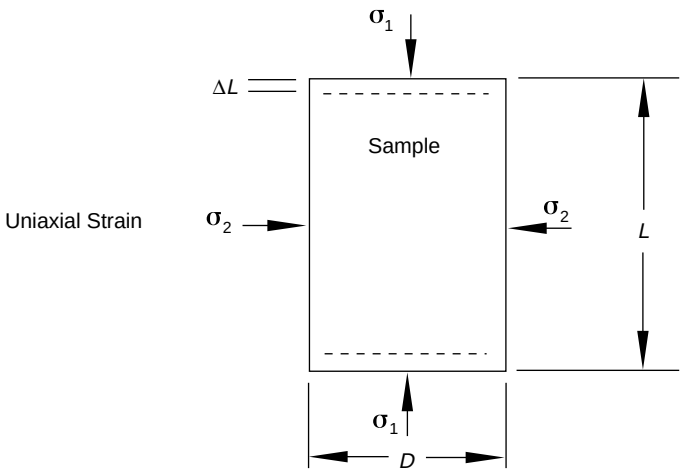


Under true triaxial stress conditions, unequal stress is applied to the three major axes of the sample. In the general case, strains will be different on each axis. Typically, a cube or rectangular prism-shaped sample will be used.



Biaxial stress loading conditions are a special case of triaxial stress loading. In the biaxial stress loading of a cylinder, the stress parallel to the cylinder axis is different than the stress applied around the sample's circumference. Strains can occur parallel to both the axis and diameter of the cylinder.

Figure 5-7—Laboratory Loading Schematic



Uniaxial strain compression is a special case of biaxial stress loading; the stress applied to the circumference is just sufficient to maintain the diameter constant as the stress parallel to the cylinder axis is increased. Strain occurs only parallel to the axis of the cylinder.

Figure 5-8—Laboratory and Reservoir Loading Schematic

5.3.2.2.1.2.5 Procedures

Procedures for elevated confining stress measurements are similar to those for low confining stress discussed in 5.3.2.2.1.1.5.

Typically, sample pore volume is determined at selected levels of increasing confining stress. Equilibrium pressure within the sample pore space at each confining stress is normally vented prior to subsequent pore volume measurement. Equations shown in 5.3.2.2.1.1.6 are used to compute pore volumes.

5.3.2.2.1.2.6 Calculations

The basic equations for pore volume (*PV*) calculation are the same as for low confining stress measurements, but calculation of porosity must also account for reduction in sample bulk volume (*BV*) as confining stress increases. Approaches that can be used include the following:

$$\text{Porosity} = PV/(\text{Non-stressed } BV - \Delta PV) \quad (22)$$

Where ΔPV = reduction in pore volume between initial *PV* and *PV* at any stress. This assumes the bulk volume reduction at any stress equals the pore volume reduction at the same stress.

$$\text{Porosity} = PV/(GV + PV) \quad (23)$$

Where *PV* = pore volume at any stress. This assumes grain volume (*GV*) as determined at zero stress is constant at all applied confining stress levels.

$$\text{Porosity} = PV/\text{calculated } BV \quad (24)$$

Measure pore volume (*PV*), sample diameter, and length at each stress imposed and calculate bulk volume. This is not routinely done.

5.3.2.2.1.2.7 Calibration

Calibration of equipment utilizes the same principles discussed in low confining stress (see 5.3.2.2.1.1.8). Validity of the system operation at elevated confining stress can be confirmed by measurement of steel check plug void volumes at the confining stresses selected for sample pore volume determination.

5.3.2.2.1.2.8 Accuracy/Precision

A properly calibrated system will yield pore volume accuracy of the same magnitude as observed at low confining stress (see 5.3.2.2.1.1.9), yielding porosity scatter of ±0.2 to 0.3 porosity units.

5.3.2.2.2 Summation of Fluids Porosity Summary

The "Summation of Fluids" technique of porosity determination is discussed in detail in the Fluid Saturation Methods under "Retort Method at Atmospheric Pressure" (see 4.2). It furnishes porosity data quickly as samples do not require cleaning or drying. The pore volume is computed by measuring and summing oil, gas, and water volumes present in a freshly recovered core sample. Porosity is determined by dividing pore volume by bulk volume of the rock sample.

When proper procedures are followed, and rocks are reasonably uniform and contain no hydratable minerals to complicate determination of water volumes, Boyle's Law porosities on adjacent core will typically agree within ±0.5 porosity units.[6] Without special precautions and techniques, the summation of fluids porosity can be erroneously high due to excess water recovery from hydratable minerals. This technique is not valid on weathered core from which liquids have evaporated. Measurements on weathered rock yield an erroneously low gas volume, and hence porosity, because the

injected mercury at the pressure utilized does not fill the smaller pores from which water has evaporated.

Grain density can be calculated from the measured volumes of rock and fluids. This can be compared to an estimated grain density based on lithology. The magnitude of the difference between calculated and estimated values yields a quality control check that will expose erroneous measurements or data calculation. This is not valid when extraneous minerals (heavy or light) are present in the rock. These conditions make the grain density variable and difficult to accurately estimate.

Retorting of the rock destroys the sample, so adjacent core must be used for permeability measurements. These permeability samples can be tested for porosity utilizing the Boyle's Law procedure, and compared with the summation of fluids values as a quality control check. Perfect sample-to-sample agreement is not expected, but the data should exhibit no bias.

5.3.2.2.3 Liquid Saturation Method

5.3.2.2.3.1 Principle

The measurement of porosity (connected pore space) by the liquid saturation method involves the gravimetric determination of pore volume by obtaining: (a) the weight of the core sample clean and dry, (b) the weight of the sample saturated with a liquid of known density, and (c) the weight of the saturated sample submerged in the same liquid.

5.3.2.2.3.2 Advantages

Advantages of this method include:

a. Many samples can be handled at one time.

b. Provides a direct measurement of pore volume.

c. Refer to 5.2.4.2, a and b.

5.3.2.2.3.3 Limitations

Limitations of this method include:

a. The procedure is slow in regard to total elapsed time of measurement.

b. The procedure is basically accurate, but limited to samples which can be 100-percent saturated and the saturated weight successfully determined.

c. Cores containing surface vugs are not recommended for porosity determination by this method because of the potential loss of liquid from the vugs during the weighing process. If liquid is lost, this volume would not be included in the pore volume, and erroneously low porosity will be calculated. However, grain volume can be accurately determined on vuggy cores by this method.

d. Refer to 5.2.4.3, a and c.

5.3.2.2.3.4 Accuracy

The method should determine porosity (connected pore space) within 0.5 porosity units and grain volume to ±0.2 percent of true value if proper technique is used.

5.3.2.2.3.5 Apparatus

Appropriate apparatus for this method include:

a. Analytical balance accurate to 1 milligram.
b. Suitable containers which can be used to hold deaerated liquid under vacuum.
c. Vacuum desiccator and pressure saturator.
d. A suitable vacuum source capable of maintaining less than 0.1 millimeter of mercury pressure.
e. A filtered, low-viscosity, low vapor-pressure liquid of known density for the purpose of saturating the core samples. Some liquids which have been used are: 1) brines, 2) refined laboratory oils, 3) decane, and 4) toluene.
f. Suitable containers which can be used to hold saturated cores under deaerated liquid.

5.3.2.2.3.6 Procedure

Obtain the weight of the dry, clean sample. The sample should be desiccated over a suitable dehydrating material, such as $CaCl_2$ or silica gel, prior to the determination of the dry weight.

Place the weighed, dry sample in a chamber (vacuum desiccator and pressure saturator) and apply a high vacuum for about 8 hours. The evacuation period may be followed by injection of CO_2 to remove the absorbed air on the rock. This replaces adsorbed gases such as O_2 or N_2 with CO_2, usually a more soluble gas in liquids typically used. Several cycles of evacuation and CO_2 injection may be required for low permeability samples. Longer CO_2 contact time and longer vacuum cycles are required to displace N_2 in the tighter rock. For very low-permeability samples, the evacuation period should be as long as 12 to 18 hours (overnight).

The liquid used to saturate the cleaned core sample should be free from dissolved air; therefore, the liquid should be deaerated before introduction into the core. Extreme care should be taken to avoid air contact with the liquid after its deaeration. Hydrocarbons are stronger wetting fluids than brines and are preferred if only porosity measurements are desired and samples saturated with brine are not required for further testing.

The deaerated liquid is drawn into the evacuated vessel containing the core sample. The core is allowed to saturate. After the sample has been completely submerged in the liquid, the vacuum is continued for an additional 30 minutes to 1 hour.

The preferred method is to then pressure the liquid surrounding the sample to 2,000 to 3,000 psi (13,800 to 20,700 kPa) for at least four hours to assure complete saturation. Pressure saturation should result in the liquid entering addi-

tional voids which are not well connected or where spontaneous imbibition has not occurred. The sample is then removed from the saturating vessel and weighed submerged in the saturating liquid.

Excess liquid is carefully removed (avoiding grain loss) from the sample and the saturated sample is weighed in air. When removing excess liquid from the sample surface, precautions must be taken to ensure fluids are not removed from pores exposed at the surface. Materials (such as dry towels) which remove liquid from surface pores due to capillary action should be avoided, as should any mechanical method such as violent shaking. Rolling the sample over a lint free cloth or paper towel dampened with the saturating liquid, or carefully wiping the sample with the dampened cloth or the fingers are suitable techniques for removing excess liquid.

After weighing each batch of samples, the density of the deaerated saturant used in the saturation chamber is determined. This must be done with accuracy, since the saturant density is probably the greatest source of error unless Equation 5.3.2.2.3.8e is used. Refer to procedures described in 5.2.4.6 for the determination of bulk volume. When coupled with the original dry weight and fluid density, grain volume is computed directly (see Equation 5.3.2.2.3.8c).

For storage until use, the core plugs are usually transferred to screw-capped jars or other suitable containers that are filled with deaerated liquid in order to minimize the presence of air in the container.

5.3.2.2.3.7 Precautions

Precautions for this method include:

a. Special precautions are necessary to ensure complete saturation. Low permeability cores may not be fully saturated by this method.
b. For acceptable accuracy, it is essential to use the correct fluid density at the time the saturated weight of the sample is determined.
c. Error in the pore volume can result from improper wiping of the sample prior to the determination of the saturated weight.

5.3.2.2.3.8 Calculations

The calculations for this method are as follows:

a. $$PV = \frac{\text{Saturated Weight} - \text{Dry Weight}}{\text{Density of Saturant}}$$

b. $$BV = \frac{\text{Saturated Weight} - \text{Immersed Weight}}{\text{Density of Immersion Liquid}}$$ (see 5.2.4)

c. $$GV = \frac{\text{Dry Weight} - \text{Immersed Weight}}{\text{Density of Immersion Liquid}}$$

d. Pore Volume check: $PV = BV - GV$ (PV by difference should agree with PV calculated in a.)

e. $$\text{Porosity} = \frac{PV}{BV} = \frac{\dfrac{\text{Saturated Weight} - \text{Dry Weight}}{\text{Density of Saturant}}}{\dfrac{\text{Saturated Weight} - \text{Immersed Weight}}{\text{Density of Immersion Liquid}}}$$

If temperature is constant, and density of saturant and immersion liquid are equal, fluid density cancels and is not needed in the above equation to calculate porosity.

$$\text{Porosity} = \frac{\text{Saturated Weight} - \text{Dry Weight}}{\text{Saturated Weight} - \text{Immersed Weight}}$$

f. Porosity check: $$\text{Porosity} = \frac{BV - GV}{BV}$$

(Porosity should agree with that calculated in e.)

g. $$\text{Grain Density} = \frac{\text{Dry Weight}}{GV}$$

Where:
PV = pore volume.
GV = grain volume.
BV = bulk volume.

5.4 HISTORICAL PROCEDURES

This section lists and summarizes techniques that have been used historically to measure porosity, bulk volume, grain volume, and grain density. These have generally been replaced by newer techniques. Details about these procedures can be found in the First Edition of API RP 40, *API Recommended Practice for Core-Analysis Procedure,* August 1960.

5.4.1 Mercury Displacement

This technique consists of immersing a dried core sample in mercury inside a calibrated pycnometer. The volume of mercury displaced by the sample is weighed to determine the bulk volume of the core sample. (API RP 40, 1960, Sec. 3.311, p. 15; Sec. 3.56, p. 26.)

5.4.2 Bulk-Volume Meter

This procedure also involves submerging a core sample under mercury. The volume of mercury displaced by the sample further displaces a second liquid into an inclined, graduated glass tube. The bulk volume of the core is read directly off the graduated glass tube. (API RP 40, 1960, Sec. 3.312, p. 15; Sec. 3.57, p. 26.)

5.4.3 Boyle's Law Single-Cell Method Using the Kobe Porosimeter

The Kobe Porosimeter consists of a mercury pump, pressure gauge, gas inlet and outlet valves, and a sample chamber. Determination of the bulk volume of the sample requires filling the sample chamber containing the core sample with mercury. This leaves the core sample immersed in mercury. Bulk

volume is determined by subtracting the volume of mercury needed to fill the sample chamber without a sample from the volume of mercury needed to fill the chamber with a sample.

Grain volume is calculated by backing the mercury pump out to a "preset" volume. During this portion of the test the sample is not immersed in mercury. The pressure in the cell is vented to atmospheric pressure (P_1). Mercury is pumped into the sample cell (gas outlet valve shut) and the new pressure (P_2) is recorded. Knowing the volumes of mercury in the cell, the empty preset volume of the cell, and the pressures, Boyle's Law can be used to calculate grain volume ($P_1 \times V_1 = P_2 \times V_2$). Porosity is calculated from the bulk volume and the grain volume. Grain density is calculated from sample weight and grain volume. (API RP 40, 1960, Sec. 3.32211, p. 17; Sec. 3.5.10, p. 28.)

5.4.4 Washburn-Bunting Method

The Washburn-Bunting porosimeter measures the pore volume of the sample. The apparatus consists of a sample chamber with a graduated capillary tube on top with a stopcock for opening and closing the system to the atmosphere. A tube is attached to the bottom of the sample chamber which connects to a glass bulb filled with mercury. A sample is placed in the sample chamber. The procedure is begun by raising the mercury bulb to force mercury into the sample chamber, thereby immersing the sample, and up into the calibrated capillary. When the mercury is above the stopcock, the stopcock is closed. The mercury bulb is now lowered until the core sample is floating on the mercury inside the sample chamber. The sample is now under vacuum, and air from the pores of the sample fill the sample chamber and calibrated capillary. After a few minutes, atmospheric pressure is restored to the escaped air by raising the mercury bulb until the mercury level is equal to that in the capillary. The air volume in the calibrated capillary is equal to the pore volume of the rock sample. Improved accuracy is obtained by subtracting the air volume measured by using a solid billet in place of the core sample to account for adsorbed air on the sample surface. (API RP 40, 1960, Sec. 3.3221, p. 17; Sec. 3.5.12, p. 30.)

5.4.5 Dry Method for Grain Density and Pore Volume

In this procedure, the rock sample is extracted, dried, crushed, and sieved. A portion of the crushed rock that passes a 60-mesh screen and is caught by a 100-mesh screen is weighed and added into the sample cup of the apparatus. A mercury pump is then used to pump mercury into the sample cup. When the system pressure reaches the preset calibration value, the volume of injected mercury is recorded. By calculating the volume of the empty cup, the bulk volume of the sample in the cup can be determined. Using the sample weight, grain density can be computed. Using a companion rock sample, grain volume is computed by dividing the companion sample dry weight by the calculated grain density from the crushed sample. Pore volume can be determined by subtracting the grain volume from measured bulk volume of the sample. (API RP 40, 1960, Sec. 3.3211, p. 16; Sec. 3.58, p. 26.)

5.4.6 Wet Method for Grain Density and Pore Volume

The bulk volume is measured and the dry sample is weighed and crushed. The crushed sample is added to a calibrated volumetric flask containing a known volume of a suitable wetting fluid (e.g., toluene, water, etc.). The volume increase is equal to the grain volume of the crushed sample. The grain density can be calculated by dividing the weight of the crushed sample by its measured volume. The pore volume can be calculated by subtracting grain volume from bulk volume. (API RP 40, 1960, Sec. 3.3212, p. 16; Sec. 3.59, p. 27.)

5.4.7 Full Diameter Summation of Fluids Utilizing the Vacuum-Retort Method

This method was used extensively in "hard rock" regions (e.g. West Texas) where production is predominately from carbonate formations. Each full diameter test sample was weighed and then the gas filled pore space was pressure saturated with water. The sample was again weighed, with the weight increase equal to the gas volume. Fluids within the pore space were distilled from the core under partial vacuum, at a maximum temperature of 450°F (232°C). Fluids were collected in glassware immersed in an alcohol/dry ice bath maintained at –75°F (–59°C). This was to condense vapors and prevent loss through the vacuum system. Condensed volumes were read, and an oil correction factor was employed. The sample pore water was computed by subtracting the gas volume from total water collected in the condensing tube. Pore volume was computed by summing the gas, corrected oil, and pore water volumes. A bulk volume determined on the test sample was used for calculation of porosity (API RP 40, 1960, Sec. 4.21, p. 39; and 4.52, p. 42).

One of the prime disadvantages of this method was the condition of the cores after the distillation process. It was not uncommon for the cores to be black from coked oil and no cleaning process could restore the samples to a condition whereby they could be deemed suitable for use in further testing procedures (e.g., a Boyle's Law Porosity, Capillary Pressure Tests, Relative Permeability). For this and other reasons, the Subcommittee charged with the rewriting of Recommended Practice 40 elected not to record this as a recommended procedure.

5.5 ORGANIC-RICH ROCKS

In recent years, there has been considerable interest in the development of coalbed methane and gas shale reservoirs. Coals and gas shales are typically highly organic fractured

formations in which much of the gas-in-place is adsorbed to the organic material. Because adsorbed gas comprises a significant portion of the gas-in-place in these reservoirs, both the adsorbed gas content and how the gas is released (desorption/adsorption isotherms) are necessary for reserve estimates and production forecasts. Oil shales contain solid organic material, typically liquefied by application of heat to generate oil. Techniques to evaluate these type of deposits are discussed in 4.7. Porosity, as discussed herein, is not normally required.

5.5.1 Coal

There are no generally accepted methods in the industry for the laboratory measurements of porosity, permeability, or relative permeability of coal samples, nor are there published laboratory measurements that are accepted standards. Standard laboratory techniques for measurement of porosity in oil and gas bearing reservoir rock must, therefore, be modified when applied to coal used in coalbed methane studies. Following are comments and references reflecting current practices.

In production of methane from coal seams, "porosity" may be defined in terms of three distinct parameters. For calculation of coalbed methane gas-in-place, these are (a) the "in-situ gas content" of the coal, which is equivalent to the gas volume contained in the gas-filled porosity in a conventional gas reservoir. In addition, coal is a dual porosity system consisting of (b) matrix (micropore) porosity wherein the methane is adsorbed on the coal surface, and (c) cleat porosity (naturally occurring microfracture porosity). The cleat network provides the path and permeability for fluid flow.

5.5.1.1 In Situ Gas Content

The only method by which total in situ gas content of coalbeds can be measured directly is with the use of pressure core technology.[7] The volume of gas evolved from the coal core in a pressure core barrel is measured as a function of time, temperature, and pressure.

Gas content and desorption rate are often determined by canister desorption methods.[8] Coal samples utilized include cores obtained using conventional coring, drilled sidewall coring, wireline retrieval, and coal drill cuttings. The coal cores (or cuttings) are sealed in the canister and the volume of gas evolved from the sample is measured as above.[7] Gas content determination by canister desorption requires an estimation of the volume of gas lost while retrieving the samples to the surface and before sealing the cores in the canister.[9,10]

Gas content from conventional and sidewall core samples by the canister desorption method is much less expensive than pressure coring, and is the most commonly used technique. Even pressure cores are often placed in desorption canisters to complete the desorption process. Gas content from different methods or samples can be compared after normalizing the coal to an ash-free basis.[7] Since the gas evolved is not always 100 percent methane,[11] the composition of the gas must be analyzed to determine methane content.

5.5.1.2 Matrix Porosity

The matrix porosity of coal is generally larger than the cleat porosity. However, since the methane is adsorbed in the matrix rather than existing as a free gas, conventional oil or gas field matrix porosity measurements are seldom performed for coalbed methane applications. If conventional pore volume is required, equilibrium moisture[12] and helium Boyle's Law porosity[13] techniques can be used to measure matrix porosity.

Note: Any gas that adsorbs will yield erroneous matrix porosity values with the Boyle's Law technique. Helium, which is not appreciably adsorbed, is often used.

5.5.1.3 Cleat Porosity

Although the bulk of coal porosity is the matrix porosity, the cleat network provides the path and permeability for fluid flow. The measurement of cleat porosity in coal is, therefore, important in determining pore volume compressibility, as well as the saturations necessary for gas-water relative permeability or capillary pressure measurements.

Cleat porosity is a function of net confining pressure. Cleat porosity should be measured with the coal core in a uniform confining pressure. Jacketing materials commonly used for unconsolidated cores[16] are often used on coal cores.

Measurements of coal porosity have been made on coal cores dried in vacuum ovens.[14] Studies on coal weathering indicate such drying alters the coal structure,[15] yielding erroneous results. A suitable procedure for cleat porosity measurement on non-dried core is referred to as the miscible drive technique. In this procedure, the fluid saturating the core is displaced from the core by a second fluid with different physical properties, but which is miscible with the saturating fluid.[16]

The miscible drive technique may employ tracer concentration in water. A tracer that is adsorbed will yield erroneous results.[17] Within experimental error, the immiscible displacement with water-vapor-saturated helium of water from a water-saturated coal core yields the same results as the miscible drive tracer technique.[17]

Note: Coal cores often have residual methane in the matrix, which can affect porosity measurements. Saturation of coal core with helium (saturated with water vapor), followed by evacuation, has been used to remove the residual methane.[17]

5.5.2 Gas Producing Shale

In gas shales, such as the Devonian, gas may be stored as free gas in the matrix or fractures, and also exist as adsorbed gas on the organic and clay surfaces. Adsorbed gas may account for as much as 80 percent of the gas-in-place, emphasizing the need for adsorbed gas isotherms. Volumes of adsorbed gas in shale are $^1/_5$ to $^1/_{50}$ of that for coal, and measurement apparatus and procedures must be tailored to supply

accurate data. As is currently the case for coal, no industry standard procedures currently exist for conducting shale measurements.

Reported results of adsorbed gas isotherm measurements on shale samples indicate that a wide disparity in test conditions currently exists between testing laboratories.[18] These disparities result in varying adsorption results, and emphasize the need for persons using the data to clearly understand what is being measured and what is reported.

Simulators utilized in reservoir modeling often utilize as input adsorbed methane content versus pressure data. Data are typically reported in units of scf/ton. Consequently, it is important to know if the isotherm measured and reported is for adsorbed gas alone, or if it represents a total sorption isotherm, which includes adsorbed gas as well as gas stored as free gas in the matrix and/or fractures.

The presence of water reduces gas volumes adsorbed. Adsorption tests on dry rock, therefore, will over estimate gas reserves, and tests on rock samples containing an equilibrium moisture condition are preferred.

Note: Techniques to establish appropriate equilibrium moisture conditions representative of the reservoir are not yet defined. Consequently, many of the adsorption isotherms measured have been completed on shale "as received," or on dry rock.

Although the technology for measurement is still evolving, certain important elements affecting measurement results and reported data have been identified.[18] Engineers utilizing these data should discuss with the measurement laboratory the following key elements. Procedures should be established that are most likely to yield data representative of the reservoir and that are required in the mathematical simulator utilized.

5.5.2.1 Key Elements Affecting Measurement

Factors affecting the measurement include:

a. Core recovery techniques (including coring fluid).
b. Sample preservation technique.
c. Sample size and shape (crushed? sieved to what size?).
d. Sample drying conditions:
   1. "As received."
   2. Dried (at what temperature and how long?).
   3. At equilibrium conditions (what temperature and how long?).
e. Standard temperature and pressure used for reporting measured gas volumes as scf/ton.
f. Metric ton or U.S. ton of "as received," dried or equilibrium weight rock used as base for scf/ton.
g. Total sorbed (including free porosity) gas or adsorbed gas only isotherm.
h. Cell calibration technique.
i. Temperature measurement and control of cell.
j. Sample density reported (bulk or grain; wet or dry).
k. Was equilibrium reached at each adsorption level, or was test step terminated at selected adsorption time?
l. Sample volume tested.
m. Sample evacuated prior to testing.
n. Gas used in adsorption measurement (usually methane).
o. Gas used in "free gas volume" porosity measurement (usually helium).

5.5.2.2 Apparatus

An apparatus designed to determine volumetric adsorption is illustrated in Figure 5-9.[19] It resembles a commonly used double-cell porosimeter.

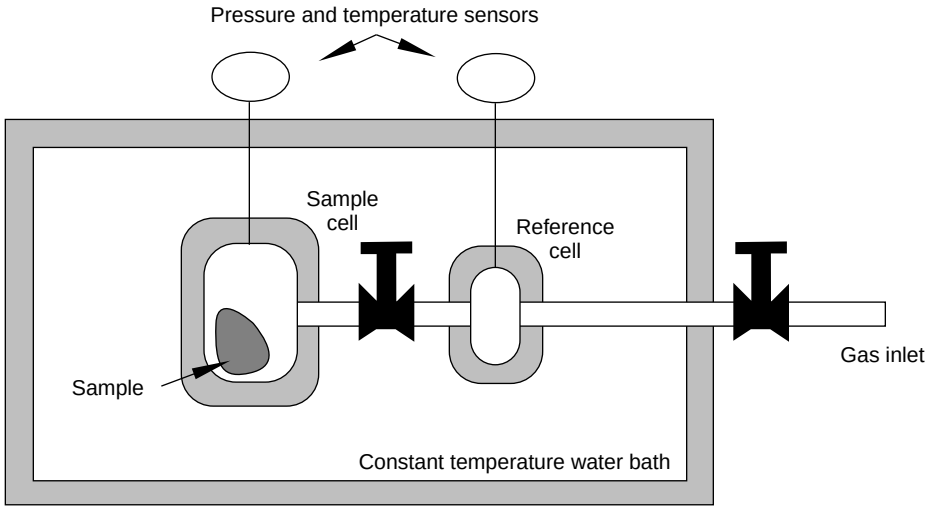


Figure 5-9—Volumetric Adsorption Measurement Method

5.5.2.2.1 Free (Fracture and Matrix) Porosity

Fracture and matrix void volume can be determined utilizing Boyle's Law double-cell porosimeter (see Figure 5-5) with helium as the gas phase.

5.5.2.2.2 Adsorption Isotherms

An isotherm measurement with helium gas is first completed. This yields free gas space with minimum adsorption. Methane gas is than charged into the reference cell and the gas is expanded into the sample chamber. The reference chamber is again isolated from the sample cell and is charged with gas at higher pressure. The procedure is repeated, and the gas adsorbed at the higher pressure level is calculated. This process is repeated until desired pressure levels are reached. The difference between the helium and methane isotherms represent adsorbed gas. Equations for calculation of isotherms, as well as an error analysis identifying factors that affect the accuracy of the measurement have been presented.[19]

5.5.3 Oil Shale

Evaluation of oil shales do not typically require measurement of rock porosity, as the oil recovered is generated from solid organic materials subjected to heat. See 4.7 for oil content determination.

5.6 REFERENCES

1. Thomas, D.C. and Pugh, V.G., "A Statistical Analysis of the Accuracy and Reproducibility of Standard Core Analysis," *The Log Analyst*, March-April 1989, p. 71–77.
2. Luffel, D.L. and Howard, W.E., "Reliability of Laboratory Measurement of Porosity in Tight Gas Sands," *SPE Formation Evaluation*, December 1988, p. 705–710.
3. Keelan, D.K., "Automated Core Measurement System for Enhanced Core Data at Overburden Conditions, SPE 15185," presented at Rocky Mountain Regional Meeting, Billings, MT, May 19–21, 1986.
4. Rhett, D.W., "Long Term Effects of Water Injection on Strain in North Sea Chalks," Third North Sea Chalk Symposium, June 1990, Copenhagen, Session 1.
5. Rhett, D.W. and Teufel, L.W., "Stress Path Dependence of Matrix Permeability of North Sea Sandstone Reservoir Rock," 33rd U.S. Symposium on Rock Mechanics, Santa Fe, New Mexico, June 8–10, 1992.
6. Fertl, W.H., *Abnormal Formation Pressures*, pp. 2–3.
7. Breckels, I.M. and VanEekelen, H.A.M., "Relationships Between Horizontal Stress and Depth in Sedimentary Basins," *JPT*, Sept. 1982, p. 2191.
8. Woodland, D.C. and Bell, J.S., "In-Situ Stress Magnitudes from Mini-frac Records in Western Canada," *JPTC*, Sept.–Oct 1989., p. 22.
9. W. Hensel, "Summation-of-Fluids Porosity Technique," *SPEJ*, April 1982, p. 193.
10. Law, B.E. and Rice, D.D., editors, *Hydrocarbons from Coal*, AAPG, in preparation.
11. Mavor, M.J., Close, J.C. and McBane, R.A., *Formation Evaluation of Exploration Coalbed Methane Wells*, presented at the Petroleum Society of CIM/SPE International Technical Meeting, Calgary, June 10–13, 1990, CIM/SPE 90–101, 101–1.
12. Diamond, W.P. and Levine, J.R., "Direct Method Determination of the Gas Content of Coal: Procedures and Results," *U.S.B.M. Report of Investigations 8515* (1981).
13. Smith, D.M. and Williams, F.I., "A New Method for Determining the Methane Content of Coal," *Proceedings of the 16th Intersociety Energy Conversion Engineering Conference* (1981) 1267–1272.
14. Hanson, W.B., "Chemistry of the Western Interior USA Coalbed Gases Based upon Desorption of Subsurface Coal Samples," presented at Rocky Mountain Section of AAPG, Denver, Colorado, September 16–19, 1990.
15. Berkowitz, N., *An Introduction to Coal Technology*, Academic Press, New York, 1979.
16. Meyers, R.A., editor: *Coal Structure*, Academic Press, New York, 1982.
17. Dabbous, M.K., Reznik, A.A., Tabor, J.J. and Fulton, P.F., "The Permeability of Coal to Gas and Water," *SPEJ* (December 1974), 556–565.
18. Nelson, C.R., editor, *Chemistry of Coal Weathering*, Elsevier, New York, 1989.
19. Lerner, D.B., Dacy, J.M., Raible, C.J., Rathmell, J.J., Swanson, G. and Wall, J.D., "SCA Guidelines for Sample Preparation and Porosity—Measurement of Electrical Resistivity Samples, Part II Sample Preparation and Porosity Measurement," *The Log Analyst*, (March–April 1990), 57–63.
20. Gash, B.W., "Measurement of 'Rock Properties' in Coal for Coalbed Methane Production," presented at the 66th SPE Annual Technical Conference and Exhibition, Dallas, Texas, October 6–9, 1991, SPE 22904.
21. Lancaster, D.E. and Hill, D.G., "A Multi-Laboratory Comparison of Isotherm Measurements on Antrim Shale Samples, Otsego County, MI," *Proceedings of the 1993 Society of Core Analysts* (April 9–11, 1993) Paper 9303.
22. Lu, X., Li, F.C. and Watson, A.T., "Adsorption Measurements in Devonian Shales," *Proceedings of the 1993 Society of Core Analysts* (April 9–11, 1993) Paper 9302.

SECTION 6—PERMEABILITY DETERMINATION

CONTENTS

Page

6 PERMEABILITY DETERMINATION 6-1
- 6.1 Introduction 6-1
- 6.2 Theory 6-5
- 6.3 Practical Applications for Steady-State Permeability Measurements. 6-18
- 6.4 Theory and Application of Unsteady-State Permeability Determinations 6-32
- 6.5 Accuracy and Precision 6-39
- 6.6 Calibration of Instruments 6-42
- 6.7 References 6-45
- 6.8 Appendices 6-47

Figures
- 6-1 Illustration of Flow Potential vs. Pressure Differences 6-3
- 6-2 Rock-Dependent Gas Slippage Radius, r_b, as a Function of Permeability and Porosity 6-13
- 6-3 Inertial Resistivity, β, as a Function of Permeability 6-15
- 6-4 Maximum Allowable Overall Pressure Gradients for Use of Slip-Corrected Darcy Equation 6-16
- 6-5 Type of Plot Used To Obtain Klinkenberg Gas Slippage Factor, b. 6-17
- 6-6 Downward Flow of Liquid with Driving Head, h 6-19
- 6-7 Upward Flow of Liquid with Driving Head, h 6-19
- 6-8 Upward Flow of Liquid with Imposed Pressure Difference, Δp. 6-19
- 6-9 Permeability Reduction with Increasing Net Hydrostatic Stress 6-20
- 6-10 Schematic of Permeability Apparatus for Axial Flow of Gas 6-21
- 6-11 Simplified Flow Diagram for Low Pressure, Axial Gas Flow Permeability Measurements. 6-22
- 6-12 Low-Pressure Hassler-Type Core Holder 6-23
- 6-13 High-Pressure Core Holder for Hydrostatic Stresses 6-23
- 6-14 High-Pressure Core Holder for Hydrostatic or Biaxial Loading 6-24
- 6-15 Schematic of Steady-State Probe Permeameter 6-26
- 6-16 Dimensionless Geometric Factor for Measurements with Probe Permeameter. 6-26
- 6-17 Transverse Flow Configuration for Gases or Liquids 6-28
- 6-18 Dimensionless Geometric Factor for Transverse Flow 6-28
- 6-19 Hassler-Type Core Holder for Transverse Permeability Measurements. 6-28
- 6-20 Schematic for Inward Radial Flow 6-30
- 6-21 Full-Diameter Radial Flow Permeameter 6-30
- 6-22 Schematic of Pressure-Falloff Gas Permeameter 6-34
- 6-23 Schematic of Pressure-Falloff Probe Permeameter. 6-36
- 6-24 Pulse Decay Apparatus for Axial Flow of Gases. 6-37
- 6-25 Schematic of Liquid Pulse-Decay Permeameter. 6-38
- 6-26 Schematic for Calibration of Volumes in Unsteady-State Permeameters. 6-45
- A.6-1 Plot for Determining Maximum Allowable Δp in Axial Flow Liquid Permeability Measurements. 6-50
- B.6-1 Construction of Flow Variables y_n and p_{gn} 6-51
- B.6-2 Dimensionless Correction Factors for Non-Constant Mass Flux in Pressure-Falloff Permeability Measurements 6-52

Page

B.6-3 Variation of k_∞, β, and Standard Error with b for Pressure-Falloff Data. 6-53
D.6-1 First Root Relationship for Pulse-Decay Equation 6-58
D.6-2 Pulse-Decay Plot for Finding f_1, from f_0 with Known Ratios of V_2 / V_1 (for Gases) or S_2 / S_1 (for Liquids) 6-58

Tables

6-1 Constants in Forchheimer or Darcy Equation with Various Sets of Units 6-4
6-2 Quick Selection and Reference Guide for Permeability Measurements Using Gases 6-6
6-3 Quick Selection and Reference Guide for Permeability Measurements Using Liquids 6-7
6-4 Viscosity of Air (for Degrees F and C) at One Atmosphere 6-8
6-5 Viscosity of Nitrogen (for Degrees F and C) at One Atmosphere 6-8
6-6 Viscosity of Helium (for Degrees F and C) at One Atmosphere 6-9
6-7 Gas Law Deviation Factors for Air, Calculated from Beattie-Bridgemann Equation of State 6-10
6-8 Gas Law Deviation Factors for Nitrogen, Calculated from Beattie-Bridgemann Equation of State 6-11
6-9 Gas Law Deviation Factors for Helium, Calculated from Beattie-Bridgemann Equation of State 6-12
D.6.1 Pulse Decay Constants When $a = b$ ($V_1 = V_2$, or $S_1 = S_2$) 6-57

6 Permeability Determination

6.1 INTRODUCTION

Permeability is a property of a porous medium and is a measure of its ability to transmit fluids. The measurement of permeability of a porous rock, or stratum, is a measurement of the fluid conductivity of the particular material. Permeability is the fluid-flow analog of electrical or thermal conductivity. The reciprocal of permeability represents the viscous resistivity that the porous medium offers to fluid flow when low flow rates prevail. This condition of flow is customarily called "viscous flow," or formally, "Stokes flow". Measurement of the flow of fluid through a sample in a particular direction yields permeability of the sample in that direction. The permeability of a homogeneous, isotropic medium is the same everywhere, and in all directions. However, real rocks are neither perfectly homogeneous nor isotropic.

6.1.1 Definitions

6.1.1.1 Darcy Flow

The mid-nineteenth century experiments and studies of Darcy[1] led to an empirical expression of the relationships among the variables involved in the flow of fluids through porous media, known now as Darcy's Law. Briefly, Darcy's Law states that the volumetric rate of flow per unit cross-sectional area of permeable medium (the volumetric flux) is directly proportional to the potential gradient, and inversely proportional to the viscosity of the fluid. The coefficient of proportionality is permeability. Thus, the potential gradient required for Stokes flow is equal to the product of the fluid's viscosity and volumetric flux, divided by the permeability of the rock. The potential gradient for horizontal liquid flow, or in any direction for practical measurements with low density gas, is equal to the pressure gradient.

6.1.1.2 Forchheimer Inertial Resistance

The work of Forchheimer[2] at the beginning of the twentieth century showed that Darcy's Law is a limiting case, restricted to low volumetric fluxes. At higher fluxes, Forchheimer observed that the potential gradient required for a given volumetric flux is greater than that predicted by Darcy's Law by an amount proportional to the product of the fluid's density and the square of its volumetric flux. The coefficient of proportionality, β, is the inertial resistivity of the porous medium. Inertial energy dissipation is due to innumerable accelerations (i.e., time-rates of change in the direction and magnitude of velocity) that a fluid undergoes as it travels tortuous paths through a porous medium. These accelerations cause secondary flow patterns, in which, part of the flow energy is converted to heat through viscous shear.

In a petroleum reservoir, especially away from a producing or injection well, volumetric fluxes are generally so low that Darcy's Law is applicable. However, high near-wellbore fluxes associated with high pressure gradients can cause non-negligible inertial effects known as "rate-sensitive skins." These are particularly prevalent near gas-producing wellbores, and in perforations, where low gas viscosity permits very high fluxes.

In laboratory measurements of high permeability samples using gas (where inertial effects are most often observed), low volumetric fluxes require low pressure gradients, which can be difficult to measure accurately. Higher fluxes will cause the calculation of permeability to be low if Darcy's Law is employed. A procedure to estimate the maximum allowable pressure gradient as a function of permeability, to avoid significant permeability errors due to inertial resistance, is presented in 6.2.1.3. An alternative is to make multiple flow rate measurements and employ Forchheimer's equation to obtain both permeability and inertial resistivity of each sample. As a practical matter, this is most quickly accomplished with the pressure-transient technique presented in 6.4.1.1.

6.1.1.3 Klinkenberg Gas Slippage

Even when inertial effects are properly accounted for, the permeability of a porous medium to gas is dependent upon the mean free path of the flowing gas, hence among other things, upon its absolute pressure. This is due to a phenomenon known as slip, a fact first pointed out to the oil industry by Klinkenberg.[3] Gas slippage had been overlooked or ignored by prior investigators of permeability despite theory and experimental data for slip in the flow of gases through small capillary tubes presented in scientific literature as early as 1875.[4] When gas slippage is ignored, permeability calculated from the Forchheimer equation, or from Darcy's Law (provided that inertial resistance is negligible), is higher than that obtained using a non-reactive liquid. When expressed as a percentage, this difference is small for high permeability samples, but becomes progressively larger with decreasing permeability. It is minimized by using high mean pore pressures in gas permeability measurements.

To avoid the problem of obtaining pore-pressure-dependent gas permeabilities, Klinkenberg presented a method in which gas permeability measurements made at several different mean pore pressures can be extrapolated to infinite pore pressure. He showed that this extrapolated gas permeability (now called "Klinkenberg permeability", k_∞) is equal to the permeability obtained using a non-reactive liquid, such as a clean, refined hydrocarbon.

Permeability of rock samples, especially those that contain certain clays, can be altered when they are cleaned and dried in preparation for gas permeability measurements. Often, k_∞ is higher in these samples than permeability measured using aqueous solutions. Special cleaning and/or drying techniques may be required.

6.1.1.4 Liquids

The use of liquids for permeability measurements eliminates the problem of gas slippage, and at reasonable and usual flow rates, inertial resistance is generally negligible. Thus, Darcy's Law can be used directly to calculate permeability from a single flow rate measurement. However, potential permeability alteration from interaction of rock constituents and liquids (especially aqueous solutions), fines movement, and microbial plugging requires special attention. Also, the liquid remaining in a sample may have to be removed before other measurements can be performed. Because of these problems, most routine permeability measurements have been made using gases. However, for some samples, such as those sensitive to drying techniques, liquid permeability measurements are considered to be the only acceptable alternative.

6.1.1.5 Confining Stress Dependence

Permeability of a porous medium is sensitive to the magnitude of net compressive stresses to which the medium is subjected, and to its stress history. As confining stresses are increased in a sample holder, the rock's permeability decreases. The decrease observed, when confining stresses are increased from 200 or 300 psi to several thousand psi, varies considerably—from just a few percent for reasonably permeable, well-cemented samples, to an order of magnitude or more for low permeability samples that contain micro fractures. In general, it is desirable to approximate reservoir stresses in laboratory permeability measurements to obtain values closer to expected in situ reservoir permeabilities. Additional, smaller permeability decreases may also be observed as a function of time after confining stresses are raised—due to creep. Creep is most prevalent at compressive stresses just lower than those that cause sample failure.

6.1.2 Unit of Permeability

6.1.2.1 Traditional Unit

In order to make practical use of the concept that porous media possess the measurable property of permeability, an arbitrary standard unit, the "darcy," has been defined. A porous medium has a permeability of one darcy when a single-phase fluid of one centipoise viscosity that completely fills the voids of the medium will flow through it under "conditions of Stokes flow" at a rate of 1 cm^3/s per square centimeter of cross-sectional area under a pressure or equivalent hydraulic gradient of 1 atm/cm. "Condition of Stokes flow" shall mean simply that "the rate of flow be sufficiently low to be directly proportional to the pressure or hydraulic gradient." The unit of permeability (the darcy), as a coefficient of proportionality between physical quantities, possesses dimensions of length squared. Permeability represents a property of the medium alone and is independent of the fluid, except as hereinafter qualified. For convenience the millidarcy sub-unit (equal to 0.001 darcy) may be used. The spelling of the plural forms of the unit have been standardized and established in the literature as "darcys" and "millidarcys." Although other sub-units are not recommended, the microdarcy (equal to 0.001 millidarcy or 10^{-6} darcy) is often used in conjunction with low-permeability gas sands. Also, the nanodarcy (equal to 0.001 microdarcy or 10^{-9} darcy) is often referred to for very tight rocks, such as micro fractured granite.

6.1.2.2 SI Unit

The fundamental SI unit of permeability, m^2, is defined as follows: a permeability of one meter squared will permit a flow of 1 m^3/s of fluid of 1 Pa·s viscosity through an area of 1 m^2 under a pressure gradient of 1 Pa/m. One darcy equals 0.986923×10^{-12} m^2. Because of the small exponent, this is an awkward unit for common usage. The Society of Petroleum Engineers of AIME preferred permeability unit is the micrometer squared (μm^2). One darcy (the traditional unit) equals 0.986923 μm^2.

6.1.2.3 Typical Units Used in Hydrology

Most environmental engineers have a background in hydrology, in which the terms intrinsic permeability, hydraulic conductivity, and transmissivity are used. The physics of fluid flow that environmental engineers, core analysts, or petroleum engineers deal with is the same, but the language used by one group is often foreign to another group. This arises from different applications. Hydrologists most often deal with the flow of ground water in aquifers, in which water viscosity, density, and compressibility exhibit rather small variations. In these aquifers, it is often convenient to determine flow potential differences by measuring differences in liquid heads—the elevations to which water rises in small non-flowing test wells drilled into the aquifer. In the laboratory, an analogous measurement technique would be to attach a series of water manometers to a core sample through which water is flowing. The height of water in the manometer nearest the upstream end of the core would be greatest, and water-level elevations (from a horizontal reference) would be progressively lower further downstream.

In petroleum reservoirs, fluid properties vary widely, more than one fluid phase is usually present, and pressures are often too great to make manometric head measurements. Pressures are measured directly with pressure gauges or transducers. Thus, equations for these applications deal with pressures, not heads. Flow potentials must be calculated from

pressures, flow rates, and fluid and rock properties through appropriate governing partial differential equations. Solution often requires numerical finite difference approximations of these equations.

6.1.2.3.1 Flow Potential Differences vs. Pressure Differences

It is important to understand that flow potential difference, not pressure difference, is the driving force for fluid flow, to know what flow potential is, and to ascertain when pressure differences can legitimately be used in flow equations. Relationships between flow potential and pressure are illustrated by Figure 6-1. Two tanks, each open to atmospheric pressure at its top and fitted with manometer tubes at several depths, are connected together by a small pipe. Each is partially filled with water to the same depth. Pressure in each tank at the air-water interface is atmospheric, and increases with depth.

We observe that the water level in every manometer tube is the same, and except for a very small capillary rise in each tube, it is the same as the water level inside the tanks. This water level in the manometer tubes is a measure of the flow potential inside the tanks, that is the same at every depth, even though pressure increases with depth. If pressure, alone, were the driving force for flow, higher pressure water from the bottom of a tank would flow upward, toward lower pressure. This is not observed. If we assume that the density of water everywhere in each tank is constant (a very close approximation), then flow potential, φ, everywhere is:

$$\varphi = p - \rho g z / C_4 \quad (1)$$

Where:

p = pressure at depth z (below the water surface; z increases downward).
ρ = density of the water.
g = local acceleration of gravity.
C_4 = conversion factor (found in Table 6-1) to make units consistent.

However, pressure at any depth in the tank is:

$$p = p_1 + \rho g z / C_4 \quad (2)$$

Where p_1 is the pressure at the air-water interface, atmospheric pressure in this case. Thus, combining Equations 1 and 2, we see that $\varphi = p_1$, and flow potential is the same at any depth. It is also the same in both tanks; so no flow occurs within or between tanks.

Condition 1:
a. Both tanks are filled with water to same depth, and are open to atmospheric pressure.
b. Water pressure increases in both tanks with increasing depth.
c. However, flow potential (indicated by water levels in manometer tubes) is the same for all depths, and is the same in both tanks.
d. Therefore, no flow occurs within or between tanks.

Condition 2:
a. Air pressure above water in left tank is increased. Then vent is capped.
b. Flow potential in left tank is increased as indicated by increased water levels in manometer tubes there.
c. Water flows from left tank to right tank until water levels in manometer tubes are the same for both tanks.

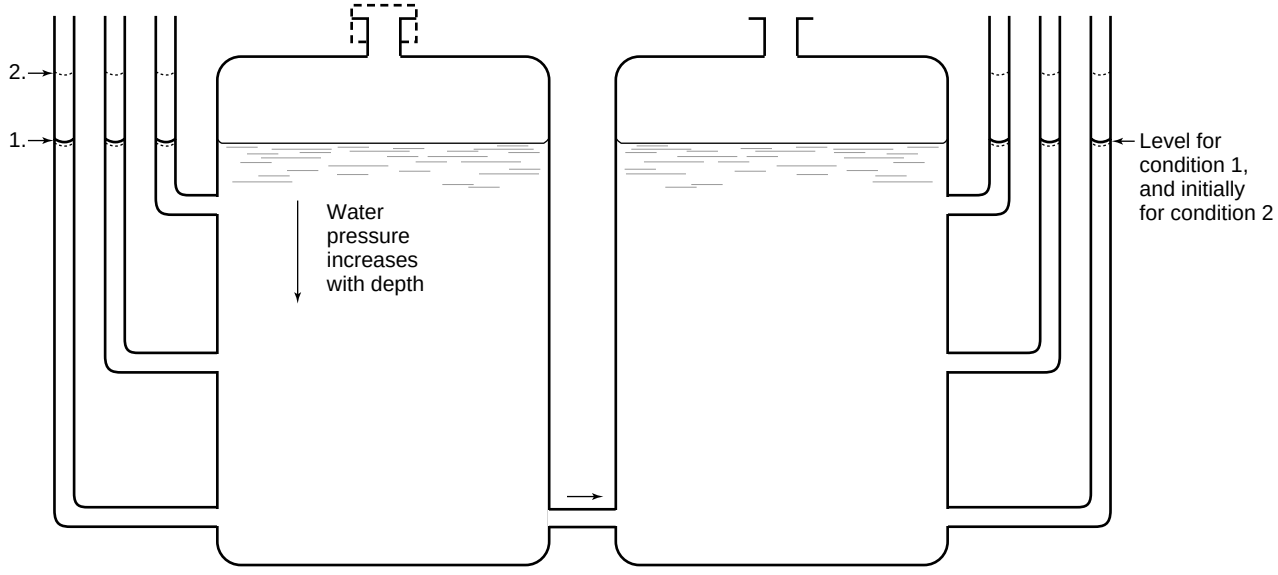


Figure 6-1—Illustration of Flow Potential vs. Pressure Differences

Table 6-1—Constants in Forchheimer or Darcy Equation with Various Sets of Units

Variable or Constant	Units or Values of Constants			
	SI	Preferred SPE	Traditional	Common Usage
A, area perpendicular to flow	m^2	m^2	cm^2	cm^2
b, Klinkenberg slip factor	Pa	Pa	atm	psi
D, diameter of sample	m	m	cm	cm
g, gravitational acceleration	m/s^2	m/s^2	cm/s^2	cm/s^2
k, permeability	m^2		darcy	millidarcy
L, length of sample	m	m	cm	cm
M, gas molecular weight	kg/kg-mole	kg/kg-mole	g/g-mole	g/g-mole
P, absolute pressure	Pa	Pa	atm	psia
p, gauge pressure	Pa	Pa	atm	psig
Δp, differential pressure	Pa	Pa	atm	psi
q, volumetric flow rate				
r, radius of sample or seal	m	m	cm	cm
S, compressive storage				
s, flow-direction distance	m	m	cm	cm
T, absolute temperature	K	K	K	K
V_p, pore volume				
V_b, total reservoir volume				
v_s, volumetric flux	m/s	m/s	cm/s	cm/s
β, inertial resistivity	m^{-1}	m^{-1}	cm^{-1}	$ft.^{-1}$
ρ, gas or liquid density	kg/m^3	kg/m^3	g/cm^3	g/cm^3
μ, gas or liquid viscosity	Pa·s	Pa·s	cp	cp
R, gas-law constant	8314	8314	82.05	1205.8
C_1, in Darcy or Forch. eq.	1.0	1.0	1.0	6.8046E-2
C_2, in Darcy or Forch. eq.	1.0	1.0E+12	1.0	1000.
C_3, constant in Forch. eq.	1.0	1.0	9.8692E-7	3.2379E-8
C_4, constant in eqs. with g	1.0	1.0	1.0133E+6	68 950.

Now, suppose air is pumped into the left tank through its vent so that air pressure above the water is greater than atmospheric pressure. Then the vent is capped. We would observe that water levels in all manometers attached to the left tank have risen equally. Except for the amount of water required to increase levels in the tubes, the water level inside the tank is unchanged. Initially, conditions in the right tank are unchanged. From the higher manometer levels in the left tank (indicated by dashed lines in Figure 6-1), relative to those in the right tank, we conclude that flow potential is higher in the former, even though water levels inside the two tanks are the same. Indeed, water flows from left to right until manometer levels are the same for both tanks. When flow ceases, the water level inside the left tank will be lower than in the right tank.

In laboratory single-phase gas permeability measurements, "gas head" differences are generally smaller than the accuracy of pressure measurements, and can be ignored. Therefore pressure differences or pressure gradients are very close approximations of flow potential differences or gradients in any flow orientation (horizontal or vertical). Conditions for single-phase liquid measurements are discussed in detail in 6.2.2. Pressure differences or gradients can be substituted for flow potential differences or gradients only in special, restricted situations.

6.1.2.3.2 Intrinsic Permeability

Intrinsic Permeability, k, as used by hydrologists is identical to permeability defined in 6.1.2.2, even though it is defined in terms of heads and kinematic viscosity, v, rather than dynamic viscosity, μ, used in the Darcy equation as defined in 6.1.2.1. Kinematic viscosity is equal to dynamic viscosity divided by density of the fluid. A porous medium has an intrinsic permeability of one unit of length squared if it will transmit in unit time a unit volume of fluid of unit kinematic viscosity through a cross section of unit area measured at right angles to the flow direction under a unit potential gradient. If SI units are used, intrinsic permeability has the unit of m^2.

6.1.2.3.3 Hydraulic Conductivity

Hydraulic conductivity, K, is not solely a property of a porous medium. It depends also upon the kinematic viscosity of the flowing fluid, and is appropriate only for ground water aquifers. Its units are those of velocity, usually feet per day or meters per day. A medium has a hydraulic conductivity of

unit length per unit time if it will transmit in unit time a unit volume of ground water at the prevailing viscosity through a cross section of unit area, measured at right angles to the direction of flow, under a hydraulic gradient of unit change in head through unit length of flow. Hydraulic conductivity is related to permeability (or intrinsic permeability) by:

$$K = \frac{k\rho g}{\mu} \quad (3)$$

Where k is permeability, and the other symbols are defined above. Any consistent set of units may be used in Equation 3. With SI units, K will have the unit of meters per second.

6.1.2.3.4 Transmissivity

The transmissivity, T, of a ground water aquifer includes the thickness of the aquifer, b, and the kinematic viscosity of the confined water. Transmissivity is the rate at which water of the prevailing kinematic viscosity is transmitted through a unit width of the aquifer under a unit hydraulic gradient. It has the unit of length squared per unit time, often ft^2/day, or m^2/day. Transmissivity is equal to Kb. It can be calculated from permeability by:

$$T = \frac{k\rho g b}{\mu} \quad (4)$$

Definitions for intrinsic permeability, hydraulic conductivity, and transmissivity are taken from Lohman.[42] Conversion factors for several sets of units can be found in *Ground Water* (1993).[43]

6.1.3 Terminology

6.1.3.1 Specific Permeability

The definition of the standard unit of permeability requires that the porous medium contain only one homogeneous, single-phase fluid. The property of the medium so determined is properly termed specific permeability; but it shall simply be called permeability, and denoted by the symbol k. By this definition, the term permeability becomes a property solely of the porous medium; and the numerical value of permeability is constant and independent of the fluid used in the measurement. Whenever certain minerals and fluids are brought in contact and interaction results, or material is deposited from the fluid, this phenomenon, in effect, produces a new medium. The permeability of the new medium may differ from that of the unaltered medium. Whenever this occurs, it should be indicated clearly by appropriate references to both medium and fluid, e.g., "the permeability of Woodbine sand to salt water (fresh water, crude oil, heptane, etc., as the case may be)."

Uncorrected gas permeabilities are precluded from the above definition due to gas slippage effects. Gas permeabilities, which shall be called k_g (or k_{air}, k_{N2}, k_{He}, etc.) depend upon the particular gas used in the permeability determination, and upon its mean pore pressure. Both must be specified to define the permeability of the medium. Only the Klinkenberg, slip-corrected gas permeability, when unaffected by high velocity (Forchheimer) effects, is constant and independent of the fluid used in the measurement.

Because the stress condition under which a measurement is made can have a significant effect upon permeability, it also should be specified. Examples are: k_{air} with a mean pore pressure of 32.3 psia and a radial confining stress of 400 psig; or k_∞ at a net hydrostatic stress of 5,000 psi. The first specification implies that a gas permeability was obtained using air at an average absolute pore pressure of 32.3 psia, and that a pressure of 400 psig was applied to the rubber boot of the sample holder. The net radial stress is therefore 400 + 14.7 – 32.3, or about 382 psi. The net axial stress is unknown. In the second example, a Klinkenberg (slip-corrected) permeability was obtained that is independent of the gas used or its mean pore pressure (except as pore pressure affects net stress). Equal magnitude radial and axial ("hydrostatic") net stresses of 5,000 psi were applied during the measurement.

6.1.3.2 Effective and Relative Permeability

This manual of routine recommended practices deals only with single-phase permeability measurements. Therefore, effective permeability and relative permeability will only be defined. The effective permeability of a porous medium is a measure of its fluid conductivity to a particular phase of a multi-phase fluid system residing within the medium, where the saturation of each phase (the fraction of its total effective pore volume that is filled by each phase) is specified. Relative permeability is the ratio of the effective permeability of a particular fluid phase, at definite saturations of all phases, to some arbitrary reference permeability. The reference permeability can be the sample's specific permeability, its Klinkenberg permeability, the effective permeability of one of the fluid phases at a specified saturation condition, etc. The particular reference permeability used must be specified.

Book three of the five volume, *History of Petroleum Engineering* (API, 1961),[39] is a rich reference source for early work on laboratory permeability measurements, including the pioneering efforts of Fancher, Lewis, and Barnes,[40] and of Wyckoff, Botset, Muskat, and Reed.[41]

6.2 THEORY

Single-phase permeability measurements can be separated into four major categories: those utilizing flowing gas or liquid under steady-state or unsteady-state (transient) conditions. Equations for steady-state flow of gases and liquids are presented in this section. Several practical variations, each with particular advantages and limitations, will be outlined in 6.3, and the theory and application of transient pressure gas

and liquid measurements are presented in 6.4. These are all considered to be direct measurements.

The permeability of a porous medium can also be inferred from empirical correlations using indirect measurements, such as those of inter granular porosity and grain size (Kozeny[5] and Carman[6]); porosity and irreducible water saturation (Wyllie and Rose[7]); pore-throat and pore-size distribution by mercury injection or capillary pressure measurements (Swanson[8], Katz and Thompson[9], etc.); effective electrical conductivity (Archie[10], Ondracek[11]); transmissivity of acoustic waves (Biot[12]); electrokinetic emf (Moran[13]); etc. Because these can be less accurate and often more time consuming than making the direct measurements, they will not be considered here.

All measurements are assumed to be conducted under isothermal conditions, which may require considerable technique and effort to achieve under certain circumstances. Also, even though temperature can affect permeability, most routine permeability measurements are made at, or near, room temperature.

Tables 6-2 and 6-3 are quick selection and reference guides for gas and liquid permeability determinations, respectively, using both steady-state and transient techniques. They list major advantages and disadvantages of each technique, its approximate permeability range, and particular application. They are intended to be a starting point only, and the reader is cautioned to be aware of details given in sections referenced by these tables.

Table 6-2—Quick Selection and Reference Guide for Permeability Measurements Using Gases*

Reference Section Numbers	Type of Measurement	Approx. Perm. Range, md	Apparatus or Application	Major Advantages	Major Limitations
6.3.1.1 6.3.1.1.1.1	Axial flow, steady state in core plugs	0.1-10,000	Low pressure apparatus with manometers, orifice flow meters	Low capital cost; simple manual system; workhorse for decades; large data base for comparison	Labor intensive; high operating cost; low-stress perms; no slip correction; must check for inertial resistance
6.3.1.1 6.3.1.1.1.2	Axial flow, steady state in core plugs	0.1-10,000	Apparatus with electronic sensors, high pressure. core holder	Can be automated; reservoir stresses can be approximated; better precision and accuracy than with manual system	Must make multiple measurements for gas slippage correction; must check to ensure negligible inertial resistance
6.4.1.1 B.6.8.2	Axial flow, pressure falloff in core plugs	0.001-30,000	Wide range; med. to high stress measurements with corrections for b and β	Well adapted for automation; no flow meters required; can yield reservoir-condition perms (k_∞), and k_g	Higher capital cost for automated system with high accuracy pressure transducers and data acquisition system
6.4.1.3 D.6.8.4	Axial flow, pulse-decay in core plugs	.00001-0.1	High stress apparatus for very low perms.	Only method for ultra-low perms; well adapted for automation; porosity can be determined in same apparatus	Requires high pressure, leak-tight system with high quality transducers and data acquisition system—higher capital cost
6.3.1.2	Probe perm., s.s., on whole core	1-10,000	Zero stress, high density, localized measurements for heterogeneous cores	No plug preparation required (core slabbing recommended); relatively fast; can be automated or made portable	Zero stress, non slip corrected perms are high at low end of range; prone to high inertial resistance at high end
6.4.1.2 C.6.8.3	Probe perm., pressure falloff on whole core	0.001-30,000	Zero stress, high density, localized measurements for heterogeneous cores	No plug preparation required (core slabbing recommended); very fast; automated; corrected for b, β	Zero stress perms are high, especially at low end of range; higher capital cost for automated system
6.3.1.3	Transverse, s.s. perm. in whole core	0.02-500	Directional perm. in whole core (or plug) for k_{max} and k_{90*}	Can measure "horizontal" perm. in various directions; averaging obtained using whole-core sample.	Cleaning and preparation of whole core sample more expensive; only k_g obtained without multiple measurements
6.3.1.4	Radial, s. s. perm. in whole core	0.01-250	Average permeability in all radial directions in whole core samples	Measures average "horizontal" permeability in large sample	Difficult to prepare samples; no radial stress; perm. critically dependent on condition of central "wellbore"

*Major advantages of using gas rather than liquid:

a. Easy to use—does not require special saturation techniques.
b. Non-reactive with rock; non-corrosive to equipment.
c. No post-measurement cleanup required.
d. Less prone than liquid to mobilizing fines in rock sample.
e. Does not support microbial growth, nor require special filtration.

Major disadvantages:

a. Requires correction for gas slippage—especially with lower perms.
b. Prone to significant high-velocity inertial resistance in high perm. rock.
c. Necessary leak-tightness harder to achieve than with liquids.
d. In some cases, may be less representative of permeability in reservoir.

Table 6-3—Quick Selection and Reference Guide for Permeability Measurements Using Liquids

Reference Section Numbers	Type of Measurement	Approx. Perm. Range, md	Apparatus or Application	Major Advantages	Major Limitations
6.3.2.2	Axial, s.s. flow in plugs using liquid heads	1000-40,000	Gravity flow of liquid through high perm., cylindrical core plugs	Simple, low cost equipment (but does require electronic balance for flow rate determination)	Low back pressure—difficult to ensure that no gas remains in core plug.
6.3.2.1	Axial flow, steady state in core plugs	0.1-20,000	Apparatus with electronic sensors, high pressure. core holder, imposed	Can be automated; reservoir stresses can be approximated; may be most representative measurement of perm. in reservior	Corrosion resistant, moderate to high pressure pumping and control equipment is expensive
6.4.2.1 E.6.8.5	Axial flow, pulse-decay in core plugs	.00001-0.1	High stress apparatus for very low perms.	Only method for ultra-low perms; well adapted for automation; no flow meters required: rate is calc. from Δp & t	Requires high pressure, leak-tight system with high quality transducers and data acquisition system—high capital cost
6.3.2.1 6.3.1.3.1	Transverse, s.s. flow in whole cores	0.005-500	Directional perm. in whole core (or plug) for k_{max} and $k_{90°}$	Can measure "horizontal" perm. in various directions; averaging obtained using whole-core sample.	Same as for s.s. axial flow (above); cleaning, preparation and handling of whole core sample more expensive
6.3.1.4 6.3.1.4.2	Radial, steady state flow in whole cores	0.002-250	Average permeability in all radial directions in whole core samples	Measures average "horizontal" permeability in large sample	Same as above; difficult to prepare samples; no radial stress; perm. critically dependent on condition of central "wellbore"

6.2.1 Steady-State Permeability Equations For Gases

The general equation for steady-state gas permeability measurements will be presented first. It includes provision for gas slippage and inertial resistance effects, which become somewhat complicated and require more measurements than is usually practicable using steady-state measurements. However it serves to unify the theory of gas measurements and provides principles and guidelines for use of the Darcy equation, which is the basis for the practical steady-state cases that follow.

Unlike liquids, gases are highly compressible. Also, under normal laboratory conditions, the density of gas is sufficiently low that gravity effects may be ignored in making permeability measurements. The viscosity of air, nitrogen, or helium (see Tables 6-4, 6-5, and 6-6, respectively) is less than $^1/_{50}$ that of water at room temperature. Therefore, volumetric fluxes of gases are correspondingly higher for a given pressure gradient, and can deviate from conditions of Stokes flow, demanded by Darcy's Law. Finally, the phenomenon of gas slippage, or the Klinkenberg effect, requires special treatment. Because of these differences, the point of departure for developing equations pertaining to the measurement of permeability using gases is the differential form of the Forchheimer equation, into which the Klinkenberg relationship will be inserted:

$$\frac{-dP}{ds} = \frac{C_2 \mu q_s}{C_1 A k_g} + \frac{C_3 \beta \rho_s^{\,2}}{C_1 A^2} \qquad (5)$$

Where:

s = distance along the direction of flow.
q_s = volumetric rate of gas flow through area A per unit time.
A = area perpendicular to the flow of gas.
$\frac{dP}{ds}$ = pressure gradient along s to which q_s / A refers.
μ = dynamic gas viscosity.
k_g = apparent permeability of the medium to a particular gas.
β = coefficient of inertial resistivity.
ρ = gas density.

The constants C_1, C_2, and C_3 allow for various sets of dimensions to be used in the gas-flow equations that follow. Table 6-1 shows values of these constants for SI, preferred-SPE, traditional, or common-usage dimensions. Note that an upper case P appears in the equation above. This non-standard symbol denotes that the pressure is *absolute*. A lower-case p is used to denote a *gauge* or *differential* pressure in any equation below. These conventions have been adopted in this document because of the confusion between the two pressures, and the frequent error made when a measured gauge-pressure is not converted to an absolute pressure when required. Thus, in keeping with this convention:

$$P = p + P_a \qquad (6)$$

Where P_a is the true ambient barometric pressure, converted to the proper units (not the barometric pressure from the local airport, which has been converted to sea-level pressure).

The density of a real gas is:

$$\rho = \frac{MP}{zRT} \qquad (7)$$

Table 6-4—Viscosity of Air (for Degrees F and C) at One Atmosphere*

Viscosity of air at one atmosphere, micropoises												
	Temperature, °F						Temperature, °C					
plus	50°F	60°F	70°F	80°F	90°F	100°F	0°C	10°C	20°C	30°C	40°C	50°C
0	176.8	179.6	182.3	185.0	187.7	190.4	171.8	176.8	181.8	186.6	191.4	196.2
1	177.1	179.9	182.6	185.3	188.0	190.6	172.3	177.3	182.3	187.1	191.9	196.6
2	177.4	180.1	182.9	185.6	188.2	190.9	172.8	177.8	182.8	187.6	192.4	197.1
3	177.7	180.4	183.1	185.8	188.5	191.2	173.3	178.3	183.2	188.1	192.9	197.6
4	177.9	180.7	183.4	186.1	188.8	191.4	173.8	178.8	183.7	188.6	193.3	198.0
5	178.2	181.0	183.7	186.4	189.0	191.7	174.3	179.3	184.2	189.0	193.8	198.5
6	178.5	181.2	184.0	186.6	189.3	192.0	174.8	179.8	184.7	189.5	194.3	198.9
7	178.8	181.5	184.2	186.9	189.6	192.2	175.3	180.3	185.2	190.0	194.7	199.4
8	179.0	181.8	184.5	187.2	189.8	192.5	175.8	180.8	185.7	190.5	195.2	199.9
9	179.3	182.1	184.8	187.4	190.1	192.8	176.3	181.3	186.2	191.0	195.7	200.3

*Calculated from Sutherland's equation:

$$\mu_{air} = \frac{14.969T^{1.5}}{T + 120}$$

Where: T is absolute temperature, degrees Kelvin (degrees Celsius + 273), and μ_{air} is the viscosity of air at one atmosphere pressure, micropoises. Multiply by 1.0 E-04 to convert to centipoises, or by 1.0 E-07 to convert to Pa·s. Constants are based on work of Montgomery[35] and Birge[36].

Table 6-5—Viscosity of Nitrogen (for Degrees F and C) at One Atmosphere*

Viscosity of air at one atmosphere, micropoises												
	Temperature, °F						Temperature, °C					
plus	50°F	60°F	70°F	80°F	90°F	100°F	0°C	10°C	20°C	30°C	40°C	50°C
0	171.3	173.8	176.4	178.9	181.4	183.8	166.6	171.3	175.9	180.4	184.8	189.2
1	171.5	174.1	176.6	179.1	181.6	184.1	167.1	171.7	176.3	180.8	185.2	189.6
2	171.8	174.3	176.9	179.4	181.9	184.3	167.5	172.2	176.8	181.3	185.7	190.0
3	172.0	174.6	177.1	179.6	182.1	184.6	168.0	172.7	177.2	181.7	186.1	190.5
4	172.3	174.8	177.4	179.9	182.3	184.8	168.5	173.1	177.7	182.2	186.6	190.9
5	172.5	175.1	177.6	180.1	182.6	185.1	168.9	173.6	178.1	182.6	187.0	191.3
6	172.8	175.3	177.9	180.4	182.8	185.3	169.4	174.0	178.6	183.0	187.4	191.8
7	173.1	175.6	178.1	180.6	183.1	185.5	169.9	174.5	179.0	183.5	187.9	192.2
8	173.3	175.9	178.4	180.9	183.3	185.8	170.3	174.9	179.5	183.9	188.3	192.6
9	173.6	176.1	178.6	181.1	183.6	186.0	170.8	175.4	179.9	184.4	188.7	193.0

*Calculated from Sutherland's equation:

$$\mu_{N_2} = \frac{13.85T^{1.5}}{T + 102}$$

Where: *T* is absolute temperature, degrees Kelvin (degrees Celsius + 273), and μ_{N_2} is the viscosity of nitrogen at one atmosphere pressure, micropoises. Multiply by 1.0 E-04 to convert to centipoises, or by 1.0 E-07 to convert to Pa·s. Constants are based on the work of Licht and Stechert[37].
The viscosity of nitrogen at temperatures reasonably close to 25°C and pressures to 253 atmospheres can be calculated from:

$$\mu_{N_2}[T, P] = \mu_{N_2}[T, 1] - 0.12474 + 0.123688P + 1.05452\text{E-}03P^2 - 1.5052\text{E-}06P^3$$

Where: $\mu_{N_2}[T, 1]$ is the viscosity of nitrogen at one atmosphere pressure (from the tables above), micropoises, and *P* is pressure, atmospheres. The pressure dependence is fitted to the data of Gracki, *et al.*[34] to within a maximum deviation of 0.1%. Pressure dependence at 0°C and 50°C is nearly the same as at 25°C.

Table 6-6—Viscosity of Helium (for Degrees F and C) at One Atmosphere*

Viscosity of air at one atmosphere, micropoises												
	Temperature, °F						Temperature, °C					
plus	50°F	60°F	70°F	80°F	90°F	100°F	0°C	10°C	20°C	30°C	40°C	50°C
0	191.7	194.2	196.8	199.3	201.8	204.4	187.0	191.7	196.3	200.8	205.4	209.8
1	191.9	194.5	197.0	199.6	202.1	204.6	187.5	192.1	196.7	201.3	205.8	210.3
2	192.2	194.7	197.3	199.8	202.3	204.9	187.9	192.6	197.2	201.7	206.3	210.7
3	192.4	195.0	197.5	200.1	202.6	205.1	188.4	193.1	197.6	202.2	206.7	211.2
4	192.7	195.3	197.8	200.3	202.9	205.4	188.9	193.5	198.1	202.7	207.1	211.6
5	193.0	195.5	198.1	200.6	203.1	205.6	189.3	194.0	198.6	203.1	207.6	212.0
6	193.2	195.8	198.3	200.8	203.4	205.9	189.8	194.4	199.0	203.6	208.0	212.5
7	193.5	196.0	198.6	201.1	203.6	206.1	190.3	194.9	199.5	204.0	208.5	212.9
8	193.7	196.3	198.8	201.3	203.9	206.4	190.7	195.4	199.9	204.5	208.9	213.4
9	194.0	196.5	199.1	201.6	204.1	206.6	191.2	195.8	200.4	204.9	209.4	213.8

*Calculated from:

$$\mu_{He} = 187.0\left(\frac{T}{273.1}\right)^{0.685}$$

Where: T is absolute temperature, degrees Kelvin (degrees Celsius + 273), and μ_{He} is the viscosity of helium at one atmosphere pressure, micropoises. Multiply by 1.0 E-04 to convert to centipoises, or by 1.0 E-07 to convert to Pa·s. Constants are from Chapman and Cowling[38].
According to the data of Gracki, *et al.*[34] the viscosity of helium at 25°C passes through a very shallow minimum with pressure. At 37 atm. it is 0.43 percent lower than at 1 atm., and at 158 atm., its viscosity is 0.17 percent higher than at 1 atm.

Where *z* is the gas deviation factor, which is equal to 1.0 for an ideal gas. Values of *z* as functions of temperature and pressure are given in Tables 6-7, 6-8, and 6-9 for air, nitrogen, and helium, respectively. They are calculated from the Beattie-Bridgeman equation of state[14]. See Table 6-1 for values of the universal gas-law constant, *R*, for the various sets of units. *M* is the gas molecular weight. Temperature in Equation 7 is absolute, in degrees Kelvin:

$$\begin{aligned} T &= °\mathrm{C} + 273.15 \\ &= (°\mathrm{F} - 32)/1.8 + 273.15 \end{aligned} \quad (8)$$

6.2.1.1 Treatment of the Gas Slippage Factor

The Klinkenberg relationship, written as a point function (i.e., pertaining to a particular point in a sample, not an average value) is:

$$k_g = k_\infty\left(1 + \frac{b}{P}\right) \quad (9)$$

Note that k_g depends upon pressure. Therefore it cannot be treated as a constant when the Forchheimer (or Darcy) equation is integrated. In Equation 9, *b* must have the same units as those of the pressure used.

Unfortunately, the gas slippage factor, *b*, is partly a rock property and partly a gas property, which has caused confusion and some problems. For example, a *b* measured using air, but not indicated as such, might be misused in calculations involving a different gas with significantly different properties. It is a fairly simple procedure to separate the gas properties from the slip factor, so that only the rock-dependent property remains. According to Klinkenberg[3], *b* is related to the mean free path of gas molecules, $\bar{\lambda}$, by the relationship:

$$\bar{\lambda} = \frac{br}{4cP} \quad (10)$$

But, from the kinetic gas theory[15], the mean free path is given by:

$$\bar{\lambda} = \frac{1.881\mu}{P}\sqrt{\frac{RT}{M}} \quad (11)$$

In Equations 10 and 11, any consistent units may be used. From these two equations, and adjusting for the units given below, we obtain:

$$b = \left[99.5\mu\sqrt{\frac{T}{M}}\right]\left(\frac{c}{r}\right) \quad (12)$$

Where:

P = absolute gas pressure.
R = universal gas law constant.
b = Klinkenberg gas slippage factor, psi. (The constant 99.5 becomes 6.77 if b has the unit of atm).
μ = gas viscosity, cp.

Table 6-7—Gas Law Deviation Factors for Air, Calculated from Beattie-Bridgemann Equation of State[14]

P,	Z-Factors for Air at Temperatures (Degrees F) of:							
psia	55	60	65	70	75	80	85	90
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	0.99968	0.99970	0.99972	0.99973	0.99975	0.99977	0.99979	0.99980
20	0.99935	0.99939	0.99943	0.99947	0.99951	0.99954	0.99957	0.99961
30	0.99904	0.99910	0.99915	0.99921	0.99926	0.99931	0.99936	0.99941
40	0.99872	0.99880	0.99888	0.99895	0.99902	0.99909	0.99916	0.99922
50	0.99840	0.99850	0.99860	0.99869	0.99878	0.99887	0.99895	0.99903
60	0.99809	0.99821	0.99833	0.99844	0.99854	0.99865	0.99875	0.99884
70	0.99778	0.99792	0.99806	0.99818	0.99831	0.99843	0.99854	0.99866
80	0.99747	0.99763	0.99779	0.99793	0.99808	0.99821	0.99834	0.99847
90	0.99717	0.99735	0.99752	0.99769	0.99784	0.99800	0.99815	0.99829
100	0.99687	0.99706	0.99726	0.99744	0.99762	0.99779	0.99795	0.99811
110	0.99657	0.99678	0.99699	0.99720	0.99739	0.99758	0.99776	0.99793
120	0.99627	0.99651	0.99673	0.99695	0.99717	0.99737	0.99757	0.99776
130	0.99597	0.99623	0.99648	0.99671	0.99694	0.99716	0.99738	0.99758
140	0.99568	0.99596	0.99622	0.99648	0.99672	0.99696	0.99719	0.99741
150	0.99539	0.99568	0.99597	0.99624	0.99651	0.99676	0.99701	0.99724
160	0.99510	0.99541	0.99572	0.99572	0.99601	0.99601	0.99629	0.99656
170	0.99481	0.99515	0.99547	0.99578	0.99608	0.99637	0.99664	0.99691
180	0.99453	0.99488	0.99522	0.99555	0.99587	0.99617	0.99646	0.99675
190	0.99425	0.99462	0.99498	0.99533	0.99566	0.99598	0.99629	0.99659
200	0.99397	0.99436	0.99474	0.99510	0.99545	0.99579	0.99611	0.99643
210	0.99369	0.99410	0.99450	0.99488	0.99525	0.99560	0.99594	0.99627
220	0.99342	0.99385	0.99426	0.99466	0.99505	0.99542	0.99577	0.99612
230	0.99315	0.99360	0.99403	0.99445	0.99485	0.99523	0.99560	0.99596
240	0.99288	0.99335	0.99380	0.99423	0.99465	0.99505	0.99544	0.99581
250	0.99261	0.99310	0.99357	0.99402	0.99445	0.99487	0.99528	0.99566
260	0.99235	0.99285	0.99334	0.99381	0.99426	0.99470	0.99511	0.99552
300	0.99132	0.99190	0.99246	0.99299	0.99351	0.99401	0.99449	0.99496
400	0.98891	0.98968	0.99041	0.99112	0.99180	0.99246	0.99.09	0.99370
500	0.98676	0.98770	0.98861	0.98948	0.99032	0.99113	0.99190	0.99265
600	0.98486	0.98598	0.98705	0.98808	0.98907	0.99002	0.99094	0.99183
700	0.98322	0.98450	0.98573	0.98692	0.98805	0.98915	0.99020	0.99122
800	0.98185	0.98329	0.98467	0.98600	0.98728	0.98850	0.98968	0.99082
900	0.98074	0.98233	0.98386	0.98532	0.98673	0.98809	0.98939	0.99065
1000	0.97989	0.98163	0.98329	0.98489	0.98642	0.98790	0.98932	0.99069
1100	0.97931	0.98118	0.98297	0.98470	0.98635	0.98794	0.98947	0.99094
1200	0.97900	0.98099	0.98290	0.98474	0.98651	0.98821	0.98984	0.99141
1300	0.97894	0.98105	0.98308	0.98503	0.98690	0.98869	0.99042	0.99208
1400	0.97915	0.98137	0.98350	0.98555	0.98751	0.98940	0.99122	0.99296
1500	0.97961	0.98193	0.98416	0.98630	0.98835	0.99033	0.99223	0.99405
1600	0.98032	0.98273	0.98505	0.98728	0.98941	0.99147	0.99344	0.99534
1700	0.98128	0.98378	0.98618	0.98848	0.99069	0.99281	0.99485	0.99682
1800	0.98248	0.98505	0.98752	0.98990	0.99218	0.99436	0.99647	0.99849
1900	0.98391	0.98656	0.98909	0.99153	0.99387	0.99611	0.98827	1.00034
2000	0.98558	0.98828	0.99088	0.99337	0.99576	0.99805	1.00026	1.00238
2100	0.98747	0.99022	0.99287	0.99540	0.99784	1.00018	1.00243	1.00460
2200	0.98957	0.99237	0.99506	0.99764	1.00011	1.00249	1.00478	1.00698
2300	0.99189	0.99472	0.99744	1.00006	1.00257	1.00498	1.00730	1.00953
2400	0.99440	0.99727	1.00002	1.00266	1.00520	1.00763	1.00998	1.01223
2500	0.99711	1.00000	1.00277	1.00543	1.00799	1.01045	1.01282	1.01509
2600	1.00000	1.00291	1.00570	1.00838	1.01095	1.01343	1.01581	1.01810
2700	1.00307	1.00599	1.00879	1.01148	1.01407	1.01655	1.01894	1.02124
2800	1.00631	1.00923	1.01204	1.01474	1.01733	1.01983	1.02222	1.02794
2900	1.00971	1.01264	1.01545	1.01815	1.02074	1.02323	1.02563	1.02794
3000	1.01327	1.01619	1.01900	1.02169	1.02428	1.02678	1.02917	1.03147

P,	Z-Factors for Air at Temperatures (Degrees C) of:							
psia	0	5	10	15	20	25	30	35
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	0.99957	0.99962	0.99966	0.99969	0.99973	0.99976	0.99979	0.99982
20	0.99915	0.99923	0.99931	0.99939	0.99946	0.99952	0.99958	0.99964
30	0.99873	0.99886	0.99897	0.99908	0.99919	0.99928	0.99937	0.99946
40	0.99831	0.99848	0.99864	0.99878	0.99892	0.99905	0.99917	0.99928
50	0.99790	0.99811	0.99830	0.99848	0.99866	0.99882	0.99897	0.99911
60	0.99748	0.99773	0.99797	0.99819	0.99839	0.99859	0.99876	0.99893
70	0.99707	0.99736	0.99764	0.99789	0.99813	0.99836	0.99857	0.99876
80	0.99666	0.99700	0.99731	0.99760	0.99788	0.99813	0.99837	0.99859
90	0.99626	0.99663	0.99698	0.99731	0.99762	0.99791	0.99818	0.99843
100	0.99585	0.99627	0.99666	0.99703	0.99737	0.99769	0.99798	0.99826
110	0.99545	0.99591	0.99634	0.99674	0.99712	0.99747	0.99779	0.99810
120	0.99505	0.99555	0.99602	0.99646	0.99687	0.99725	0.99761	0.99794
130	0.99466	0.99520	0.99571	0.99618	0.99662	0.99703	0.99742	0.99778
140	0.99426	0.99485	0.99539	0.99590	0.99638	0.99682	0.99724	0.99762
150	0.99387	0.99450	0.99508	0.99563	0.99613	0.99661	0.99705	0.99747
160	0.99349	0.99415	0.99477	0.99535	0.99589	0.99640	0.99687	0.99732
170	0.99310	0.99381	0.99447	0.99508	0.99566	0.99619	0.99670	0.99717
180	0.99272	0.99347	0.99416	0.99481	0.99542	0.99599	0.99652	0.99702
190	0.99234	0.99313	0.99386	0.99455	0.99519	0.99579	0.99635	0.99687
200	0.99196	0.99279	0.99356	0.99428	0.99496	0.99559	0.99618	0.99673
210	0.99159	0.99246	0.99327	0.99402	0.99473	0.99539	0.99601	0.99659
220	0.99121	0.99212	0.99297	0399377	0.99450	0.99520	0.99584	0.99645
230	0.99084	0.99180	0.99268	0.99351	0.99428	0.99500	0.99568	0.99631
240	0.99048	0.99147	0.99239	0.99326	0.99406	0.99481	0.99552	0.99617
250	0.99011	0.99115	0.99211	0.99300	0.99384	0.99462	0.99535	0.99604
260	0.98975	0.99082	0.99182	0.99275	0.99362	0.99444	0.99520	0.99591
300	0.98833	0.98957	0.99071	0.99178	0.99278	0.99371	0.99459	0.99540
400	0.98498	0.98660	0.98812	0.98952	0.99084	0.99207	0.99321	0.99428
500	0.98190	0.98391	0.98578	0.98751	0.98913	0.99064	0.99206	0.99337
600	0.97911	0.98149	0.98370	0.98576	0.98767	0.98945	0.99112	0.99268
700	0.97662	0.97935	0.98189	0.98425	0.98645	0.98850	0.99041	0.99219
800	0.97442	0.97750	0.98035	0.98301	0.98547	0.98777	0.98992	0.99192
900	0.97253	0.97593	0.97908	0.98202	0.98474	0.98728	0.98965	0.99185
1000	0.97093	0.97465	0.97809	0.98129	0.98426	0.98702	0.98960	0.99200
1100	0.96965	0.97366	0.97737	0.98081	0.98401	0.98699	0.98977	0.99235
1200	0.96867	0.97295	0.97692	0.98060	0.98402	0.98719	0.99016	0.99291
1300	0.96800	0.97254	0.97674	0.98064	0.98426	0.98762	0.99076	0.99368
1400	0.96763	0.97241	0.97683	0.98093	0.98474	0.98828	0.99157	0.99464
1500	0.96756	0.97256	0.97718	0.98147	0.98545	0.98915	0.99260	0.99580
1600	0.96779	0.97299	0.97780	0.98226	0.98640	0.99024	0.99382	0.99716
1700	0.96832	0.97369	0.97867	0.98329	0.98757	0.99155	0.99525	0.99870
1800	0.96913	0.97466	0.97979	0.98455	0.98896	0.99306	0.99688	1.00043
1900	0.97021	0.97590	0.98116	0.98604	0.99057	0.99478	0.99869	1.00234
2000	0.97157	0.97738	0.98276	0.98775	0.99238	0.99669	1.00069	1.00442
2100	0.97320	0.97912	0.98460	0.98968	0.99440	0.99879	1.00287	1.00668
2200	0.97508	0.98109	0.98666	0.99182	0.99662	1.00108	1.00523	1.00909
2300	0.97720	0.98329	0.98893	0.99416	0.99902	1.00354	1.00775	1.01167
2400	0.97957	0.98571	0.99141	0.99670	1.00161	1.00618	1.01044	1.01440
2500	0.98216	0.98835	0.99410	0.99943	1.00438	1.00899	1.01328	1.01728
2600	0.98497	0.99120	0.99697	1.00233	1.00732	1.01196	1.01627	1.02030
2700	0.98799	0.99423	1.00003	1.00541	1.01042	1.01507	1.01941	1.02345
2800	0.99120	0.99746	1.00327	1.00866	1.01367	1.01834	1.02269	1.02674
2900	0.99461	1.00086	1.00667	1.01206	1.01708	1.02175	1.02610	1.03016
3000	0.99820	1.00444	1.01023	1.01562	1.02063	1.02529	1.02964	1.03369

Table 6-8—Gas Law Deviation Factors for Nitrogen, Calculated from Beattie-Bridgemann Equation of State[14]

P,	Z-Factors for Air at Temperatures (Degrees F) of:							
psia	55	60	65	70	75	80	85	90
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	0.99975	0.99977	0.99979	0.99981	0.99983	0.99984	0.99986	0.99987
20	0.99950	0.99954	0.99958	0.99962	0.99965	0.99969	0.99972	0.99975
30	0.99926	0.99932	0.99938	0.99943	0.99948	0.99953	0.99958	0.99963
40	0.99902	0.99910	0.99917	0.99925	0.99932	0.99938	0.99945	0.99951
50	0.99878	0.99888	0.99897	0.99907	0.99915	0.99924	0.99932	0.99940
60	0.99855	0.99867	0.99878	0.99889	0.99899	0.99909	0.99919	0.99928
70	0.99832	0.99845	0.99858	0.99871	0.99883	0.99895	0.99906	0.99917
80	0.99809	0.99824	0.99839	0.99854	0.99868	0.99881	0.99894	0.99906
90	0.99786	0.99804	0.99820	0.99837	0.99852	0.99867	0.99882	0.99896
100	0.99764	0.99783	0.99802	0.99820	0.99837	0.99854	0.99870	0.99885
110	0.99742	0.99763	0.99784	0.99803	0.99822	0.99841	0.99858	0.99875
120	0.99720	0.99743	0.99766	0.99787	0.99808	0.99828	0.99847	0.99866
130	0.99698	0.99724	0.99748	0.99771	0.99793	0.99815	0.99836	0.99856
140	0.99677	0.99704	0.99730	0.99755	0.99779	0.99803	0.99825	0.99847
150	0.99656	0.99685	0.99713	0.99740	0.99766	0.99790	0.99814	0.99837
160	0.99636	0.99667	0.99696	0.99725	0.99752	0.99779	0.99804	0.99829
170	0.99615	0.99648	0.99680	0.99710	0.99739	0.99767	0.99794	0.99820
180	0.99595	0.99630	0.99663	0.99695	0.99726	0.99756	0.99784	0.99812
190	0.99576	0.99612	0.99647	0.99681	0.99713	0.99744	0.99775	0.99804
200	0.99556	0.99595	0.99631	0.99667	0.99701	0.99734	0.99765	0.99796
210	0.99537	0.99577	0.99616	0.99653	0.99689	0.99723	0.99756	0.99788
220	0.99518	0.99560	0.99601	0.99639	0.99677	0.99713	0.99747	0.99781
230	0.99500	0.99544	0.99586	0.99626	0.99665	0.99703	0.99739	0.99774
240	0.99482	0.99527	0.99571	0.99613	0.99654	0.99693	0.99730	0.99767
250	0.99464	0.99511	0.99557	0.99600	0.99643	0.99683	0.99722	0.99760
260	0.99446	0.99495	0.99542	0.99588	0.99632	0.99674	0.99715	0.99754
300	0.99378	0.99435	0.99489	0.99541	0.99591	0.99639	0.99686	0.99731
400	0.99230	0.99304	0.99375	0.99443	0.99508	0.99571	0.99632	0.99691
500	0.99112	0.99202	0.99289	0.99372	0.99453	0.99530	0.99604	0.99675
600	0.99024	0.99130	0.99232	0.99330	0.99424	0.99514	0.99601	0.99685
700	0.98967	0.99088	0.99204	0.99315	0.99422	0.99525	0.99624	0.99720
800	0.98940	0.99074	0.99204	0.99328	0.99447	0.99562	0.99672	0.99779
900	0.98943	0.99091	0.99232	0.99368	0.99499	0.99625	0.99746	0.99862
1000	0.98976	0.99136	0.99289	0.99436	0.99577	0.99713	0.99844	0.99969
1100	0.99039	0.99209	0.99373	0.99530	0.99681	0.99826	0.99966	1.00100
1200	0.99131	0.99311	0.99484	0.99651	0.99811	0.99964	1.00112	1.00254
1300	0.99251	0.99441	0.99623	0.99797	0.99965	1.00126	1.00281	1.00430
1400	0.99399	0.99597	0.99787	0.99969	1.00144	1.00312	1.00474	1.00629
1500	0.99575	0.99780	0.99976	1.00165	1.00346	1.00521	1.00688	1.00849
1600	0.99777	0.99988	1.00191	1.00385	1.00572	1.00751	1.00924	1.01090
1700	1.00005	1.00221	1.00429	1.00628	1.00820	1.01004	1.01181	1.01351
1800	1.00258	1.00479	1.00690	1.00894	1.01089	1.01277	1.01458	1.01631
1900	1.00535	1.00759	1.00974	1.01181	1.01379	1.01570	1.01754	1.01930
2000	1.00834	1.01061	1.01279	1.01489	1.01690	1.01883	1.02069	1.02248
2100	1.01156	1.01385	1.01605	1.01816	1.02019	1.02214	1.02402	1.02582
2200	1.01499	1.01729	1.01950	1.02163	1.02367	1.02563	1.02752	1.02934
2300	1.01862	1.02093	1.02315	1.02528	1.02733	1.02930	1.03119	1.03301
2400	1.02244	1.02475	1.02697	1.02910	1.03115	1.03312	1.03501	1.03684
2500	1.02645	1.02875	1.03096	1.03309	1.03513	1.03710	1.03899	1.04081
2600	1.03063	1.03292	1.03512	1.03724	1.03927	1.04123	1.04311	1.04492
2700	1.03497	1.03725	1.03943	1.04153	1.04355	1.04550	1.04736	1.04916
2800	1.03947	1.04173	1.04389	1.04597	1.04797	1.04990	1.05175	1.05353
2900	1.04412	1.04635	1.07849	1.05054	1.05252	1.05443	1.05626	1.05802
3000	1.04891	1.05110	1.05321	1.05524	1.05720	1.05907	1.06088	1.06262

P,	Z-Factors for Air at Temperatures (Degrees C) of:							
psia	0	5	10	15	20	25	30	35
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	0.99965	0.99969	0.99973	0.99977	0.99980	0.99983	0.99986	0.99989
20	0.99930	0.99939	0.99946	0.99954	0.99960	0.99967	0.99973	0.99978
30	0.99896	0.99908	0.99920	0.99931	0.99941	0.99950	0.99959	0.99968
40	0.99862	0.99878	0.99894	0.99908	0.99922	0.99934	0.99946	0.99957
50	0.99828	0.99849	0.99868	0.99886	0.99903	0.99919	0.99933	0.99947
60	0.99795	0.99819	0.99843	0.99864	0.99884	0.99903	0.99921	0.99937
70	0.99761	0.99790	0.99817	0.99843	0.99866	0.99888	0.99909	0.99928
80	0.99729	0.99762	0.99792	0.99821	0.99848	0.99873	0.99896	0.99918
90	0.99696	0.99733	0.99768	0.99800	0.99830	0.99858	0.99885	0.99909
100	0.99664	0.99705	0.99744	0.99779	0.99813	0.99844	0.99873	0.99900
110	0.99632	0.99677	0.99719	0.99759	0.99795	0.99830	0.99862	0.99892
120	0.99601	0.99650	0.99696	0.99739	0.99779	0.99816	0.99851	0.99883
130	0.99569	0.99623	0.99672	0.99719	0.99762	0.99802	0.99840	0.99875
140	0.99538	0.99596	0.99649	0.99699	0.99745	0.99789	0.99829	0.99867
150	0.99508	0.99569	0.99626	0.99680	0.99729	0.99776	0.99819	0.99860
160	0.99478	0.99543	0.99604	0.99660	0.99713	0.99763	0.99809	0.99852
170	0.99448	0.99517	0.99582	0.99642	0.99698	0.99750	0.99799	0.99845
180	0.99418	0.99491	0.99560	0.99623	0.99683	0.99738	0.99790	0.99838
190	0.99389	0.99466	0.99538	0.99605	0.99667	0.99726	0.99780	0.99831
200	0.99360	0.99441	0.99517	0.99587	0.99653	0.99714	0.99771	0.99825
210	0.99331	0.99416	0.99496	0.99569	0.99638	0.99703	0.99763	0.99819
220	0.99303	0.99392	0.99475	0.99552	0.99624	0.99691	0.99754	0.99813
230	0.99275	0.99368	0.99454	0.99535	0.99610	0.99680	0.99746	0.99807
240	0.99247	0.99344	0.99434	0.99518	0.99596	0.99669	0.99738	0.99802
250	0.99220	0.99321	0.99414	0.99502	0.99583	0.99659	0.99730	0.99796
260	0.99193	0.99298	0.99395	0.99485	0.99570	0.99649	0.99722	0.99791
300	0.99089	0.99208	0.99320	0.99424	0.99520	0.99611	0.99695	0.99774
400	0.98851	0.99008	0.99154	0.99289	0.99416	0.99534	0.99644	0.99747
500	0.98647	0.98839	0.99018	0.99185	0.99339	0.99484	0.99618	0.99744
600	0.98478	0.98704	0.98914	0.99109	0.99291	0.99460	0.99618	0.99766
700	0.98343	0.98602	0.98841	0.99064	0.99271	0.99464	0.99644	0.99811
800	0.98244	0.98532	0.98800	0.99048	0.99279	0.99494	0.99694	0.99881
900	0.98180	0.98496	0.98789	0.99061	0.99315	0.99550	0.99769	0.99974
1000	0.98151	0.98493	0.98810	0.99104	0.99378	0.99632	0.99869	1.00090
1100	0.98156	0.98522	0.98861	0.99176	0.99468	0.99740	0.99993	1.00229
1200	0.98196	0.98583	0.98942	0.99276	0.99585	0.99873	1.00141	1.00390
1300	0.98270	0.98676	0.99053	0.99403	0.99728	1.00030	1.00312	1.00574
1400	0.98376	0.98800	0.99193	0.99558	0.99897	1.00212	1.00505	1.00778
1500	0.98515	0.98955	0.99362	0.99740	1.00091	1.00417	1.00721	1.01004
1600	0.98686	0.99138	0.99558	0.99947	1.00308	1.00645	1.00958	1.01249
1700	0.98886	0.99350	0.99780	1.00179	1.00550	1.00894	1.01215	1.01514
1800	0.99117	0.99589	1.00028	1.00435	1.00813	1.01165	1.01493	1.01798
1900	0.99375	0.99855	1.00301	1.00715	1.01099	1.01457	1.01790	1.02100
2000	0.99661	1.00147	1.00598	1.01017	1.01406	1.01768	1.02105	1.02419
2100	0.99973	1.00463	1.00918	1.01340	1.01733	1.02098	1.02439	1.02756
2200	1.00310	1.00802	1.01259	1.01684	1.02079	1.02447	1.02789	1.03108
2300	1.00670	1.01163	1.01622	1.02047	1.02444	1.02812	1.03156	1.03476
2400	1.01053	1.01546	1.02004	1.02430	1.02826	1.03195	1.03538	1.03859
2500	1.01458	1.01949	1.02405	1.02830	1.03225	1.03593	1.03936	1.04256
2600	1.01882	1.02371	1.02824	1.03247	1.03640	1.04006	1.04348	1.04666
2700	1.02326	1.02371	1.00824	1.03247	1.03640	1.04006	1.04348	1.04666
2800	1.02788	1.03267	1.03713	1.04128	1.04515	1.04875	1.05211	1.05524
2900	1.03267	1.03740	1.04181	1.04591	1.04973	1.05329	1.05661	1.05971
3000	1.03762	104228	1.04663	1.05067	1.05444	1.05796	1.06123	1.06429

Table 6-9—Gas Law Deviation Factors for Helium, Calculated from Beattie-Bridgemann Equation of State[14]

P,	Z-Factors for Air at Temperatures (Degrees F) of:								P,	Z-Factors for Air at Temperatures (Degrees C) of:							
psia	55	60	65	70	75	80	85	90	psia	0	5	10	15	20	25	30	35
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	1.00038	1.00038	1.00037	1.00037	1.00037	1.00036	1.00036	1.00036	10	1.00040	1.00039	1.00038	1.00038	1.00037	1.00036	1.00036	1.00035
20	1.00076	1.00075	1.00074	1.00074	1.00073	1.00073	1.00072	1.00071	20	1.00079	1.00078	1.00077	1.00075	1.00074	1.00073	1.00072	1.00071
30	1.00114	1.00113	1.00112	1.00111	1.00110	1.00109	1.00108	1.00107	30	1.00119	1.00117	1.00115	1.00113	1.00111	1.00109	1.00108	1.00106
40	1.00152	1.00150	1.00149	1.00148	1.00146	1.00145	1.00144	1.00143	40	1.00158	1.00155	1.00153	1.00150	1.00148	1.00146	1.00144	1.00141
50	1.00189	1.00188	1.00186	1.00184	1.00183	1.00181	1.00180	1.00178	50	1.00198	1.00194	1.00191	1.00188	1.00185	1.00182	1.00179	1.00177
60	1.00227	1.00225	1.00223	1.00221	1.00219	1.00217	1.00216	1.00214	60	1.00237	1.00233	1.00229	1.00226	1.00222	1.00219	1.00215	1.00212
70	1.00265	1.00263	1.00260	1.00258	1.00256	1.00254	1.00251	1.00249	70	1.00276	1.00272	1.00267	1.00263	1.00259	1.00255	1.00251	1.00247
80	1.00303	1.00303	1.00297	1.00295	1.00292	1.00290	1.00287	1.00285	80	1.00316	1.00311	1.00306	1.00301	1.00296	1.00291	1.00287	1.00282
90	1.00341	1.00338	1.00335	1.00332	1.00329	1.00326	1.00323	1.00320	90	1.00355	1.00349	1.00344	1.00338	1.00333	1.00328	1.00323	1.00318
100	1.00378	1.00375	1.00372	1.00368	1.00365	1.00362	1.00359	1.00356	100	1.00395	1.00388	1.00382	1.00376	1.00370	1.00364	1.00358	1.00353
110	1.00416	1.00412	1.00409	1.00405	1.00402	1.00398	1.00.95	1.00391	110	1.00434	1.00427	1.00420	1.00413	1.00406	1.00400	1.00394	1.00388
120	1.00454	1.00450	1.00446	1.00442	1.00438	1.00434	1.00430	1.00427	120	1.00473	1.00465	1.00458	1.00450	1.00443	1.00436	1.00430	1.00423
130	1.00491	1.00487	1.00483	1.00478	1.00474	1.00470	1.00466	1.00462	130	1.00513	1.00504	1.00496	1.00488	1.00480	1.00473	1.00465	1.00458
140	1.00529	1.00524	1.00520	1.00515	1.00511	1.00506	1.00502	1.00498	140	1.00552	1.00543	1.00534	1.00525	1.00517	1.00509	1.00501	1.00493
150	1.00567	1.00562	1.00557	1.00552	1.00547	1.00542	1.00538	1.00533	150	1.00591	1.00581	1.00572	1.00563	1.00554	1.00545	1.00537	1.00529
160	1.00604	1.00599	1.00594	1.00588	1.00583	1.00578	1.00573	1.00568	160	1.00630	1.00620	1.00610	1.00600	1.00591	1.00581	1.00572	1.00564
170	1.00642	1.00636	1.00631	1.00625	1.00620	1.00614	1.00609	1.00604	170	1.00670	1.00659	1.00648	1.00637	1.00627	1.00617	1.00608	1.00599
180	1.00680	1.00673	1.00668	1.00662	1.00656	1.00650	1.00645	1.00639	180	1.00709	1.00697	1.00686	1.00675	1.00664	1.00654	1.00644	1.00634
190	1.00717	1.00711	1.00704	1.00698	1.00692	1.00686	1.00680	1.00675	190	1.00748	1.00736	1.00724	1.00712	1.00701	1.00690	1.00679	1.00669
200	1.00755	1.00748	1.00741	1.00735	1.00728	1.00722	1.00716	1.00710	200	1.00787	1.00774	1.00762	1.00749	1.00737	1.00726	1.00715	1.00704
210	1.00792	1.00785	1.00778	1.00771	1.00765	1.00758	1.00752	1.00745	210	1.00826	1.00813	1.00799	1.00787	1.00774	1.00762	1.00750	1.00739
220	1.00830	1.00822	1.00815	1.00808	1.00801	1.00794	1.00787	1.00781	220	1.00866	1.00851	1.00837	1.00824	1.00811	1.00798	1.00786	1.00774
230	1.00867	1.00859	1.00852	1.00844	1.00837	1.00830	1.00823	1.00816	230	1.00905	1.00890	1.00875	1.00861	1.00847	1.00834	1.00821	1.00809
240	1.00905	1.00897	1.00889	1.00881	1.00873	1.00866	1.00858	1.00851	240	1.00944	1.00928	1.00913	1.00898	1.00884	1.00870	1.00857	1.00844
250	1.00942	1.00934	1.00925	1.00917	1.00909	1.00902	1.00894	1.00886	250	1.00983	1.00967	1.00951	1.00935	1.00921	1.00906	1.00892	1.00879
260	1.00980	1.00971	1.00962	1.00954	1.00946	1.00937	1.00929	1.00922	260	1.01022	1.01005	1.00988	1.00973	1.00957	1.00942	1.00928	1.00914
300	1.01129	1.01119	1.01109	1.01099	1.01090	1.01081	1.01071	1.01062	300	1.01178	1.01158	1.01139	1.01121	1.01103	1.01086	1.01069	1.01053
400	1.01502	1.01488	1.01475	1.01462	1.01450	1.01437	1.01425	1.01413	400	1.01567	1.01541	1.01515	1.01491	1.01468	1.01445	1.01423	1.01401
500	1.01873	1.01856	1.01840	1.01824	1.01808	1.01792	1.01777	1.01762	500	1.01953	1.01921	1.01890	1.01859	1.01830	1.01801	1.01774	1.01747
600	1.02242	1.02222	1.02202	1.02183	1.02164	1.02145	1.02127	1.02109	600	1.02338	1.02229	1.02262	1.02226	1.02191	1.02157	1.02124	1.02092
700	1.02609	1.02586	1.02563	1.02541	1.02519	1.02497	1.02476	1.02455	700	1.02721	1.02676	1.02633	1.02590	1.02550	1.02510	1.02472	1.02434
800	1.02974	1.02948	1.02922	1.02897	1.02872	1.02847	1.02823	1.02799	800	1.03102	1.03051	1.03001	1.02953	1.02907	1.02862	1.02818	1.02776
900	1.03338	1.03309	1.03280	1.03251	1.03223	1.03195	1.03168	1.03142	900	1.03482	1.03424	1.03368	1.03315	1.03262	1.03212	1.03163	1.03115
1000	1.03700	1.03668	1.03635	1.03604	1.03573	1.03542	1.03512	1.03483	1000	1.03859	1.03795	1.03734	1.03674	1.03616	1.03560	1.03506	1.03454
1100	1.04061	1.04025	1.03990	1.03955	1.03921	1.03887	1.03854	1.03822	1100	1.04235	1.04165	1.04097	1.04032	1.03969	1.03907	1.03848	1.03790
1200	1.04420	1.04381	1.04342	1.04305	1.04267	1.04231	1.04195	1.04160	1200	1.04609	1.04533	1.04460	1.04388	1.04320	1.04253	1.04188	1.04126
1300	1.04777	1.04735	1.04693	1.04653	1.04613	1.04573	1.04535	1.04497	1300	1.04981	1.04899	1.04820	1.04743	1.04669	1.04597	1.04527	1.04459
1400	1.05133	1.05087	1.05043	1.04999	1.04956	1.04914	1.04872	1.04832	1400	1.05352	1.05264	1.05179	1.05096	1.05016	1.04939	1.04864	1.04792
1500	1.05487	1.05438	1.05391	1.05344	1.05298	1.05253	1.05209	1.05165	1500	1.05721	1.05627	1.05536	1.05448	1.05363	1.05280	1.05200	1.05122
1600	1.05839	1.05788	1.05737	1.05688	1.05639	1.05591	1.05544	1.05498	1600	1.06089	1.05988	1.05892	1.05798	1.05707	1.05620	1.05534	1.05452
1700	1.06190	1.06136	1.06082	1.06030	1.05978	1.05927	1.05877	1.05828	1700	1.06454	1.06348	1.06246	1.06147	1.06051	1.05958	1.05867	1.05780
1800	1.06540	1.06482	1.06426	1.06370	1.06316	1.06262	1.06210	1.06158	1800	1.06819	1.06707	1.06599	1.06494	1.06392	1.06294	1.06199	1.06107
1900	1.06888	1.06827	1.06768	1.06710	1.06652	1.06596	1.06540	1.06486	1900	1.07181	1.07064	1.06950	1.06839	1.06733	1.06629	1.06529	1.06432
2000	1.07235	1.07171	1.07109	1.07047	1.06987	1.06928	1.06870	1.06813	2000	1.07543	1.07419	1.07299	1.07184	1.07072	1.06963	1.06858	1.06756
2100	1.07580	1.07513	1.07448	1.07384	1.07321	1.07259	1.07198	1.07138	2100	1.07902	1.07773	1.07648	1.07527	1.07409	1.07296	1.07186	1.07079
2200	1.07924	1.07854	1.07786	1.07719	1.07653	1.07588	1.07525	1.07462	2200	1.08260	1.08125	1.07994	1.07868	1.07746	1.07627	1.07512	1.07401
2300	1.08266	1.08194	1.08123	1.08053	1.07984	1.07917	1.07850	1.07785	2300	1.08617	1.08476	1.08340	1.08208	1.08080	1.07957	1.07837	1.07721
2400	1.08607	1.08532	1.08458	1.08385	1.08314	1.08243	1.08174	1.08107	2400	1.08972	1.08826	1.08684	1.08547	1.08414	1.08285	1.08161	1.08040
2500	1.08947	1.08869	1.08792	1.08716	1.08642	1.08569	1.08497	1.08427	2500	1.09326	1.09174	1.09027	1.08884	1.08746	1.08613	1.08483	1.08358
2600	1.09285	1.09204	1.09124	1.09046	1.08969	1.08893	1.08819	1.08746	2600	1.09679	1.09521	1.09368	1.09220	1.09077	1.08939	1.08804	1.08674
2700	1.09622	1.09538	1.09456	1.09375	1.09295	1.09217	1.09140	1.09064	2700	1.10030	1.09866	1.09708	1.09555	1.09407	1.09263	1.09124	1.08990
2800	1.09958	1.09871	1.09786	1.09702	1.09619	1.09538	1.09459	1.09381	2800	1.10379	1.10210	1.10047	1.09888	1.09735	1.09587	1.09443	1.09304
2900	1.10293	1.10203	1.10114	1.10028	1.09943	1.09859	1.09777	1.09696	2900	1.10728	1.10553	1.10384	1.10221	1.10062	1.09909	1.09761	1.09617
3000	1.10626	1.10533	1.10442	1.10353	1.10265	1.10179	1.10094	1.10010	3000	1.11075	1.10894	1.10720	1.10551	1.10388	1.10230	1.10077	1.09929

T = absolute temperature of the gas during the measurement of b, degrees K.

M = gas molecular weight.

c = dimensionless "acceptance coefficient," which is slightly less than 1.0, according to Klinkenberg.[3]

r = radius of randomly distributed (spatially) capillaries that approximate a porous medium in Klinkenberg's model, μm.

In Equation 12, the factor in brackets depends upon gas properties only, and the factor in parentheses is rock dependent. If we define the rock-dependent factor, r/c, as the rock's "effective gas slippage radius," r_b, and solve for it by rearranging Equation 12:

$$r_b = \frac{99.5\mu}{b}\sqrt{\frac{T}{M}}, \tag{13}$$

then this is the recommended gas slippage factor to be reported, which is independent of the gas used and temperature. It should be reported in addition to b, or instead of it.

Klinkenberg's randomly distributed capillary analogy relates r_b to permeability and porosity by:

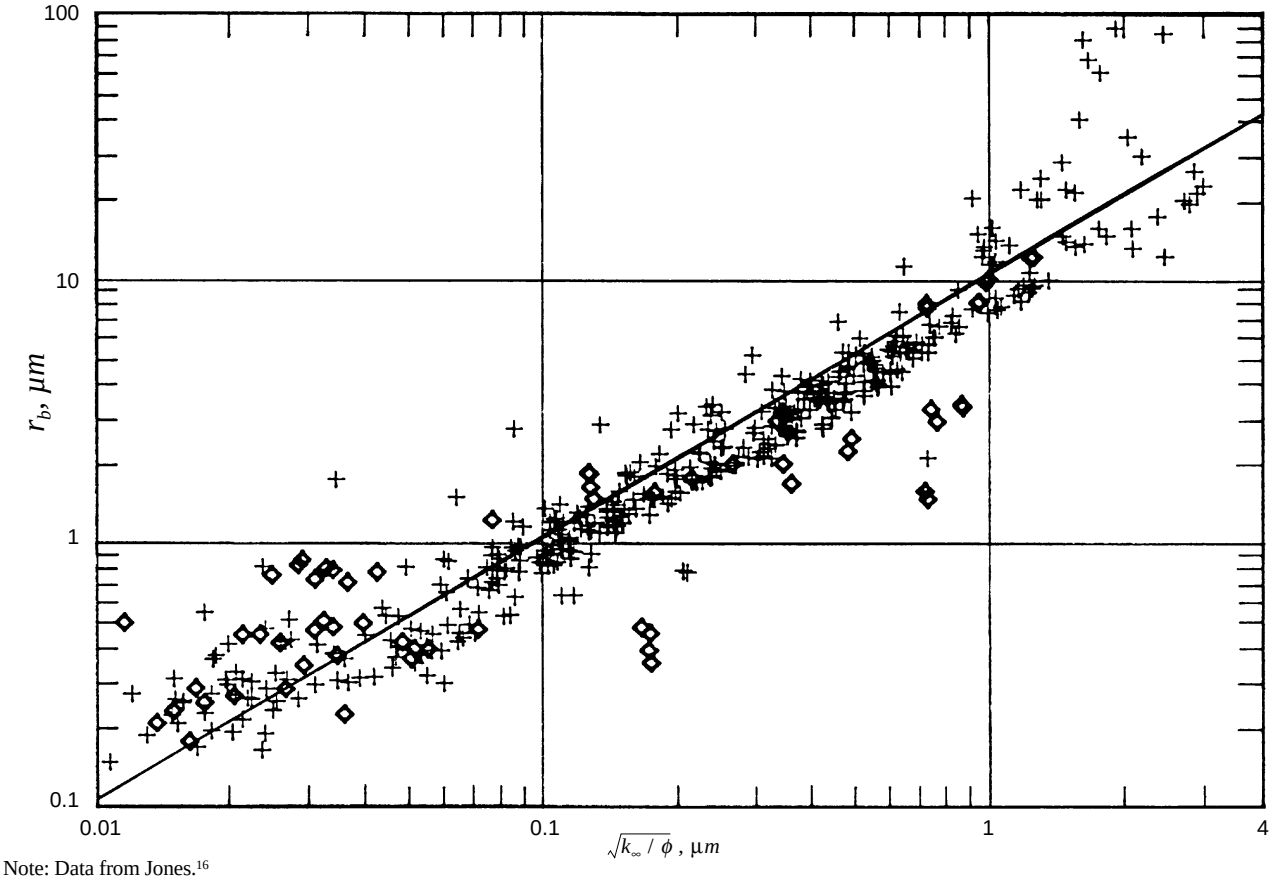
$$r_b = C_b\sqrt{\frac{k_\infty}{\phi}} \tag{14}$$

where k_∞ is Klinkenberg permeability, (μm^2), and ϕ is porosity (fraction). In Klinkenberg's model, C_b is equal to $\sqrt{24}/c$, or approximately 5. If we relax this "too simple" model, C_b can be found empirically. In Figure 6-2, r_b is plotted vs. $\sqrt{k_\infty/\phi}$. The slope of the line drawn in this log-log plot is unity. From its position, C_b is calculated to be approximately 11. The data, obtained using helium in several core plugs, show considerable scatter. Except for value of the constant, they qualitatively support Klinkenberg's model. The gas slippage parameters, b or r_b, are anticipated to be functions of pore structure, which may account for some of the scatter.

6.2.1.2 Steady-State Flow of Gas—The Forchheimer Equation

Steady state is achieved when the upstream and downstream pressures and flow rate all become invariant with time. At steady state, mass flow rate is constant throughout the sample and does not change with time. Therefore:

$$\rho q_s = \rho_r q_r \tag{15}$$



Note: Data from Jones.[16]

Figure 6-2—Rock-Dependent Gas Slippage Radius, r_b, as a Function of Permeability and Porosity

The subscript r refers to reference conditions of temperature and pressure under which the mass flow rate is measured. Thus at steady-state, isothermal conditions, where the reference temperature is the same as the flowing gas temperature:

$$\frac{Pq_s}{z} = \frac{P_r q_r}{z_r} \tag{16}$$

Substituting Equations 7, 9, and 16 into Equation 5 yields the differential form of the Forchheimer equation for isothermal, steady-state flow of gases, corrected for gas slippage:

$$C_1\left(\frac{-dP}{ds}\right) = \frac{C_2 \mu P_r q_r z}{A k_\infty z_t (P+b)} + \frac{C_3 \beta M P_r^{\,2} q_r^{\,2} z}{A^2 R T z_r^{\,2} P} \tag{17}$$

The integrated form of this equation can be used to obtain k_∞, b, and β. This requires a minimum of three, and preferably at least six measurements in which the upstream pressure (and/or optionally the downstream pressure) is varied widely to cause substantial variations in the mean pore pressure and flow velocity. For routine measurements, this is not practicable (cost-wise) under steady-state conditions. However, for most cases, adequate information for the calculation of these parameters can be obtained from a single pressure-falloff run (see 6.4.1.1 and 6.4.1.2).

6.2.1.3 The Slip-Corrected Darcy Equation

Whenever its last term becomes negligibly small, Equation 17 reduces to the differential form of Darcy's Law, corrected for gas slippage, and is the basis for most practical, routine measurements. Small pressure gradients lead to small flow velocities, that are squared in the last term, and are therefore small. The term's magnitude also depends on the values of β and gas density. Density is a function of the temperature, pressure, and molecular weight of the gas used.

Figure 6-3 shows typical values of β as a function of permeability for sandstone (open circles) and carbonate rocks (squares) (data from Jones[16]), and for homogeneous unconsolidated sands (solid circles, data from Geertsma[17]). The considerable scatter is believed to be related to heterogeneity in the rock.[16] Samples with fractures or thin layers of higher permeability material parallel to the flow direction have higher β's than homogeneous rock, due to locally high velocities in these high permeability "streaks." Figure 6-3 contains two dashed curves that encompass most of the points, and a central "best fit" curve.

Figure 6-4, which is based on the data in Figure 6-3, presents maximum allowable overall pressure gradients, $\Delta p/L$, that are sufficiently small to permit use of the slip-corrected Darcy equation for the calculation of permeability without incurring significant error due to inertial resistance. The curves shown are for air at 72°F, and for downstream pressures of 14.7, 50, 100, and 200 psia. Because of the scatter in β, these curves are approximate, and are to be interpreted as follows: If the actual β for a particular sample happens to lie on the upper dashed line in Figure 6-3, and the maximum $\Delta p/L$ as shown in Figure 6-4, for the conditions used, were applied for the permeability measurement, then the calculated permeability would be 5 percent low if the final term in Equation 17 were ignored. If the actual β were twice the value shown on the upper dashed line of Figure 6-3, and the same $\Delta p/L$ were applied, then the calculated permeability would be 10 percent too low. On the other hand, if the actual β were a factor of 10 smaller than the one indicated by the upper dashed line (thus, is closer to the high density of points), the permeability error would be only 0.5 percent, etc. Use of the maximum $\Delta p/L$ values shown in Figure 6-4 will result in errors (due to inertial resistance) of less than 2 percent for most samples when the slip-corrected Darcy equation is used for the calculation of permeability.

Starting with the highest permeabilities for the 14.7 psia downstream pressure in Figure 6-4, the maximum allowable pressure drop increases with decreasing permeability, as expected. However, for permeabilities below about 0.3 millidarcys, $\Delta p_{max}/L$ decreases with decreasing permeability. This unexpected result is a consequence of gas slippage. Slippage is greatest at the lowest permeabilities, causing higher gas flow rates than would occur without slippage.

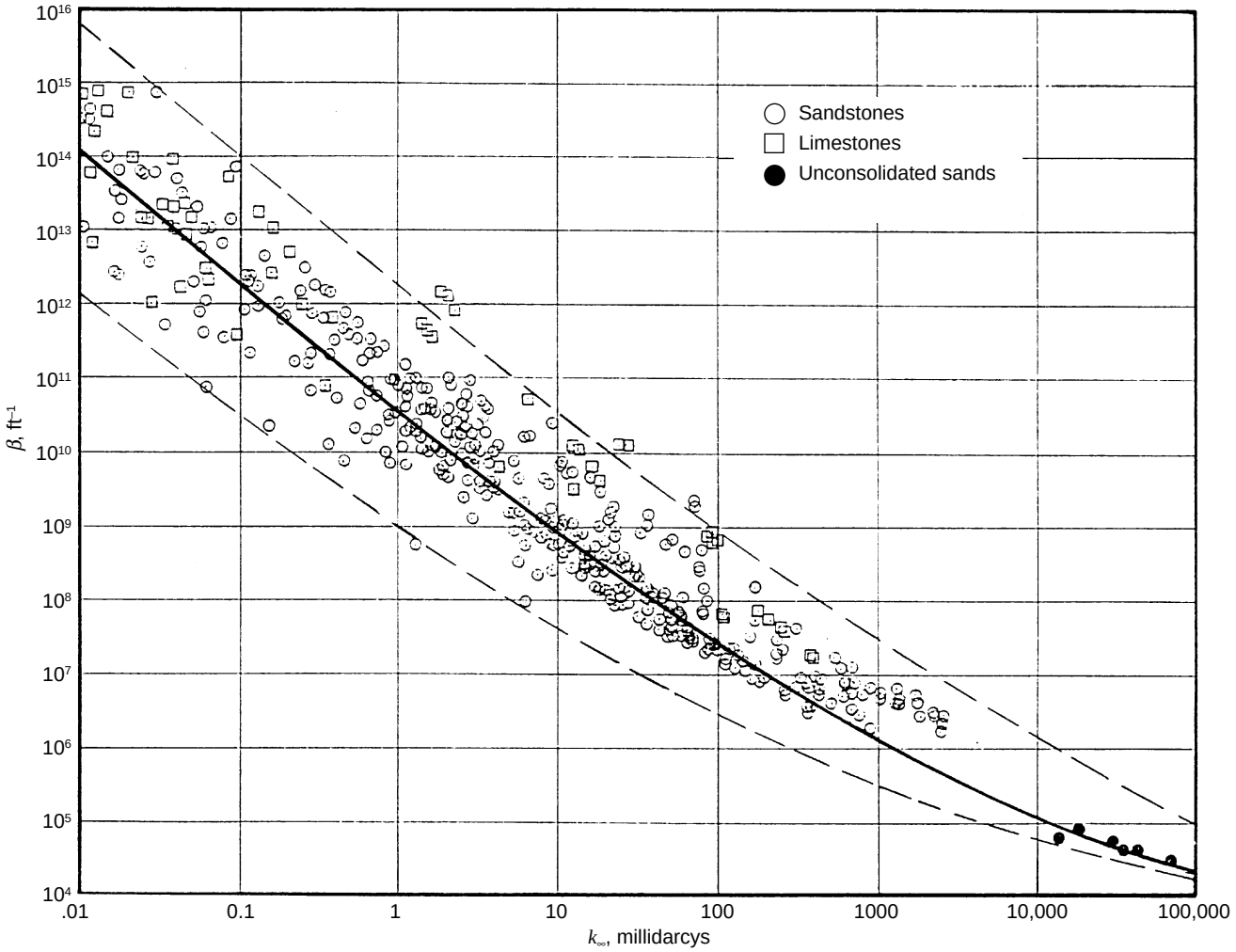
Except for very low permeabilities, the maximum allowable overall gradient is reduced as downstream pressure is increased. Increased back pressure increases gas density, thereby increasing the significance of the final term in Equation 17. This result should be acknowledged when making multiple measurements with increasingly high pore pressures for the Klinkenberg gas slippage correction. Apparent gas permeabilities measured at high pore pressures can be too low due to the inadvertent introduction of non-negligible inertial resistance.

Maximum $\Delta p/L$ values are gas specific. Values for nitrogen and air are similar, but those for helium are higher. Helium's viscosity is higher, and its molecular weight is less than that of air. Equations for calculating $\Delta p/L$ for other gases and liquids are developed in A.6.8.1.

The condition of Stokes flow is met when the last term of Equation 17 is negligible. The remaining slip-corrected Darcy equation can be integrated and used for calculation of Klinkenberg permeabilities for several frequently used flow configurations:

$$k_\infty = \frac{2 C_2 \mu P_r q_r z_m}{C_1 z_r G_f (P_1 - P_2)(P_1 + P_2 + 2b)} \tag{18}$$

Where C_1 and C_2 are conversion constants from Table 6-1 to allow for various sets of units for the variables. P_1 and P_2 are the injection and outflow absolute gas pressures, respectively.



Note: Data from Jones[16], and Geertsma[17].

Figure 6-3—Inertial Resistivity, β, as a Function of Permeability

To perform this integration, it was assumed that z in Equation 17 is replaceable by its mean value, z_m. Gas deviation factors z_m and z_r are calculated at the mean pore pressure, P_m (see Equation 20, below), and the absolute reference pressure, P_r, at which q_r is measured, respectively, and at the temperature of the gas. G_f is a geometric factor, which has the dimension of length. The particular factor for each of several flow configurations is given in sections following 6.3.1. The Klinkenberg gas slippage factor, b, must be determined from multiple measurements, or from a correlation. If it is obtained from a correlation, this fact shall be clearly stated.

To obtain b, from which r_b is calculated using Equation 13, the apparent gas permeability, k_g, is measured at a minimum of three (and preferably at least four) different mean pore pressures. Each permeability is calculated from:

$$k_g = \frac{2C_2\mu P_r q_r z_m}{C_1 z_r G_f (P_1 - P_2)(P_1 + P_2)} \tag{19}$$

and is plotted against the reciprocal of the mean pore pressure as illustrated by Figure 6-5. Mean pore pressure is defined as:

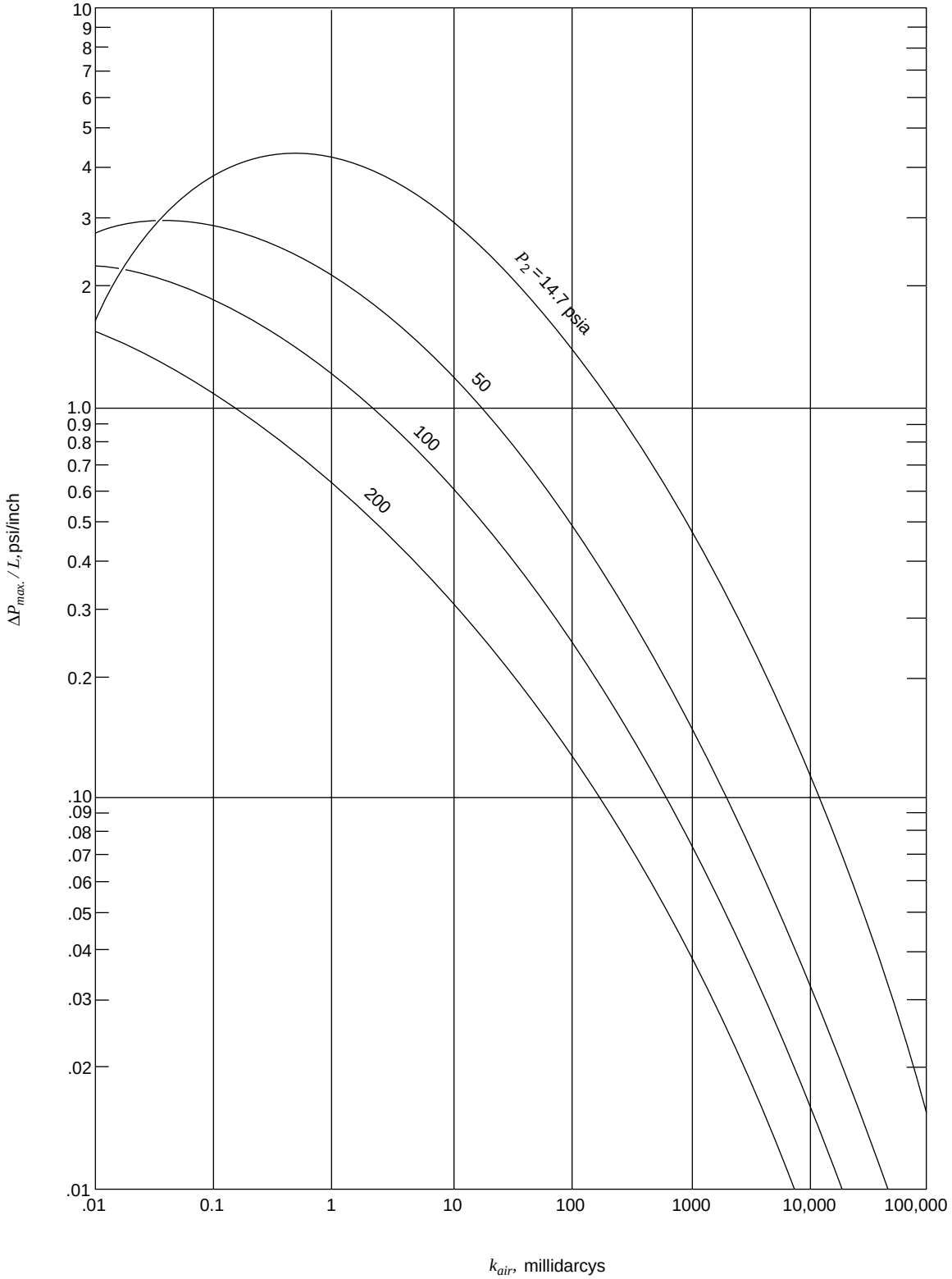
$$P_m = \tfrac{1}{2}(P_1 + P_2) \tag{20}$$

The intercept of such a plot is k_∞, and its slope is equal to bk_∞, in accordance with the Klinkenberg relationship:

$$k_g = k_\infty\left(1 + \frac{b}{P_m}\right) \tag{21}$$

The slope and intercept can be found by linear regression. It is essential to ensure that inertial resistance is negligible for each measurement. Otherwise, k_g will be low, especially at higher P_m values. Departure from linearity provides a self check on consistency.

Often, for routine permeability measurements, only gas permeabilities (uncorrected for gas slippage), as calculated from Equation 19, are reported. For these measurements, it is



Note: For air at 72°F.

Figure 6-4—Maximum Allowable Overall Pressure Gradients for Use of Slip-Corrected Darcy Equation

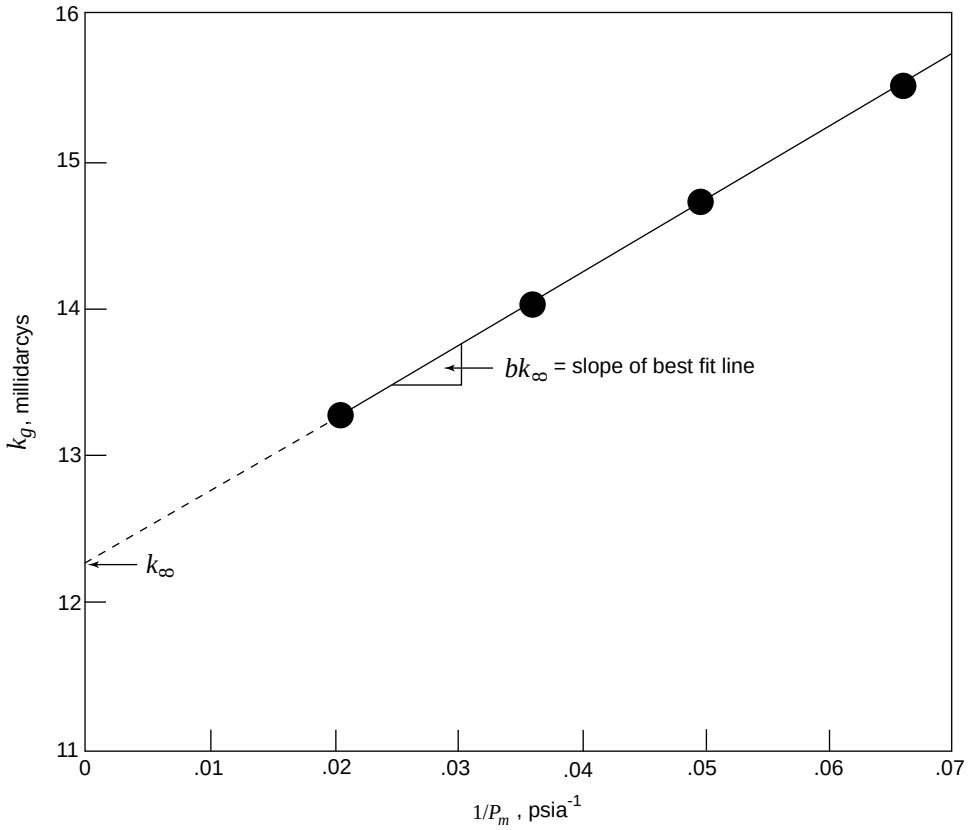


Figure 6-5—Type of Plot Used To Obtain Klinkenberg Gas Slippage Factor, *b*

essential to report the gas used and its mean pore pressure. Otherwise, the reported permeabilities are not fully specified.

6.2.2 Steady-State Permeability Equations For Liquids

Liquids have lower compressibilities, higher densities, and higher viscosities than gases. For steady-state measurements involving small pressure differences, liquids can be treated as incompressible fluids. Due to the higher liquid densities, gravity effects normally cannot be ignored in permeability measurements, except for horizontal flow. Departure from Darcy's Law is rarely a problem with liquids. Their higher viscosities result in significantly lower volumetric fluxes than observed for gases at the same pressure gradients. But their higher densities somewhat offset the viscosity effect with regard to inertial resistance. See A.6.8.1 for the calculation of maximum allowable $\Delta p/L$ values to be used for liquid permeability measurements when Darcy's Law is used.

The mean free path between flowing liquid molecules is so small that slippage is not observed. However, liquids—especially aqueous solutions—can react with constituents of a porous rock, altering its permeability. Also, care must be taken to avoid mobilizing fines in a rock at high liquid fluxes.

Darcy's Law, including gravity effects, for Stokes flow of liquids through permeable media may be stated in general terms as:

$$v_s = \frac{q}{A} = \frac{-C_1 k}{C_2 \mu}\left(\frac{dp}{ds} - \frac{pg}{C_4}\frac{dz}{ds}\right) \qquad (22)$$

Where:

s = distance along the direction of flow.

v_s = volumetric flux (volume of flow through a unit area of the porous medium per unit time).

z = vertical coordinate (increasing downward).

ρ = density of the liquid.

$\frac{dp}{ds}$ = pressure gradient along s at the point to which refers.

μ = viscosity of the liquid.

k = permeability of the medium.

q = volume rate of flow.

A = cross-sectional area perpendicular to lines of flow.

C_1, C_2, and C_4 are constants, found in Table 6-1, that make the units consistent.

6.2.2.1 Special Cases for Steady-State Flow of Incompressible Liquids

Equation 22 may be reduced to simpler forms under special conditions of flow. Some of the more important of these are as follows.

6.2.2.1.1 Horizontal Flow of Liquids

Because there is no vertical component of flow, $dz/ds = 0$, and integration of Equation 22 results in:

$$k = \frac{C_2 q \mu}{C_1 G_f (p_1 - p_2)} \tag{23}$$

Where G_f is a geometrical factor, which has the dimension of length. Factors for axial, transverse, or radial flow are given by Equations 27, 29, or 30, respectively, following 6.3.1.

6.2.2.1.2 Vertical Flow of Liquids

Two sub-cases arise, depending whether the flow is downward or upward, and dz/ds is equal to +1 or -1, respectively:

a. Downward flow with a driving head h (as defined by Figure 6-6):

$$k = \frac{C_2 C_4 q \mu L}{C_1 A \rho g (h + L)} \tag{24}$$

where L is the length of the sample and h is the height of the driving head. If the driving head, h, is zero, we have free downward flow, in which flow rate is independent of sample length.

b. Upward flow with driving head, h (as defined by Figure 6-7):

$$k = \frac{C_2 C_4 q \mu L}{C_1 A \rho g h} \tag{25}$$

If upward steady-state flow is achieved with an imposed pressure difference, Δp, which is measured with a differential pressure transducer with both ports at the same elevation (as shown in Figure 6-8) and both transducer lines completely filled with the same liquid as the flowing liquid, then:

$$k = \frac{C_2 q \mu L}{C_1 A \Delta p} \tag{26}$$

After steady-state is achieved (i.e., the Δp and flow rate are invariant with time), the inlet and outlet valves should be closed simultaneously, and the differential pressure transducer allowed to reach equilibrium. The equilibrium reading should be zero. If it is not zero, this reading should be subtracted from the steady-state reading.

6.2.3 Permeability Decrease At Elevated Net Stresses

The permeability of reservoir rock decreases with increasing net stress. Net stress for permeability is usually defined as the difference between confining stress and mean pore pressure. Figure 6-9 illustrates typical permeability reduction with increasing net hydrostatic stress. Percentage reductions are usually greater with lower permeability plugs. For example, zero-stress permeabilities of plugs 6-2, 8-2, and 11-2 in Figure 6-9 are about 700, 40, and 10 millidarcys, respectively. The permeability of a tight gas sand (e.g., 0.01 millidarcy or less at zero stress) can be reduced to 10 percent or less of its zero-stress permeability by high net compressive stresses.

6.3 PRACTICAL APPLICATIONS FOR STEADY-STATE PERMEABILITY MEASUREMENTS

Several practical configurations for routine, steady-state measurement of permeability are described, first for gases, then for liquids. Each may have particular advantages and limitations (e.g., speed; cost; accuracy; permeability range that can be accommodated; stress levels achievable; directional measurements; susceptibility to error; etc.) that are discussed. Certain precautions to avoid major pitfalls are presented.

Even though we ultimately calculate the permeability of a sample, which is a measure of its fluid conductivity, it is the conductance of the sample that dictates its fluid flow behavior. Conductance depends upon path length and area available for flow, as well as the permeability of the sample. Therefore, the measurable permeability range can be extended in some flow configurations by judicious choice of sample dimensions. For example, to maintain reasonable flow rates with axial flow when permeabilities are very high, long, small-diameter samples can be prepared. Conversely, for low permeability samples, short, larger-diameter plugs are desirable. Other conditions being equal, the maximum permeability that can be measured accurately with a three-inch-long plug is three times as large as can be measured on an otherwise identical one-inch-long plug.

Samples submitted for measurement often range in permeability from less than 0.001 to more than 30,000 millidarcys—a ratio of more than 3×10^7. To accommodate this variation, multiple ranges of pressure and flow rate measuring and/or controlling devices must either be insertable into a permeameter, or selectable from multiple devices that are built in. For simplicity, only a single set of devices is illustrated for each configuration listed below.

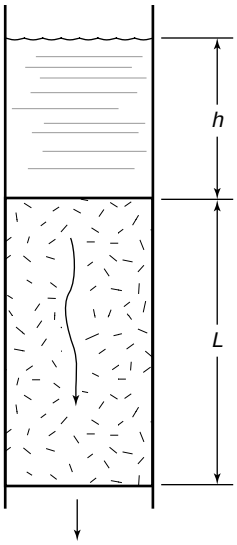


Figure 6-6—Downward Flow of Liquid with Driving Head, *h*

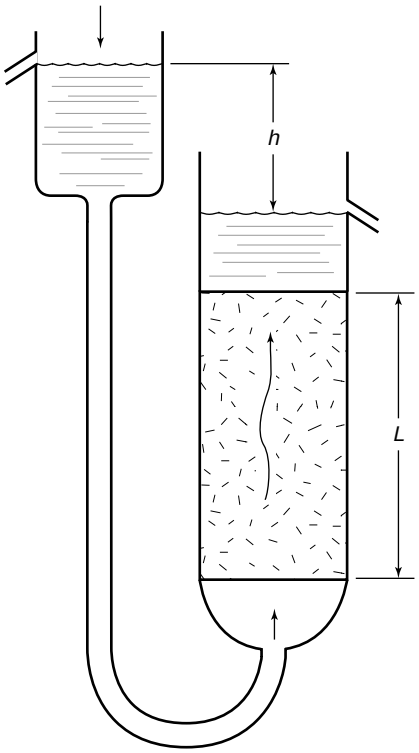


Figure 6-7—Upward Flow of Liquid with Driving Head, *h*

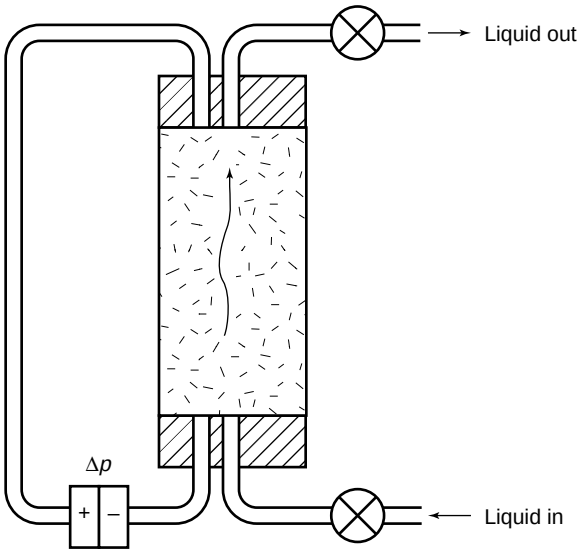
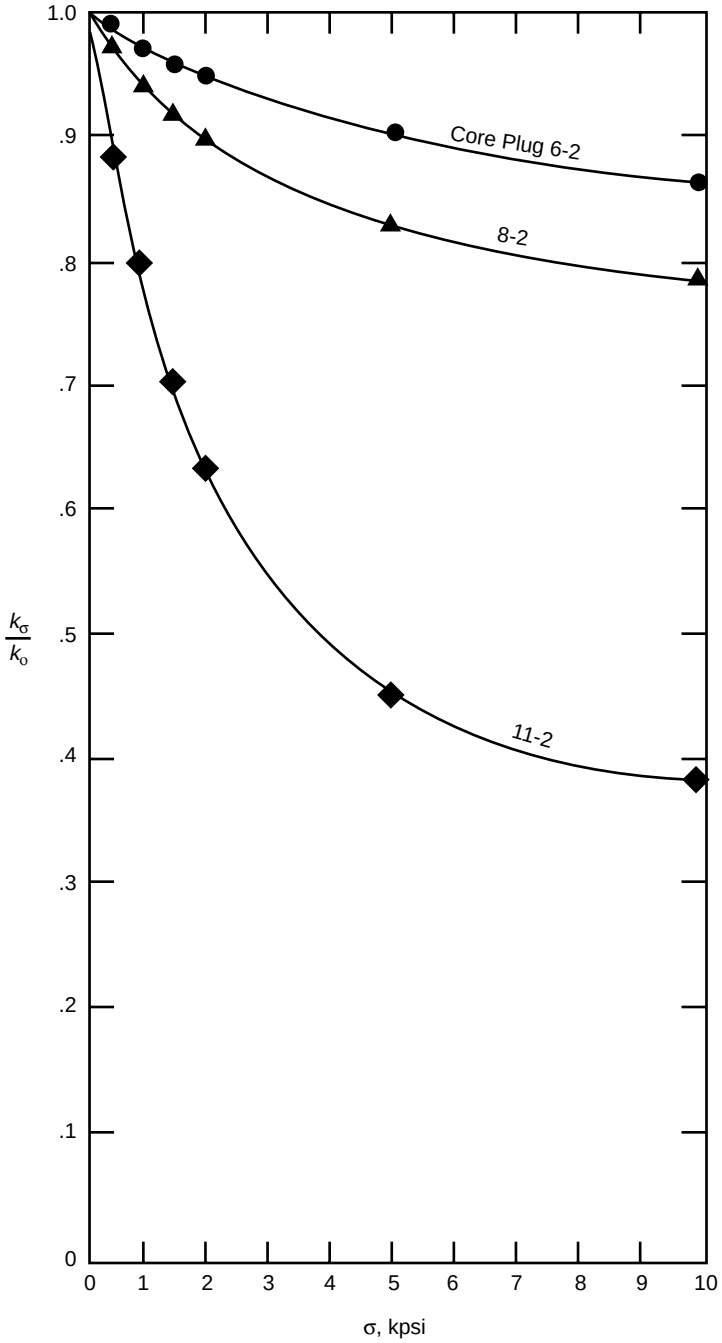


Figure 6-8—Upward Flow of Liquid with Imposed Pressure Difference, Δp



Note: Data from Jones[18].

Figure 6-9—Permeability Reduction with Increasing Net Hydrostatic Stress

6.3.1 Gases

6.3.1.1 Axial, Steady-State Flow of Gases

A schematic arrangement for axial flow of gases is shown in Figure 6-10. A cleaned and dried cylindrical core plug or whole core sample of length L and diameter D is mounted in a sample holder. The holder contains a flexible sleeve for purposes of making a gas-tight seal on the cylindrical walls of the sample, and for applying radial confining stresses. Axial stress can be transmitted to the sample by applying force to one or both end plugs, by mechanical, pneumatic, or hydraulic means. If the magnitudes of the radial and axial stresses are equal, the sample is said to be *isostatically* or *hydrostatically* stressed. If the magnitudes are unequal, the sample is biaxialy stressed. When gases at relatively low pressures (up to a few hundred psig) are used, gravity effects are negligible, and the sample holder may be oriented either horizontally or vertically.

The two end plugs are provided with an axial port for transporting gas to and from the sample. Each should have radial and circular grooves or other means for distributing gas to its entire injection face, and for collecting gas from all parts of its outflow face. Each end plug, preferably, also contains a second port for measuring upstream and downstream pressures, P_1 and P_2, respectively; the upstream pressure and differential pressure, Δp, as illustrated in Figure 6-10; or the differential and downstream pressures. However, lines for connecting the pressure-measuring devices can be teed into the flow lines near the axial ports, provided that the latter are sufficiently large not to cause significant pressure drops between each tee and the corresponding end face of the sample. It is essential that the branch of each tee (not the run) is connected to the pressure transducer. Otherwise, dynamic pressure effects may influence the pressure measurements.

Note that Δp, as used throughout this document is always a positive number, therefore is equal to $P_1–P_2$ or to $p_1–p_2$. As indicated previously, an upper case P denotes an absolute pressure, and a lower case p, a gauge or differential pressure.

The outflow line can be vented directly to atmospheric pressure (when the flow meter is upstream of the sample), connected to a flow rate measuring device, or to a back pressure regulator for purposes of creating elevated mean pore pressures. In the latter case, the flow meter can be either upstream of the sample or downstream of the back pressure regulator.

The volumetric flow rate, q_r, can be measured at the upstream or downstream pressure, or some other pressure, which in all cases is denoted as P_r, an absolute pressure. The temperature at which the flow rate is measured is assumed to be the same as the flowing gas temperature. Alternatively, the group $(q_r\ P_r)/(z_r\ T_r)$, which is proportional to mass flow rate, can be determined by a mass flow meter. See 6.6.2 for calibration of mass flow meters and application of results to the calculation of permeability.

6.3.1.1.1 Apparatus

6.3.1.1.1.1 Low Confining Stress

Figure 6-11 shows a simplified flow diagram for low pressure measurements. Figure 6-12 shows a low pressure Hassler-type core holder, in which a 400 psi radial confining stress and axial stress of unknown magnitude is typically applied to

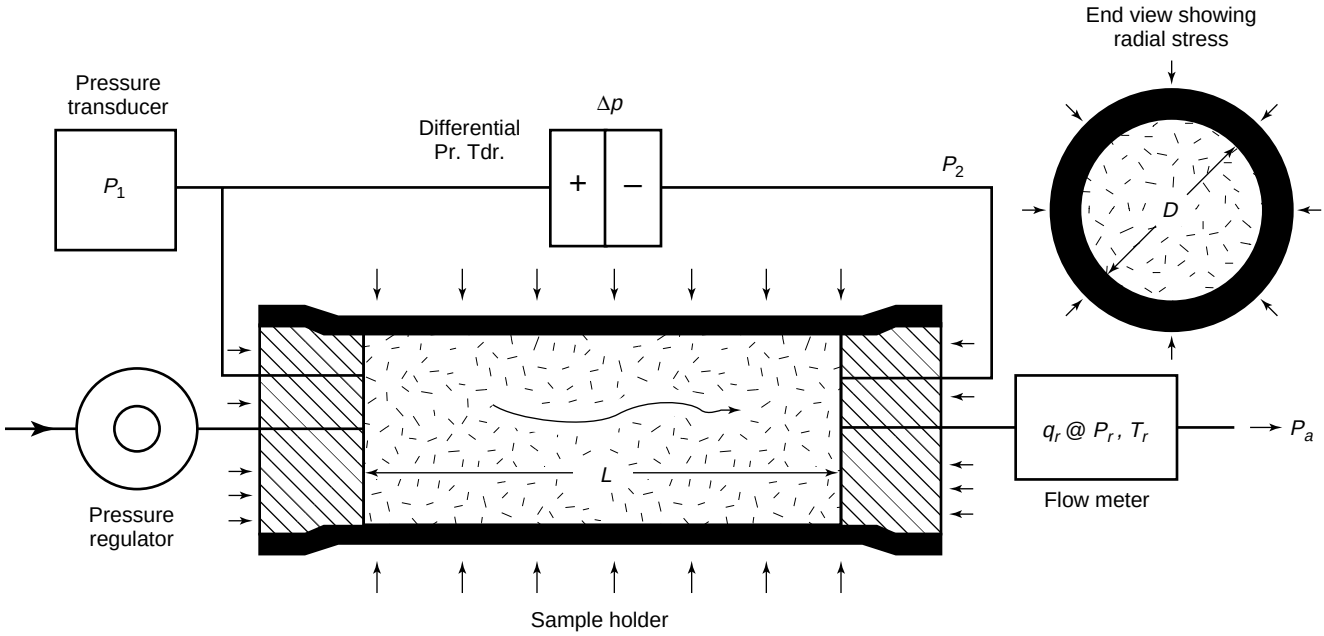


Figure 6-10—Schematic of Permeability Apparatus for Axial Flow of Gas

either a 1.0- or 1.5-inch (25- or 38-millimeter) diameter, 0.75- to 3-inch (19- to 76-millimeter) long core plug. These dimensions and stresses have become virtually standard in conventional, low stress core analysis. Radial stress is transmitted to the plug when pneumatic or hydraulic pressure is applied to the annular space between the rubber sleeve and core holder. Axial stress is typically transmitted from a screw that constrains the lower end plug. Normally, this stress is not measured, and is somewhat operator dependent.

Fancher-type core holders (see page 35 of API RP 40, 1960) are not recommended due to operator-dependent and non-reproducible sealing and confining stresses produced.

6.3.1.1.1.2 Elevated Confining Stress

A core holder for applying hydrostatic stresses, typically to a maximum of 10,000 psi, is illustrated in Figure 6-13. For safety, minimization of gas diffusion through the sleeve, and prevention of corrosion, hydraulic oil, or a heavy mineral oil is strongly recommended for stress generation. Axial stress in this core holder is reduced by the cross-sectional area of the tube connecting the internal end plug to the outer pressure vessel. For $^1/_8$-inch O.D. tubing, the reduction is not serious, but with larger diameters, the axial stress can be significantly less than the radial stress generated. For room temperature operation, 40 to 70 durometer (Shore A hardness), nitrile (Buna-N) rubber is recommended in sleeve thicknesses of 0.125 to 0.25 inch. The lower durometer and thickness can be used for lower stresses. Cold flow occurs with increasing stress and temperature. At stresses above about 6,500 psi, durometers higher than 70 are desirable for extended sleeve life.

Figure 6-14 illustrates a high pressure core holder that can be used to apply either hydrostatic or biaxial loading to core plugs. Axial stress is generated by a stress intensifier. Either pneumatic or hydraulic pressure bears on a piston of larger diameter than that of the end plug. The resulting force is transmitted by the lower end plug to the sample. The pressure required is inversely proportional to the ratio of areas. For example, if the larger piston has an area 10 times that of the end plug, a pressure of 600 psig applied to the large piston would transmit a stress of 6,000 psi to the core plug. If the axial stress is equal to the hydraulic pressure applied to the rubber sleeve, then the core plug is hydrostatically stressed. Otherwise, it is biaxialy stressed. In addition to its capability for providing unequal stresses, the main advantage of this type of core holder is that no disassembly is required for changing samples. Confining pressure is relieved (and often a vacuum is applied to expand the sleeve), the lower end plug is withdrawn, the previous sample is removed, and the new sample is inserted.

Strict safety precautions must be observed in both the design and operation of high pressure equipment. All parts that contain high pressures must suffer no permanent deformation at pressures at least equal to 1.5 times the maximum working pressure, or otherwise, must conform to local, national, or international codes that apply for high pressure vessels.

6.3.1.1.2 Calculations

Gas permeability, k_g, is calculated from Equation 19 (see 6.2.1.3). If multiple measurements are made at different mean pore pressures, k_∞ and b are obtained from a Klinkenberg plot

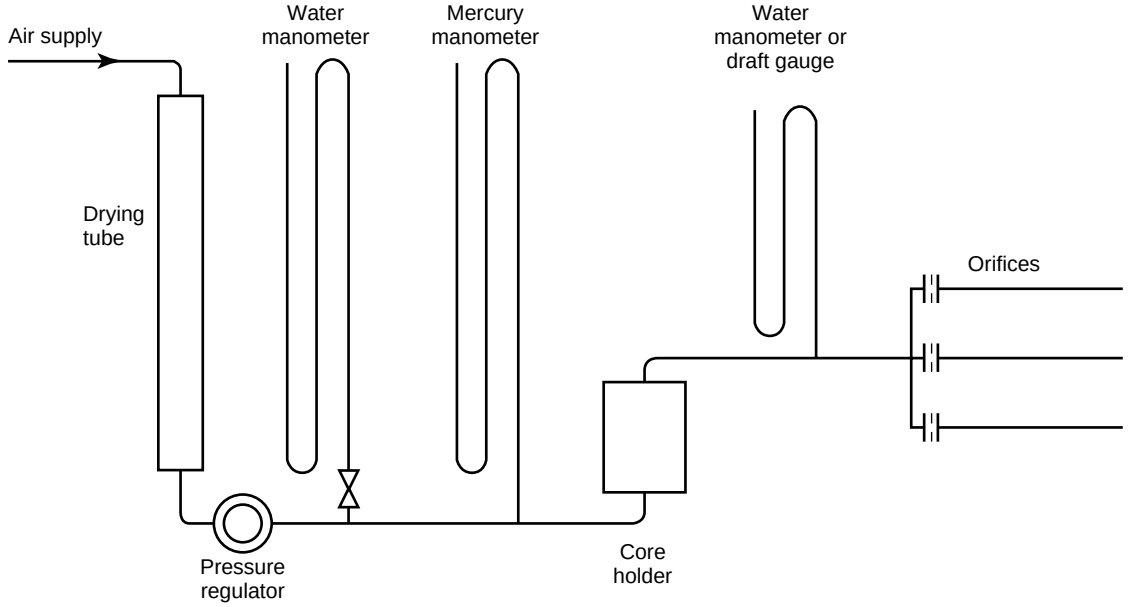


Figure 6-11—Simplified Flow Diagram for Low Pressure, Axial Gas Flow Permeability Measurements

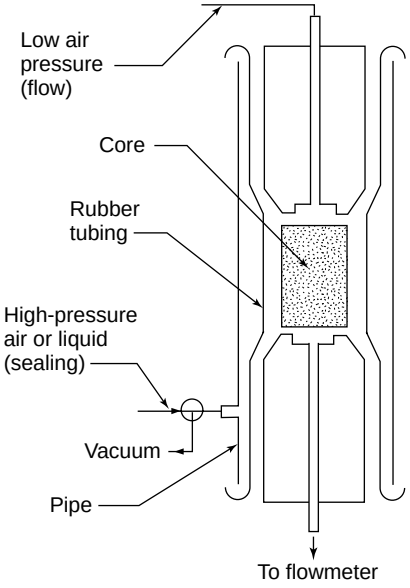


Figure 6-12—Low-Pressure Hassler-Type Core Holder

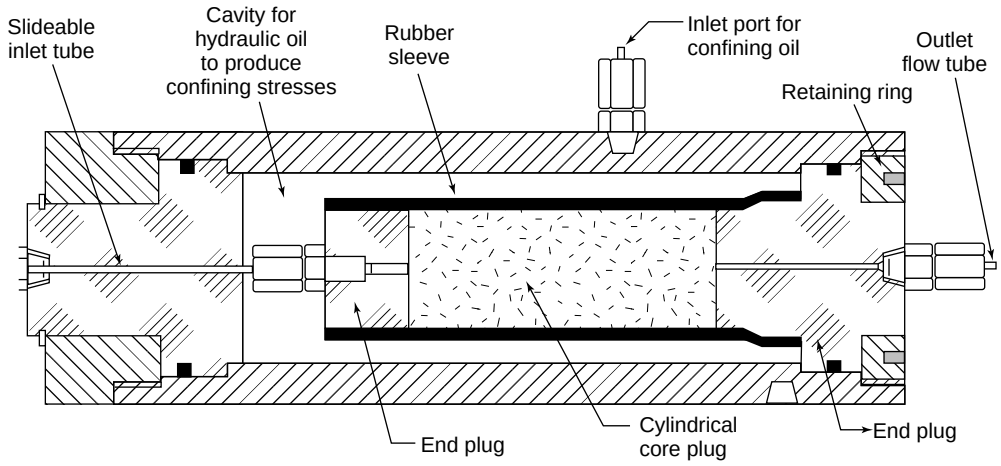


Figure 6-13—High-Pressure Core Holder for Hydrostatic Stresses

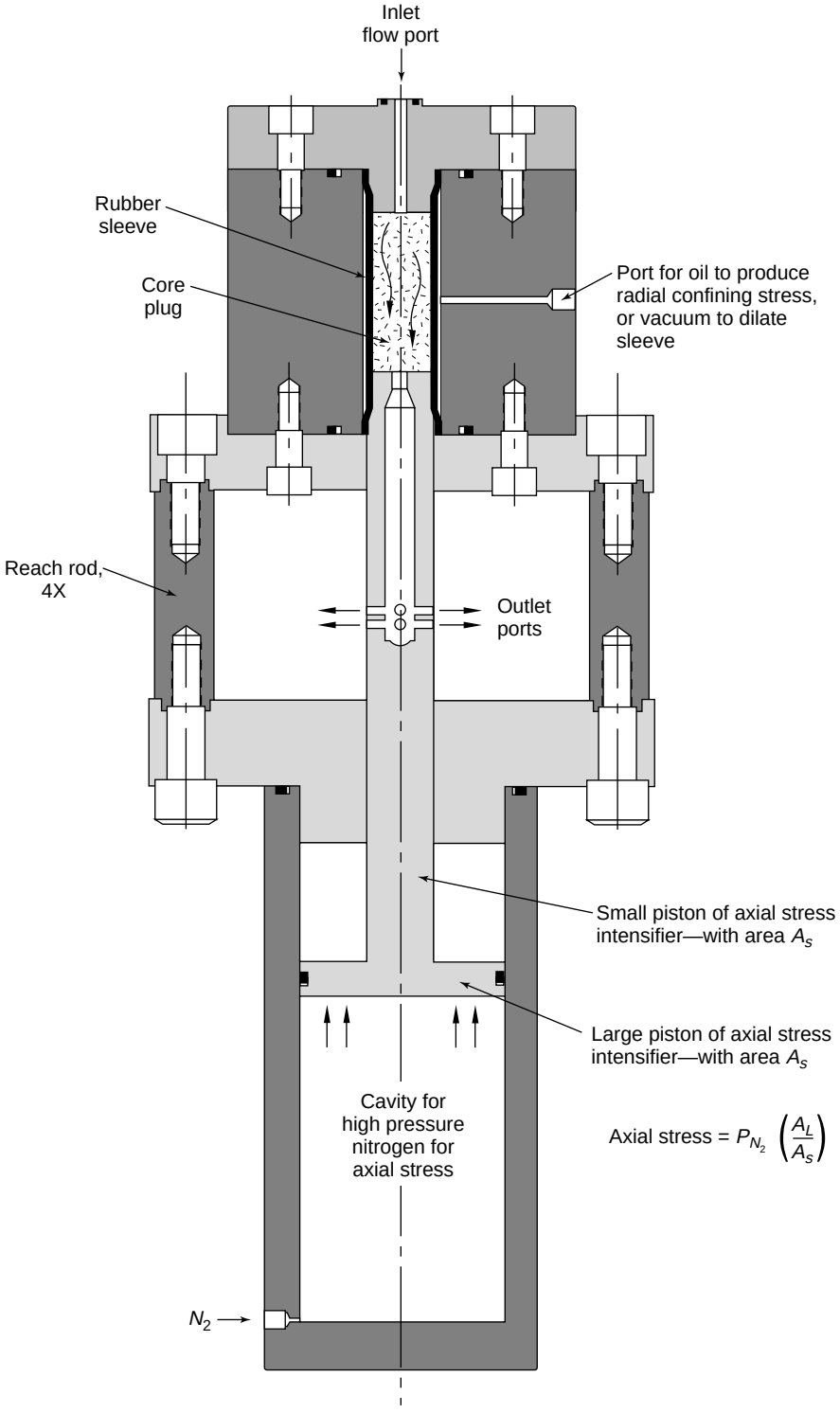


Figure 6-14—High-Pressure Core Holder For Hydrostatic or Biaxial Loading

(see Figure 6-5), or linear regression as described earlier. If b is found from a correlation, then k_∞ is calculated from Equation 18. These statements also apply to all other gas flow configurations listed below. The geometric factor for axial flow in Equations 18 and 19 is:

$$G_f = \frac{\pi D^2}{4L} \tag{27}$$

Traditionally, when a plug or whole core is cut so that its axis is parallel to bedding planes or permeability strike, the measured permeability is called its "horizontal" permeability, k_H. Similarly, a "vertical" permeability, k_V, is obtained when the sample's axis is perpendicular to bedding planes.

6.3.1.1.3 Advantages

Advantages of this method include:

a. The experimental method is simple and has been an industry standard for many years. Consequently, there is a large historic data base for direct comparison.
b. Gas does not react with rock, and samples are clean at the conclusion of measurements.
c. The technique lends itself to a wide range of equipment. With a low-pressure Hassler holder, manometers, orifice-type volumetric flow devices, and manual data acquisition, capital costs are low. However, an upgraded apparatus that includes a high pressure hydrostatic or biaxial core holder and automated control and data acquisition systems is strongly recommended.
d. Flow meters and pressure transducers can easily be exchanged to extend permeability range.

6.3.1.1.4 Precautions and Limitations

Precautions and limitations of this method include:

a. The lower practical permeability limit with this technique is about 0.1 millidarcy. In low permeability rocks the time required to achieve steady state becomes long, and flow measurements can become rather inaccurate.
b. The primary disadvantage of non-slip-corrected, low stress measurements (typically, 400 psi radial confining stress, and unknown axial stress) is that they over estimate in situ reservoir permeability, especially in tight samples. The Klinkenberg slip correction can be made from measurements at several mean pore pressures, but routine measurements are typically made at only one pressure, and this pressure is seldom reported. If this pressure is not reported, the permeability is difficult to normalize, and is incompletely specified. The gas used and the mean pore pressure must be reported. If a gas slippage correction is made from a correlation, this fact must be reported.
c. Attainment of Stokes flow, especially with high permeability samples, can be difficult to ascertain with single point measurements. Measurements should be checked per section 6.2.1.3 to make sure that the maximum allowable $\Delta p/L$ has not been exceeded.
d. It is critical to cut perfectly square ends on core plugs, especially with high confining stresses. A high point will receive a disproportionately high stress, which may crush this portion of the plug. This, of course, applies to all techniques involving cylindrical core plugs.

6.3.1.2 Steady-State Probe Permeability Measurements With Gases

When gas flows from the end of a small-diameter tube (or "probe") that is sealed against the surface of a slabbed or unslabbed whole-core sample, or a permeable rock outcrop, the flow pattern is somewhat similar to that of hemispherical flow (see Figure 6-15). This configuration is widely used in making inexpensive, non-destructive, zero-stress gas permeability measurements within small radii of influence. If the cores are not cleaned and dried, a measurement yields an effective permeability at some unknown oil and water saturation. Such measurements can be made at close spacing to determine permeability variation in a heterogeneous formation. Instruments for making these measurements are generically called "probe permeameters," or "mini-permeameters."

True hemispherical flow geometry would require: (a) a large, homogeneous, isotropic sample with a plane upper surface, (b) a hemispherical cavity under this surface, immediately under the probe, with a radius, r_i, equal to the inside radius of the probe seal, (c) a very large (infinite) extent of the sample in all directions under the plane of contact, and (d) a seal everywhere on this plane except over the hemispherical cavity. Because these conditions are totally impractical, Goggin, et al.[19] performed numerical calculations to produce dimensionless geometrical flow factors that account for the lack of a hemispherical cavity on the surface of a small, finite sample, and a probe seal of small, finite interior and exterior radii.

These dimensionless factors are displayed in Figure 6-16 as a function of the ratio of the outer radius, r_o, of the probe seal to its inner radius, r_i. For true, semi-infinite, hemispherical flow, G_0 would equal 2π. The relationship shown in Figure 6-16 is intended for samples with a plane upper surface and sufficient lateral extent and depth that exterior boundaries have no influence on permeability measurements. For practical purposes, the factors shown can be used with little error on samples that have a depth at least equal to four times the interior radius of the probe seal, and where the nearest lateral boundary is at least $4r_i$ from the axis of the probe. Also, little error is introduced if the probe is placed on a cylindrical surface of a sample, provided that a positive, gas-tight seal is made, the radius of the sample is at least $12r_i$, and the above sample boundary restrictions are observed. See Goggin, et al.[19] for dimensionless geometric factors that relate to nearer boundaries.

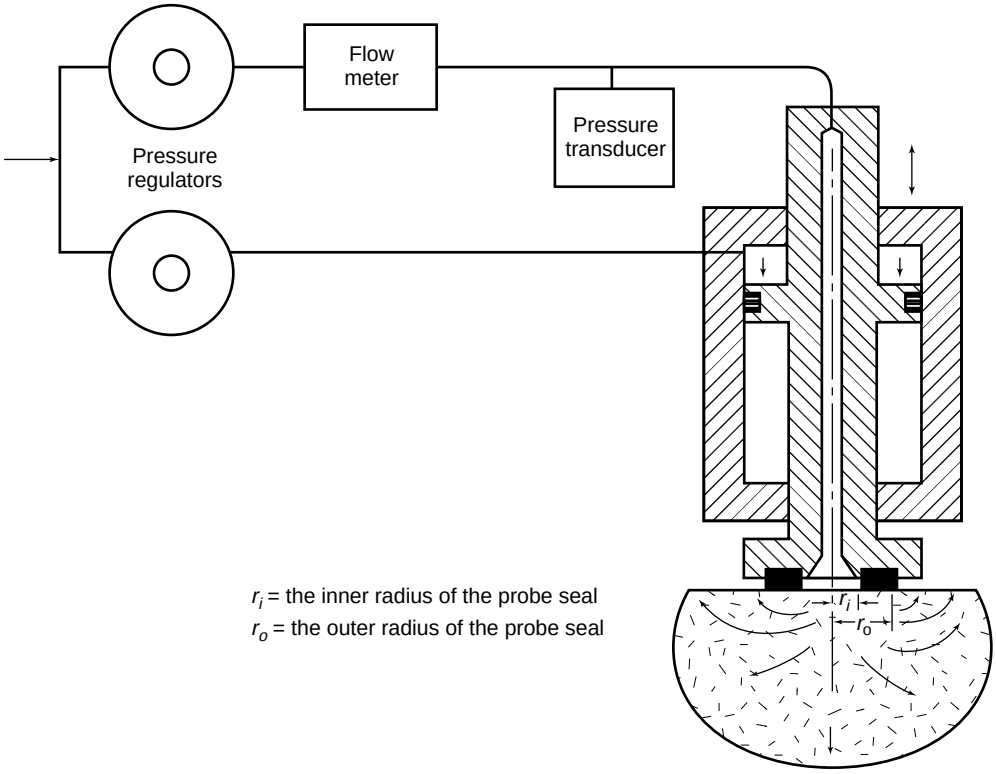
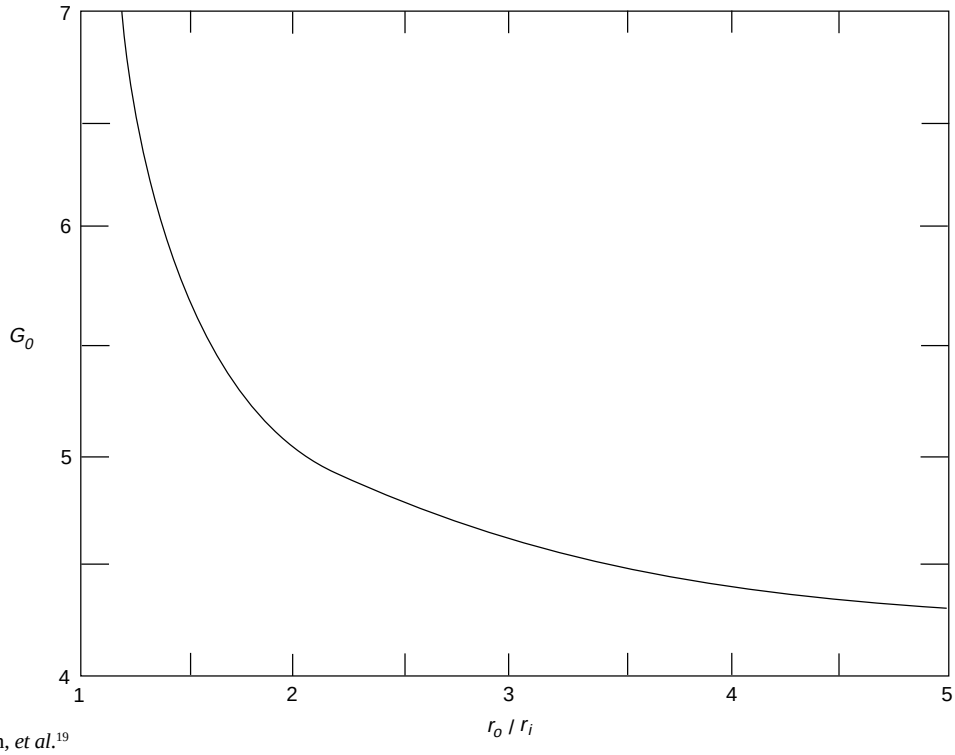


Figure 6-15—Schematic of Steady-State Probe Permeameter



Note: From Goggin, *et al.*[19]

Figure 6-16—Dimensionless Geometric Factor for Measurements with Probe Permeameter

6.3.1.2.1 Apparatus

For gas permeability measurements (see Figure 6-15), the probe must be provided with a soft rubber seal that has dimensionally stable (or reproducible) r_i and r_o when pressed against the sample, and means for delivering a constant, reproducible sealing force to it. This sealing force must be adequate to prevent gas from escaping anywhere on the contact surface between the seal and the rock. The inner radius of the seal is typically 2 to 3 millimeters, and the ratio of its outer to inner radii is typically 1.5 to 2.5. The smaller radii and ratios yield more resolution with regard to small scale heterogeneity, and larger values are used to obtain more permeability averaging. These dimensions must also be scaled to the coarseness of the rock texture.

The probe is positioned above a desired point on a sample, which must be related to depth and position relative to the core's axis or some other reference point. The probe is lowered to the rock's surface, and sealed against it. Gas at pressure P_1 is delivered from the probe to the sample. It exits from the sample at atmospheric pressure. Provision must be made for regulating pressure in the probe, and for measuring it and the volumetric gas flow rate at this pressure. Flow meter range is selected on the basis of the permeability being investigated. Pressure and flow rate are recorded when both become constant.

6.3.1.2.2 Calculations

From these measurements, plus the seal dimensions, atmospheric pressure, and gas viscosity, k_g can be calculated from Equation 19, wherein, if p_1 is the measured upstream gauge pressure:

$$P_1 = p_1 + P_a \quad ,$$

$$P_2 = P_a \quad ,$$

$$P_r = P_1 \quad ,$$

and

$$G_f = G_0 r_i \tag{28}$$

The permeability obtained is most heavily weighted (in non-homogeneous samples) within a radius equal to the inside seal radius, and progressively less with increasing radii. Because of the small volume of rock investigated, permeability on a macro scale must be obtained by appropriate averaging techniques.

6.3.1.2.3 Advantages

Advantages of this method include:

a. The method is nondestructive in the sense that there is no need to cut core plugs. It is rapid and low cost, thereby permitting high-density sampling.

b. The probe investigates only a small volume of rock. The measurement is well suited for investigation of spatial permeability variation in cores containing thin laminations and small scale heterogeneity. Directional permeability variation around the circumference of a whole core can be measured.

c. The device can be engineered for portable well site service, and for use on outcrops, provided that an adequate seal can be achieved.

d. With multiple flow meters a permeability range of about 1 to 10,000 millidarcys can be measured.

6.3.1.2.4 Precautions and Limitations

Precautions and limitations of this method include:

a. Minimum flow path lengths in a sample (from r_i to r_o) range from 0.2 to 0.4 cm for typical probe tip seals. Most of the total pressure drop occurs within this distance, which is an order of magnitude shorter than the path length for axial flow through most core plugs. The injection pressure, p_1, must be reduced correspondingly to avoid significant inertial flow resistance. Even though Goggin, et al.[19] proposed a "high velocity flow correction" using a correlation to obtain β, this correction can only be approximated with single-rate measurements, because β varies by plus or minus at least an order of magnitude at a given permeability for most permeability ranges. However, this correction is smaller the lower the injection pressure.

b. Nearly zero stress is applied to the rock during a measurement. Therefore, permeabilities tend to be very optimistic, especially in tight samples. If Klinkenberg corrections are not made (from a correlation), permeability is further overestimated, especially in low permeability rock.

c. Unless the tip seal is laterally confined, and essentially flat on the bottom (in the unstressed condition) its geometric factor can be very sensitive to the sealing stress applied. This stress, therefore, must be highly reproducible.

d. Although the technique can be used on whole core segments, slabbing is strongly recommended to remove mud solids invasion and other surface contamination, and to provide a flat, smooth surface for probe tip sealing, which otherwise is hard to guarantee.

e. If the surface of the core is not dry, measured permeability may be erroneously low due to severe relative permeability effects. Permeability may increase with time, then stabilize as mobile liquids are evaporated or blown away from the probe tip.

f. Probe tip seals should be inspected regularly for abrasion, deterioration, and imbedded sand grains, and replaced as needed. Periodic leak-testing on a smooth, flat impermeable surface is useful for detection of flaws or inclusions.

g. Due to the small volume of rock investigated by this technique, permeabilities on a macro scale must be obtained by appropriate averaging techniques.

6.3.1.3 Transverse, Steady-State Flow of Gases

This technique is used primarily to measure directional, "horizontal" permeability in (but not limited to) whole core samples. Gas flows from an inlet screen, covering a subtended angle θ and the entire length, L, of a whole-core sample (or core plug), across the sample, and into a similar outlet screen placed diametrically across from the inlet screen (see Figure 6-17). The flow pattern is complex, and the area normal to the streamlines for gas flow is variable throughout the flow path. Collins[20], using a conformal mapping transformation, computed a dimensionless geometrical factor, G_θ. It is shown in Figure 6-18, and is a function of the angle subtended by the screens. Permeability is normally measured in two directions—one giving the maximum value (usually along the direction of principal fracturing or permeability strike), and the other at 90 degrees to the maximum.

6.3.1.3.1 Apparatus and Calculations

The flow system and measurements required for calculation of transverse flow permeabilities are similar to those for steady-state axial flow. Figure 6-19 shows a Hassler-type core holder for transverse permeability measurements. Gas is allowed to enter and leave the sample through diametrically opposed openings through the sleeve. These connect to two mesh screens that cover the entire length of the sample and a known subtended angle on its circumference. If the subtended angle of each screen is 90 degrees, then the dimensionless geometrical factor, G_θ, is equal to 1.0. The seal materials must not penetrate the mesh and restrict flow. Both ends of the sample are sealed with soft rubber disks.

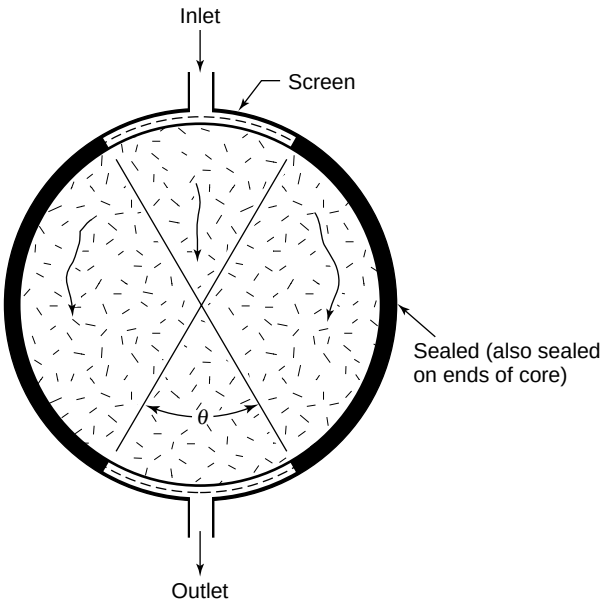
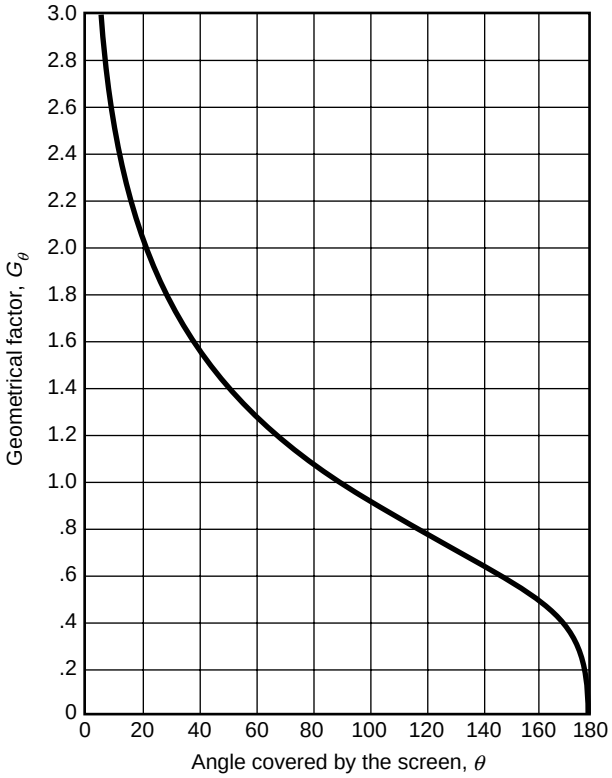


Figure 6-17—Transverse Flow Configuration for Gases or Liquids



Note: From Collins[20].

Figure 6-18—Dimensionless Geometric Factor for Transverse Flow

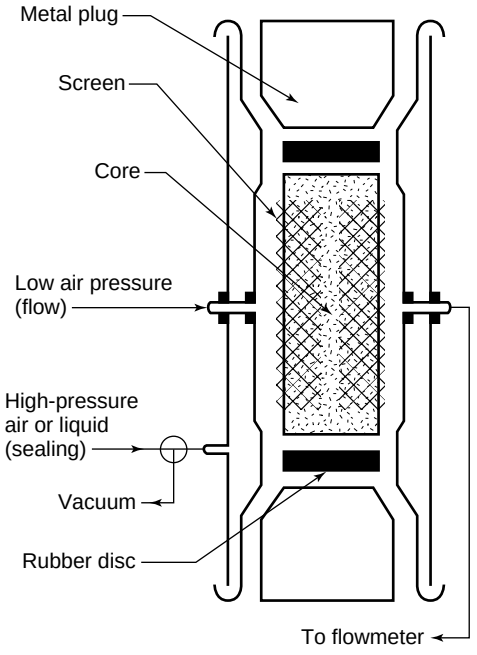


Figure 6-19—Hassler-Type Core Holder for Transverse Permeability Measurements

The dimensional geometric factor to be used in Equations 18 or 19 for transverse flow is:

$$G_f = \frac{L}{G_\theta} \tag{29}$$

A whole core sample can be placed in a large core holder for longitudinal "vertical" permeability measurements, before or after the transverse measurements. Calculations are those for axial flow (see 6.3.1.1.2).

6.3.1.3.2 Advantages

Advantages of this method include:

a. Measurements on whole core samples involve large volumes of rock, thereby permitting considerable permeability averaging. This minimizes the effect of small scale heterogeneity.

b. On oriented whole core samples, orthogonal measurements can be made to determine directional permeability.

6.3.1.3.3 Precautions and Limitations

Precautions and limitations of this method include:

a. A whole core sample is seldom cut precisely normal or parallel to the rock's bedding planes. Therefore conventionally defined "vertical" and "horizontal" permeabilities may not be obtained exactly with longitudinal and transverse measurements, respectively, on these samples.

b. Larger samples take considerably longer to clean, dry, and reach steady state. Experiments therefore take longer and are more expensive than those on core plugs.

c. The same precautions relating to gas slippage and inertial resistance, given in 6.3.1.1.4 for axial flow, should be observed.

6.3.1.4 Radial, Steady-State Flow of Gases

Figure 6-20 shows the flow configuration for steady-state radial flow. The upper and lower faces of a clean, dry cylindrical sample of length L and exterior radius r_e, into which an axial bore of radius r_w is cut, are sealed everywhere except over the central bore. Gas at pressure P_1 flows from the unsealed cylindrical surface radially to the central bore, and exits at pressure P_2. Otherwise, the flow system and measurements are the same as for axial flow. In principle, the flow direction can be reversed, so that gas is injected into the central bore at pressure P_1, and exits through the sample's exterior cylindrical surface at pressure P_2. However, injection into the well bore is generally not recommended because its smaller flow area is more prone to damage from trace amounts of particulates in the injected fluids.

6.3.1.4.1 Apparatus and Procedure

The full diameter radial permeameter, shown in Figure 21, consists of three parts: the cell, which is sufficiently large to maintain a uniform inlet pressure; a piston to apply the sealing force; and the floating plate assembly that consists of a lower fixed plate, a pivot ball, three springs 120 degrees apart, and the upper floating plate.

The core is placed on a 1-inch thick solid rubber gasket that is attached to the lower floating plate. The core is then raised against the closed lid, the center hole of the core matching that of the upper gasket. As the piston pressure increases, the lower floating plate automatically adjusts if the ends are not parallel. To check for an air leak between the ends of the core and the rubber gaskets, the piston pressure is increased. A decrease in the flow rate indicates that a leak had existed. This test is repeated until no change in flow rate is noted.

6.3.1.4.2 Calculations

Flow rates and pressure drops are measured and used in the same manner as for axial flow measurements. Steady state is reached when both become invariant with time. Permeability, k_g, is calculated from Equation 19, wherein the geometrical factor for radial flow is:

$$G_f = \frac{2\pi L}{\ln[r_e / r_w]} \tag{30}$$

Average permeability of non-homogeneous samples is most heavily weighted by the permeability at the inner wellbore radius, r_w (where the pressure gradient is greatest), and progressively less at larger radii.

6.3.1.4.3 Advantages

The measured permeability is averaged throughout the length of the sample and in all radial directions.

6.3.1.4.4 Precautions and Limitations

Precautions and limitations of this method include:

a. Permeability is critically dependent upon the internal wellbore radius. Small errors in its value, especially with small diameter holes, will cause large permeability errors. Similarly, induced fractures or surface damage near this hole will greatly affect apparent permeability.

b. Sample preparation for this measurement is not easy. Therefore the test tends to be somewhat more costly than alternative configurations.

c. Axial stresses are not balanced by radial stresses. The method is not easily adapted to application of elevated stresses for measurement of realistic in situ reservoir permeability.

d. A large piece of core material is altered by the central bore hole required for this technique, and the sample cannot usually be used for other tests.

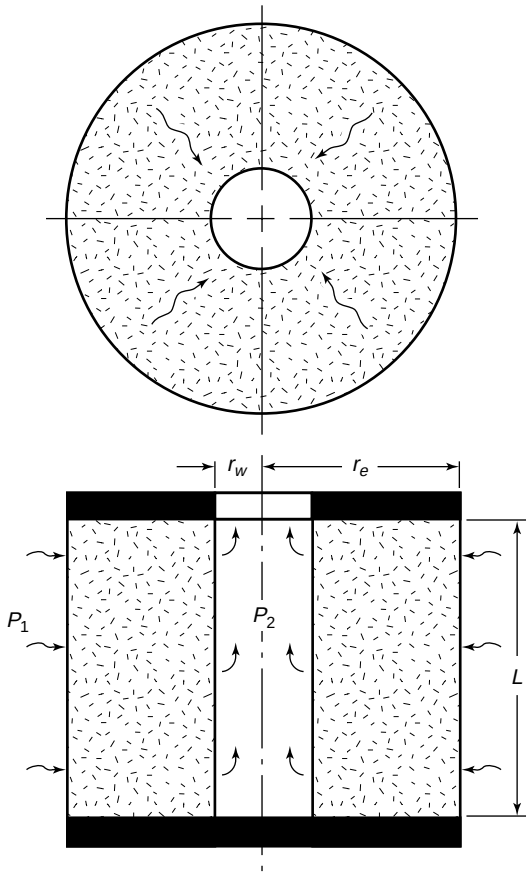


Figure 6-20—Schematic for Inward Radial Flow

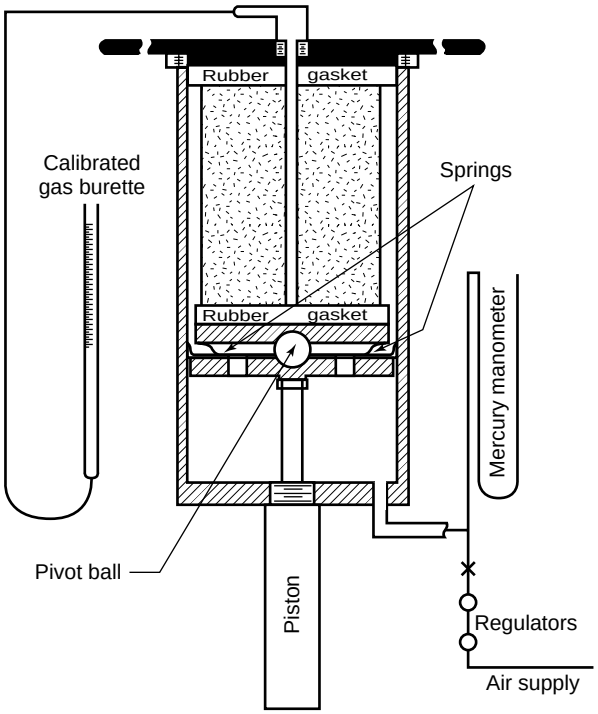


Figure 6-21—Full-Diameter Radial Flow Permeameter

6.3.2 Liquids

Steady-state liquid permeability apparatus have many features in common with those for gas. Differences between liquids and gases that may require equipment modifications are: liquid viscosity and density are considerably greater and compressibility is much smaller than the corresponding gas properties. Furthermore, liquids (especially aqueous solutions containing salts) can be corrosive, support microbial activity, and react with clay and mineral constituents of the rock.

Permeability to liquids is often observed to be lower than slip-corrected gas permeability. This can be due in part to liquid-clay interaction (especially with aqueous solutions), fines migration with high liquid flow rates, or incomplete liquid saturation.

6.3.2.1 Steady-State Liquid Permeability Measurements with an Imposed Δp

6.3.2.1.1 Apparatus

This type of permeability apparatus does not depend upon liquid heads for delivery of fluid. It comprises three separate units: sample holder, pressure measurement system, and fluid delivery system. Except for corrosion resistance and compatibility of the rubber sleeve with a particular liquid, the sample holder, delivery lines, and pressure measuring equipment for liquid permeability measurements are not different from systems for gases. The higher liquid density requires that attention be paid to liquid heads. These normally cancel out with horizontal flow, provided that both ports of the differential pressure transducer used are at the same elevation, and that all pressure-measuring lines are completely filled with the same fluid as the flowing fluid, and are at the same temperature. In this case, when flow through the sample is terminated, the Δp reading should go to zero. Under these circumstances Equation 23 (see 6.2.2.1.1), which applies to horizontal flow, also becomes valid for upward or downward vertical flow (see comments following Equation 26 in 6.2.2.1.2).

Because of the 50-fold (or more) higher viscosity of most liquids compared to gases, liquid flow rates are correspondingly lower at the same pressure gradient. The fluid delivery system is normally either a primary constant flow rate source or a constant pressure source that can be regulated to result in a constant flow rate. Single-stroke, positive displacement pumps offer the most accurate means of delivering a liquid at a controlled constant rate. The use of such pumps precludes the need to collect produced liquids from a core to determine

flow rate. However, the volume contained in a single-stroke pump may be insufficient to reach steady state. To completely eliminate pressure upsets and flow rate discontinuities, two such pumps and a sophisticated control system are recommended for a "once-through" flow system, and three are desirable for a recirculating flow system.

Slow attainment of steady state, provided that the sample's permeability is not changing due to progressive plugging, clay or mineral interaction, etc., is due to high compressive storage in the system. Compressive storage is the product of the volume and effective compressibility of each component in the system. Effective compressibility is the sum of fluid and vessel compressibilities. Fluid compressibility can be reduced markedly (if any gas is present) by applying a high back pressure to the system, thereby dissolving gas bubbles. Care should be taken to remove trapped gas from the system, and to de-gas liquids by applying a vacuum. Vessel compressibility is reduced by making vessels more rigid. System volumes should be minimized wherever possible. Use of transfer vessels (where, for example, oil from a pump displaces brine that is injected into a core plug) at least doubles the volume, compared to displacing brine directly from the pump. However, corrosion considerations may preclude direct pumping. Whenever compressive storage is nearly zero and permeability remains constant, achievement of steady state is virtually instantaneous.

When using a chromatography-type pump, the operator should ensure that its delivery is tested over the entire range to be employed. To calculate permeability, flow rates should be measured independently. This can be accomplished by collecting and weighing produced liquid in a weighing vessel on an electronic balance at frequent, timed intervals. Volumetric flow rate is calculated by dividing the rate of mass accumulation by the liquid's density.

Constant pressure systems utilize either a gas or liquid source to displace de-gassed liquid from a vessel and through the sample. The liquid to be displaced is contained in a piston accumulator or collapsible rubber bladder. The displacing fluid passes through a pressure regulator. As for the case of chromatography type pumps, flow rate must be determined independently.

6.3.2.1.2 Procedure and Calculations

The liquid to be used should be well-filtered through a fine filter shortly before use. A 0.2 micron filter will remove bacteria as well as solid particles. A fully saturated test sample should be carefully loaded into an appropriate sample holder, ensuring that no air is trapped in the process. Confining stresses are then applied. In the absence of any other permeability data, a low Δp or flow rate should be applied initially to allow the operator to make an initial approximation of permeability, and thereby set conditions of flow rate and pressure drop, and select appropriate equipment for these measurements. See A.6.8.1 to determine the maximum $\Delta p/L$ to avoid significant inertial resistance. However, this is generally not a serious problem with liquids. Care should be taken to avoid the mobilization of rock fines. The effluent should be collected and observed for contamination. Confining stresses should be adjusted to obtain the desired net stress.

Close temperature control must be maintained to ensure constant viscosity, and avoid volumetric flow rate changes with liquid expansion or contraction. This is particularly critical when large vessels are involved, accompanied by low displacement rates from a positive displacement pump, with low permeability plugs. Huge flow rate variations can occur in this situation with small changes in temperature over short time intervals. The critical factor is the thermally induced volumetric expansion rate, Δq:

$$\Delta q = \beta_T V\left(\frac{dT}{dt}\right)$$

relative to the pump's displacement rate.

Where:

β_T = coefficient of volumetric thermal expansion of liquid, $°C^{-1}$ or $°F^{-1}$.

V = volume of liquid being displaced.

$\frac{dT}{dt}$ = time rate of temperature change.

To illustrate, suppose that a positive displacement pump contains 1,000 cm^3 of decane that is being injected into a low permeability plug at a nominal pump displacement rate of 1.000 cm^3/hr., and that the temperature of the decane is increasing at a rate of 0.02°F/minute, due to changes in the ambient temperature. The coefficient of thermal expansion of decane is about 5.5×10^{-4} volumes per volume per degree F. Therefore the rate of thermal expansion of decane is $5.5 \times 10^{-4} \times 1000 \times 0.02 = 0.011$ cm^3/min. With the pump's displacement rate of 0.0166 cm^3/min, this causes an error in flow rate of 66 percent of the nominal rate! If the displacement rate were increased by a factor of 10, the error would be reduced to 6.6 percent. Additionally, if the pump's volume were reduced to 10 cm^3, the error would be only 0.066 percent. Another way to reduce volumetric expansion is to increase the thermal mass of the pump, and improve its insulation.

With a constant-pressure delivery system (in which flow rate must be determined independently), thermal expansion of liquid in its displacement vessel is not a problem, provided that the upstream pressure regulator is self-relieving. In this case pressure will remain constant in spite of thermal expansion.

After both flow rate and differential pressure have stabilized they should be read and recorded, then the flow terminated. The transducer lines must be completely filled with the

test liquid, and both ports must be at the same elevation. After the differential pressure stabilizes with zero flow, it is read. If it is not zero, the reading should be subtracted from the stabilized, flowing Δp. Liquid permeability is calculated from Equation 23, using the appropriate dimensional geometric factor, G_f. These factors for axial flow, transverse flow, and radial flow are given by Equations 27, 29, and 30, respectively. With the above transducer line restrictions, Equation 23 applies to vertical, upward or downward axial flow as well as to horizontal flow for all three geometric configurations.

After the measurement has been completed and the sample is to be removed for further analysis, care should be taken to ensure that, as the sample dilates while confining stress is reduced, liquid is imbibed and the ingress of air is precluded.

6.3.2.1.3 Advantages

Advantages of this method include:

a. Pressure differentials for Stokes flow with liquids are higher and easier to measure than those with gases, especially for high permeability samples.
b. No gas slippage correction is required.
c. Carefully made liquid permeability measurements may be more representative of reservoir permeabilities.
d. Sample drying, a potentially damaging operation, is not required.
e. Liquid permeability measurements may be advantageous if additional analyses require the plug to be saturated with the same liquid.

6.3.2.1.4 Precautions and Limitations

Precautions and limitations of this method include:

a. Saturation and preparation of the rock sample, and preparation and handling of liquids is generally more difficult and time consuming than preparations for gas measurements.
b. Corrosion resistant, high pressure pumping and control equipment is expensive.
c. Care must be taken to avoid fluids that interact with rock constituents.
d. Steady state liquid measurements with low permeability plugs may be difficult to make accurately, and require long periods to reach steady state. Small volumes, short plug lengths, low compressive storages, and very tight temperature control are strongly recommended.

6.3.2.2 Steady-State Liquid Permeability Measurements Using Liquid Heads

Figures 6-6 and 6-7 (Section 6.2.2.1.2) show two configurations for axial flow measurements in which the liquid is delivered at a constant rate and pressure by means of constant liquid heads. The configuration of Figure 6-7 is particularly useful for accurate, low cost measurements with moderate to high permeability core plugs. No pump or pressure transducer is required. The adjustable-height inlet reservoir and fixed outlet reservoir are both provided with overflow drains. Liquid is allowed to flow into the inlet reservoir from another, higher-elevation supply vessel through a metering valve at a slightly higher rate than the rock sample will accept. Excess liquid flows out the drain vent and is collected for future recycle to the supply vessel. Liquid overflowing from the outlet reservoir drops into a weighing vessel on an electronic balance. Volumetric flow rate, q, is calculated by dividing the rate of mass accumulation (from weight and elapsed time measurements) by the density of the liquid, which must be determined. Permeability is calculated from Equation 25, where ρ is the density of the liquid at the measurement temperature, g is the local value of gravitational acceleration, and h is the elevation difference between the free surfaces of the two liquid heads. Because very little back pressure is exerted, extreme care must be taken to exclude air from all portions of the system, including the core plug.

For very high permeability samples (10 to 40 darcys), the configuration of Figure 6-7 is useful in that the overflow vent in the downstream vessel minimizes variation in the liquid head due to capillary retentive forces. These forces can be significant in the tip of small diameter exit nozzles or ports, creating a large relative variation in the Δp.

6.4 THEORY AND APPLICATION OF UNSTEADY-STATE PERMEABILITY DETERMINATIONS

The advent of high speed data-acquisition systems, accurate pressure transducers, and digital computers has made it not only feasible, but convenient to measure permeabilities under transient, or unsteady-state flow conditions. Transient measurements employ fixed-volume reservoirs for gas or liquid. These may be located either upstream of the sample—from which the gas or liquid flows into the sample being measured, or downstream—into which it flows from the sample, or in both places. When fluid flows from an upstream reservoir, its pressure in that reservoir declines with time. Similarly, when fluid flows into a downstream reservoir, pressure there builds with time. Instantaneous flow rates can be calculated from the volume of the reservoir and the instantaneous rate of pressure change, obviating the need for a flow-rate measuring device.

When fluid expands, it performs flow work at the expense of loss of internal energy. This is observed as a decrease in its temperature. Similarly, when fluid is compressed, work is done on it, and its temperature increases. Because instantaneous flow rates are calculated from rates of pressure change, it is mandatory to maintain isothermal conditions, or to measure instantaneous temperatures and use appropriate mathematical formulations. Equations presented herein assume isothermal conditions.

6.4.1 Transient Pressure Techniques For Gases

Fortunately, gas has low heat capacity, and work-related temperature changes can be virtually eliminated by fabricating reservoirs from material of high thermal conductivity and packing them with copper tubes parallel to the flow axis. Also, where high flow rates are anticipated (with high permeability samples), helium is the gas of choice. Its thermal diffusivity is much higher than that of air or nitrogen.

Other potential thermal problems can arise from changes in ambient temperature in long-duration measurements of low permeability samples, and from Joule-Thomson expansion as gas flows through a sample. The first is minimized with excellent thermal insulation, improved ambient temperature control, and by reducing measurement time. Joule-Thomson expansion is best handled by using helium, which increases in temperature (at ambient conditions) upon expansion, unlike all other gases except hydrogen. This warming tends to counteract work-related cooling.

Two main categories of transient pressure techniques have emerged for measuring permeability in the laboratory. One is called the "pulse-decay" method. It is characterized by using both upstream and downstream reservoirs, one or both of which are relatively small in volume. These and the sample are filled with gas to a fairly high pressure, 1,000 to 2,000 psig, which reduces gas slippage and compressibility. After pressure equilibrium is achieved throughout the system, pressure in the upstream reservoir is increased, typically by 2 to 3 percent of the initial pressure, causing a pressure pulse to flow through the sample. This technique is well suited for low permeability samples, 0.1 millidarcy to about 0.01 microdarcy. Small differential pressures and low permeabilities virtually eliminate inertial flow resistance. Only "late time" techniques will be discussed in this document. They yield overall permeabilities that are comparable to steady-state values. "Early time" transients provide information regarding heterogeneity in samples, which is beyond the intended scope of this document.

The other technique is called the "pressure falloff" method. It is characterized by upstream reservoir(s) only. The downstream end of the sample is vented to atmospheric pressure. The maximum upstream pressure used is fairly low, 10 to 250 psig (varying inversely with the permeability to be measured). A single transient pressure falloff produces data for 6 to 30 separate permeability calculations, each at a different flow velocity and mean pore pressure. Adequate variation of flow conditions during a single transient test makes possible the calculation of slip-corrected (Klinkenberg) permeability (k_∞), Klinkenberg slip factor (b), and inertial resistivity (β) of the porous medium. This technique, which has a useful permeability range of 0.001 to 30,000 millidarcys (through the use of multiple upstream gas reservoirs and pressure transducers), complements the pulse-decay method. For high permeability samples (>1,000 md), where b is small, it is difficult to determine b accurately when a sample is vented to atmospheric pressure. It is more reliably approximated from a correlation.

In addition to physical differences between flow systems, approaches to the derivation of flow equations are quite different for the pulse-decay and pressure-falloff techniques. In the former, Darcy's equation and the continuity equation (which is a statement of the conservation of mass) are solved simultaneously. In the pressure-falloff method, the steady-state solution of the slip-corrected Forchheimer equation is used as a starting point. This solution, which does not properly account for the increase in mass flux with distance along a sample's length at a particular instant (with non-steady-state flow), is differentiated. It is then inserted into the continuity equation, which is integrated to provide a correction (and improvement) to the final flow equation. This process is iteratively repeated until both the Forchheimer and continuity equations are satisfied.

The deviation between steady-state and pressure-falloff solutions thus obtained depends upon the ratio of the sample's pore volume to the volume of the upstream gas reservoir. When this ratio is small, the steady-state solution is nearly exact. As the ratio increases (larger PV or smaller reservoir volume), the correction that must be applied to the steady-state solution progressively increases.

6.4.1.1 Pressure-Falloff, Axial Gas Flow

6.4.1.1.1 Apparatus and Procedure

The pressure falloff apparatus (see Figure 6-22) employs an upstream gas manifold that is attached to a sample holder capable of applying hydrostatic stresses to a cylindrical sample of diameter D and length L. An upstream gas reservoir of calibrated volume can be connected to, or isolated from, the calibrated manifold volume by means of a valve. (Multiple reservoir volumes are used to accommodate a wide range of permeabilities.) The outlet port from the sample holder is vented to the atmosphere. This port is provided with a valve, which can be closed for start-up purposes. An accurate pressure transducer that measures gauge pressure is connected to the manifold immediately upstream of the sample holder.

The reservoir, manifold, and sample—to the outlet valve—are filled with gas. After a few seconds for thermal equilibrium, the outlet valve is opened to initiate the pressure transient. When the upstream pressure has decayed to about 85 percent of the fill pressure, during which time a smooth pressure profile is established throughout the length of the sample, data collection is started. Pressures at selected intervals and corresponding elapsed times are read and recorded.

6.4.1.1.2 Calculations

Procedures for calculating k_∞, b, and β from axial pressure falloff data are given in B.6.8.2.

6.4.1.1.3 Advantages

Advantages of this method include:

a. Slip-corrected permeability, k_∞, Klinkenberg gas slippage factor, b (up to about 1,000 md), and Forchheimer coefficient of inertial resistivity, β, can all be determined from a single pressure falloff test.

b. Non-slip-corrected permeability, k_g, can be calculated (see 6.4.1.1.5) from these results for any desired gas at any mean pore pressure for purposes of comparison with conventional measurements.

c. No flow meters are required for the method. Flow rates are calculated from pressure-time measurements.

d. For a core plug of a given permeability, the time required for pressure to fall from one pressure to another pressure is directly proportional to the volume of the upstream gas reservoir. Therefore, measurement times can be controlled for a wide range of permeabilities by providing several reservoir volumes—small for low permeabilities and larger for higher permeabilities.

e. The practical permeability range is from 0.001 to 30,000 millidarcys if multiple reservoir volumes and transducer ranges are used.

f. Departure from Stokes flow is not a concern. This is accounted for in the data analysis scheme.

g. Slip-corrected permeability measurements at high confining stresses more accurately reflect reservoir conditions, especially for low permeability samples.

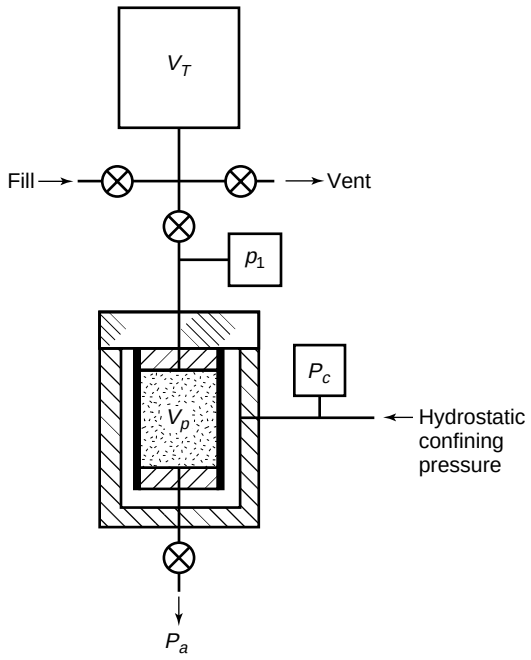


Figure 6-22—Schematic of Pressure-Falloff Gas Permeameter

6.4.1.1.4 Precautions and Limitations

Precautions and limitations of this method include:

a. To minimize thermal effects with high permeability plugs, small fill pressures (<25 psig), small diameter (1 inch), and long lengths (≥2 inches) should be used.

b. To minimize measurement time with low permeability samples, plug diameter should be large (1.5 inches) and its length short (≤1.5 inches).

c. The technique requires high quality pressure transducers, fast data acquisition equipment, and a high computational demand. As a practical matter, therefore, it requires a digital computer.

d. Gas reservoirs must be provided with considerable thermal mass and have high heat transfer capability to avoid work-related temperature changes during gas expansion, especially with high permeability samples. Temperature changes are further minimized by using helium, and by reducing the starting pressure for these falloff tests. High permeability samples also demand large diameter ports in the gas delivery system to minimize internal pressure differentials.

e. Low permeability samples require a leak-tight system (for this or any other technique), and no bypass in the sample holder (past the rubber sleeve). Adequate thermal insulation must be provided to minimize temperature changes induced from ambient changes.

f. Low permeability samples often exhibit extreme stress sensitivity. As mean pore pressure decreases, net stress increases (with a constant confining stress), and permeability decreases. In calculations, permeability is assumed to remain constant during the entire pressure falloff. This assumption can result in the calculation of low or negative β for very stress-sensitive samples. Because the largest changes in permeability occur at low net stresses, stress sensitivity is minimized by making measurements at higher net stresses (>2000 psi), and by reducing the variation in pore pressure during a test.

6.4.1.1.5 Calculation of Gas Permeability From Klinkenberg Permeability

Due to the historical prevalence of steady-state axial gas permeability measurements made without the Klinkenberg correction, huge data bases have been developed from these measurements. It is often desired to compare new data with previous measurements on the same reservoir. To do this, k_∞ must be "uncorrected" for gas slippage. The non-slip-corrected permeability, k_g, for any gas at any desired temperature and mean pore pressure can be calculated from a measured k_∞ and b as follows:

a. Calculate the sample's "gas slippage radius," r_b, (from Equation 13 in 6.2.1.1) using appropriate values of molecular

weight, viscosity, and the b obtained for the gas used in the measurement at the absolute temperature of the measurement.

b. From this r_b, find b from Equation 12 for the new gas, using its molecular weight and viscosity at the desired temperature.

c. From the new b, the measured k_∞, and the desired mean pore pressure, calculate k_g for the new gas from Equation 21.

These steps can be combined into a single equation:

$$k_g = k_\infty \left(1 + \frac{\frac{b_m \mu_c}{\mu_m} \sqrt{\frac{T_c M_m}{T_m M_c}}}{P_{m_c}} \right) \quad (31)$$

Where the subscript m refers to the measurement gas or condition, and c refers to the desired gas or condition for which k_g is to be calculated. To illustrate the use of Equation 31, suppose that for a particular rock, the measured k_∞ and b are 10.62 millidarcys and 6.57 psi, respectively, using helium at 72°F. For purposes of comparing this measurement with old data taken on the same formation, it is desired to calculate k_{air} at a mean pore pressure of 18.0 psia and temperature of 75°F. The molecular weight of helium is 4.0026, and its viscosity at 72°F is 0.01967 cp. The average molecular weight of air is 29.0, and its viscosity at 75°F is 0.01837 cp. Therefore, from Equation 31, k_{air} at 75°F and 18.0 psia is:

$$k_{air} = 10.62 \left(1 + \frac{\frac{6.57 \times 0.01837}{0.01967} \sqrt{\frac{(75 + 459.7)4.0026}{(72 + 459.7)29.0}}}{18.0} \right) = 11.97 \text{ md}$$

A note of warning: many of the earlier measurements were made at low stress levels, typically at 250 or 400 psi. Even with the above correction, the old k_{air} values on comparable rock may be higher if the new measurements were made at significantly higher stresses. Furthermore, old databases may not include the mean pressure and temperature required to perform these calculations.

6.4.1.2 Pressure Falloff, Probe Permeability Measurements with Gases

The pressure falloff technique is easily adapted for probe-permeameter measurements to provide rapid, non-destructive gas permeabilities that are corrected for both gas slippage and inertial resistance. As with steady-state devices, measurements are typically made at high sampling densities and nearly zero stress on slabbed or unslabbed whole core samples.

6.4.1.2.1 Apparatus and Procedure

Figure 6-23 schematically shows the instrument described by Jones.[22] To operate, the upstream volumes are filled with gas (usually dry nitrogen) to a pressure of about 10 psig. The probe is positioned above a sample, and then lowered and sealed against its surface by a fixed pneumatic pressure on the probe operator piston.

The pressure falloff is initiated by opening the internal valve nearest the probe. The appropriate gas reservoir volume for the particular permeability measurement is selected via internal valves in the manifold. This selection is made on the basis of the rate of pressure decline when all internal volumes are connected to the probe. If the rate is slower than a predetermined value, appropriate valve(s) to the larger tank(s) are closed.

Pressures at preselected intervals and corresponding elapsed times are read and recorded for the calculation of k_∞.

6.4.1.2.2 Calculations

Calculation procedures are discussed in C.6.8.3.

6.4.1.2.3 Advantages

Advantages of this method include:

a. The practical permeability range of this technique is from 0.001 to 30,000 millidarcys.

b. Measurement times in this range vary from 35 to 2 seconds.

c. No flow meters are required. Only time-pressure measurements are made.

d. Both slip-corrected (k_∞) and nonslip-corrected (k_g) permeabilities are obtained from a single pressure falloff test.

e. Inertial flow resistance effects, which can be severe even with small pressure differences and low permeability samples, are eliminated from the calculation of permeability with this technique.

f. The method is nondestructive in the sense that there is no need to cut core plugs. It permits high density sampling in sections that exhibit frequent and severe permeability variation.

g. The volume of rock sample investigated by a single test can be varied somewhat by varying the size of the probe tip seal dimensions. However, in general this volume is quite small (the permeability measured is localized). This statement also applies to steady-state measurements (see 6.3.1.2.3).

6.4.1.2.4 Precautions and Limitations

Except for the problems of inertial resistance and gas slippage, which are handled by the calculation procedures, the precautions and limitations that apply to steady-state probe permeability measurements (see 6.3.1.2.4) also apply to the pressure-falloff measurements.

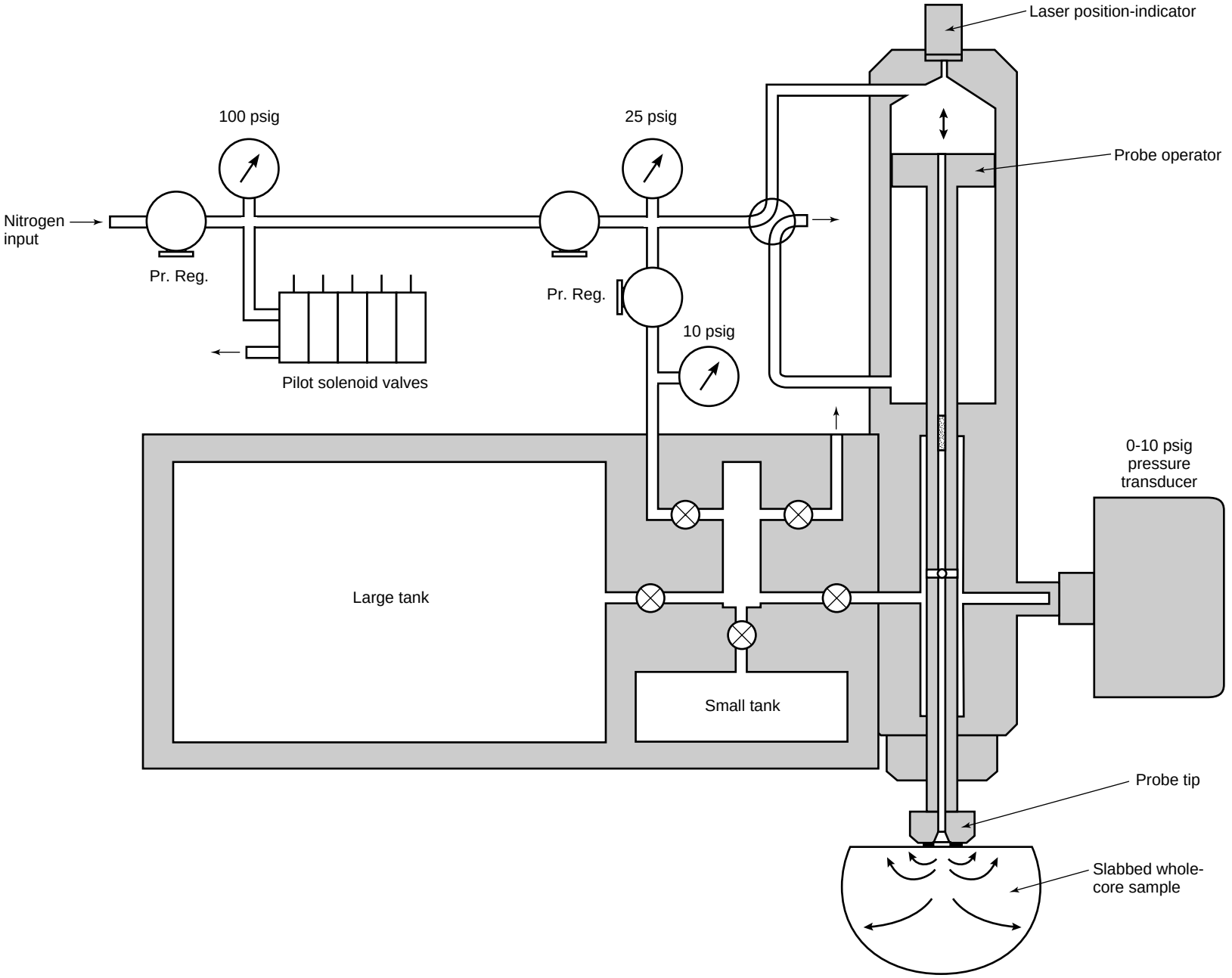


Figure 6-23—Schematic of Pressure-Falloff Probe Permeameter (U.S. Patent 5,237,854)

6.4.1.3 Pulse-Decay, Axial Gas Flow

6.4.1.3.1 Apparatus

The pulse decay apparatus, shown in Figure 6-24, consists of an upstream gas reservoir of volume V_1, a sample holder capable of applying high confining stresses (usually isostatic), which contains the sample of nominal pore volume V_p, and a downstream reservoir of volume V_2. A differential pressure transducer measures the pressure difference between the reservoirs, and a second transducer measures the absolute pressure in the downstream reservoir.

6.4.1.3.2 Procedure

With valves 1 and 2 open, both reservoirs and the sample are filled with gas (usually dry nitrogen) to a pressure typically between 1,000 and 2,000 psig. The fill period must allow adequate time for the gas to diffuse into the (typically) low-permeability sample. After the fill period, the fill valve is closed and the pressure is monitored until no further change is observed, indicating thermal and pressure equilibrium. This stabilized pressure is $P_2[0]$. All valves should be of the type that have no change in internal volume when opened or closed.

After equilibrium, valves 1 and 2 are closed, and the pressure in the upstream reservoir is increased by Δp_1, which is 2 to 3 percent of $P_2[0]$. After the pressure in V_1 becomes stable, i.e., when Δp_1 becomes constant, valve 1 is opened, initiating the pressure-transient portion of the measurement.

6.4.1.3.3 Calculations

See D.6.8.4 for details.

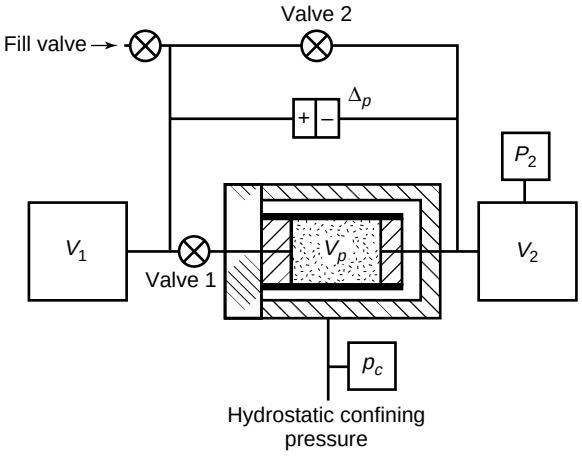


Figure 6-24—Pulse Decay Apparatus for Axial Flow of Gases

6.4.1.3.4 Advantages

Advantages of this method include:

a. The pulse decay technique is applicable to the measurement of very low permeabilities, 0.1 millidarcy to about 0.01 microdarcy, and possibly can be extended in either direction by careful selection of gas reservoir volumes and pressure transducer ranges.
b. No flow meters are required. Only time-pressure measurements are made.
c. These measurements are well adapted for application of reservoir-condition stresses to provide representative permeabilities.
d. Porosity can be measured simultaneously or separately in the same apparatus.

6.4.1.3.5 Precautions and Limitations

Precautions and limitations of this method include:

a. Due to the very low permeabilities that can be measured by this technique, leak-tightness of the apparatus is of ultimate importance. Control of ambient temperature variations is also critical.
b. Even though high back pressures are employed, permeabilities obtained are not corrected for gas slippage and may be somewhat high. For example if b for nitrogen were 100 psi for a particular plug, and the mean pore pressure were 1,000 psia, the gas permeability obtained would be 10 percent higher than k_∞.

6.4.2 Transient Pressure Techniques for Liquids

6.4.2.1 Pulse Decay, Axial Liquid Flow

Unless large pressure gradients are imposed (100 to 1,000 psi/cm or more), liquid permeabilities of tight rocks (k <0.1 millidarcy) are difficult to measure, or are at least time-consuming with steady-state methods. However, permeabilities down to about 0.01 microdarcy can be measured using pulse decay techniques.

Very low flow rates are calculated from rates of pressure change in liquid-filled reservoirs of known compressive storage—as liquid expands from an upstream reservoir or is compressed into one downstream of the sample. Thus, important parameters in these measurements are effective compressive storages of the upstream and downstream reservoirs and of the rock sample. The effective compressive storage of each reservoir is its internal volume multiplied by the sum of the liquid compressibility and the compressibility of the vessel. The sample's compressive storage is the product of its pore volume and the sum of liquid and PV compressibilities. The liquid pulse decay apparatus should have provision for measuring compressive storages of the upstream and downstream volumes (see E.6.8.5.1).

6.4.2.1.1 Procedure

For a pulse decay liquid permeability measurement (see Figure 6-25), a completely liquid-saturated rock sample is loaded into the sample holder, which is then pressurized, preferably to a fairly high (usually hydrostatic) confining stress. The high stress minimizes permeability variation from changes in net stress due to varying pore pressure. The system is filled with liquid, as described in E.6.8.5, and pressurized to the initial pore pressure. The fill valve is left open long enough to allow the high pressure liquid to diffuse into the sample. With valves 1 and 2 still open, the fill valve is closed, and the downstream transducer is monitored until no further change in pressure is observed. Then valves 1 and 2 are closed and the pressure pulse, Δp_1, is generated by screwing in the needle valve. All valves, except for the needle valve, should be of the type that have no change in internal volume when opened or closed.

The pulse decay is initiated by opening valve 1. The initial pressure difference across the sample,$\Delta p[0]$, will be slightly less than Δp_1, which was generated by the needle valve with valve 1 closed, thereby excluding the upstream dead volume, V_d, from the total upstream volume, V_1. Therefore $\Delta p[0]$ must be calculated, as shown in E.6.8.5.

6.4.2.1.2 Indication of Fairly Homogeneous Sample

The upstream pressure decreases while the pressure pulse traverses the length of the sample, but the downstream pressure remains constant until the pulse "emerges." Thereafter, the upstream pressure continues to decrease, and the downstream pressure rises. If the sample is reasonably homogeneous and upstream and downstream compressive storages are equal, then shortly after the pulse-propagation period, the upstream pressure decrease is offset by an equal downstream increase, and the mean pore pressure will remain constant as the $\Delta p[t]$ continues to decrease linearly—in a semi-logarithmic decay.

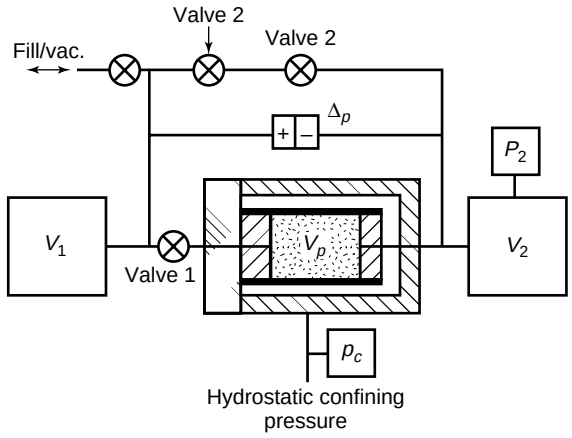


Figure 6-25—Schematic of Liquid Pulse-Decay Permeameter

6.4.2.1.3 Heterogeneous Sample Indication

However, if the sample is not homogeneous, but is characterized by a fracture system of relatively high fluid conductivity and a matrix porosity of low conductivity, and if the compressive storage of the sample is nearly as large, or larger, than those of the upstream and downstream reservoirs (small reservoirs), the mean pore pressure will continue to decrease even after $\Delta p[t]$ has reached zero, and the linearity of the semi-logarithmic pressure decay may have been distorted. In this case $\Delta p[t]$ is fairly quickly dissipated by the fracture system, but liquid continues to diffuse slowly into the tight matrix. If the reservoir storages are large compared to that of the matrix, the mean-pore-pressure decrease, due to liquid movement into the matrix, is barely observable. For a more complete discussion of the measurement of heterogeneity in core samples, see Kamath, et al.[29]

6.4.2.1.4 Calculations

See E.6.8.5.3 for details of calculations for axial liquid pulse decay measurements.

6.4.2.1.5 Advantages

Advantages of this method include:

a. No corrections for slippage are required for liquid measurements.
b. High stress liquid permeability measurements may be more representative of reservoir conditions than gas measurements.
c. This technique is useful for measurement of low permeability rocks (from about 0.01 microdarcy to 0.1 millidarcy).
d. The work of expansion or compression is small in liquid-filled systems. Hence work-related temperature changes are correspondingly small.

6.4.2.1.6 Precautions and Limitations

Precautions and limitations of this method include:

a. Maintenance of isothermal conditions is of utmost importance. Small ambient temperature changes can cause large pressure changes in high-pressure liquid-filled systems.
b. High quality pressure transducers and data acquisition system are required.
c. A leak tight system is essential for high pressure measurements of low permeability plugs.
d. The usual precautions of corrosion resistance, fluid compatibility, careful filtration of liquids, and exclusion of gas from the system must be observed.

6.5 ACCURACY AND PRECISION

6.5.1 Part 1

6.5.1.1 Introduction

Permeability data represent quantitative measurements, and correct values are important. All analytical methods are subject to random scatter and to operator, equipment, and technique-related errors, which, taken together, comprise experimental uncertainty. To distinguish between a real trend in a data set and variation due to experimental uncertainty, it is recommended that core analysis data be reported together with a statement of the uncertainty with which these data were recorded.

6.5.1.2 Definitions

6.5.1.2.1 precision (or reproducibility) of a measurement: An expression of the limits within which the measurement, with a stated probability, can be reproduced provided that the measurement is subject only to stochastic variation. Precision is determined from repeated measurements on a single sample, and is normally given as a standard deviation or relative standard deviation, also known as the coefficient of variation.

6.5.1.2.2 accuracy of a measurement: An expression of the closeness of agreement between the experimentally obtained value and the true value. The difference between the measured and the true value is called the error. Results falling outside the maximum allowable error or tolerance may be caused by operating errors or systematic errors.

6.5.1.2.3 bias (or systematic) error: A deviation that persists during a measurement cycle and affects all measurements in the same way. Bias is normally due to instrumental drift, or faulty calibration, or operating procedures.

6.5.1.2.4 applicability: The concept of applicability, in addition to precision and accuracy, is appropriate here. Permeability measurements may have been performed with high precision and accuracy, producing results close to true values of permeability for the reported conditions under which they were measured. Yet, these precise, accurate values might be quite different from those that would be obtained for conditions that exist in the formation from which the samples were taken. Reasons for differences include different stress conditions, rock-fluid interaction, differences in pore pressure, or gas composition if gas slippage is ignored, etc.

6.5.1.3 Error Statements in Permeability Measurements

The concept of accuracy requires that the true value of a sample be known. This is usually not the case. Instead certified standard reference materials for scientific or industrial use are obtained from recognized standard institutions. These materials are analyzed at regular intervals together with routine samples. Accuracy is then obtained by comparing the mean value, calculated from repeated analyses on the standard material, with the certified value for that standard.

In permeability measurement, suitable standard materials could be natural rocks or permeable synthetic materials. However, synthetic samples cannot yet be produced in a way that allows an accurate pre-determination of permeability. Therefore, each standard must pass an extensive testing program before it is certified, and afterwards there is risk that the standard will change during regular handling in the laboratory. So far, no certified permeability standards have been available from recognized standard institutions. Therefore, a different approach for determining accuracy in permeability measurement is recommended.

6.5.1.4 Internal Standards

The concept of internal permeability standards is introduced for determining accuracy, and as a facility in testing equipment for bias or systematic errors. Internal standards can be synthetic and/or natural samples covering a large range of permeability values, which have been analyzed repeatedly by different operators on different (carefully calibrated) apparatus using the required fluid. When many measurements have been collected, a mean value and standard deviation is calculated for the sample. If the variation about the mean is small, i.e., the standard deviation is low, the sample is accepted as an internal standard, and can be used routinely to check accuracy of equipment and correct for bias in the results.

Synthetic porous and permeable media are available in different materials, e.g., as porous ceramics and glasses, and as sintered plastic, glass, or metal beads. These materials are often supplied from the manufacturer with a specified permeability value, which, however, should only be regarded as approximate.

6.5.1.5 Methodological Errors

All equations for calculating permeability presented herein assume fluid flow through homogeneous, isotropic samples, under isothermal conditions. Thus they define an average or effective permeability in the overall direction of flow dictated by the experiment (i.e., axial, radial, transverse, etc.). Unless the internal, macroscopic structure of each sample is determined by independent means, it is virtually impossible to model the flow otherwise. Because real rocks are almost *never* isotropic or homogeneous, the assumption that they are will invariably distort actual flow patterns, and consequently, the calculated permeability, and also inertial resistivity and gas slippage factor, whenever they apply.

Equations for unsteady-state analyses require correction for nonconstant mass flow (with distance from the injection point) at any instant during the pressure transient. Therefore,

a sample's pore volume must be determined, either independently or in conjunction with the permeability measurement itself. Inaccuracies in the estimation of pore volume will lead to permeability errors, especially when *PV* is nearly as large, or larger than the gas reservoir volume. Small *PV* errors are inconsequential when reservoir volumes are large on a relative basis.

Furthermore, the transient techniques assume constant permeability throughout the duration of a test. Changes in pore pressure change the net stress to which the sample is subjected, thereby slightly changing its permeability during a test. In a pressure falloff measurement, this can distort the calculation of inertial resistivity (β). These errors are minimized by limiting the magnitude of the pressure falloff, and by increasing the confining stress (where changes in net stress have less effect on permeability—per Figure 6-9).

Serious errors can result when operating conditions are such that Darcy's law does not apply because of significant inertial resistance. These are most prevalent with steady-state, axial flow gas measurements on high permeability plugs (several darcys), or steady-state probe permeability measurements on samples of about 10 millidarcys or more. These errors are minimized by using very small values.

Other errors can be introduced by improper sample preparation techniques, such as inadequate cleaning, or nonremoval of salt crystals from extracted samples that previously contained nearly saturated brine. Improper drying of samples with high clay content can either enhance or reduce permeability. Embedded fines from cutting or grinding the ends of samples can reduce permeability. High points on nonsquare ends of a core plug can be crushed at high axial stresses, altering permeability. Incompatible or dirty liquids cause plugging. High velocity fluids (especially liquids) can cause fines migration and plugging.

Gas permeabilities (uncorrected for gas slippage), measured at low confining stress, if properly reported (including the gas used, mean pore pressure, and net stress), are properly specified and do not per se constitute an error. However, if they are expected to predict reservoir performance accurately without correction, they may not be particularly applicable, especially for lower permeability samples, where the corrections are substantial.

6.5.1.6 Equipment Component Errors

Main error sources associated with test equipment are leaks, sample bypass, pressure transducer error, flow meter error, and ambient temperature variation. Additionally, nonisothermal fluid expansion, inaccurate calibration of reservoir volumes, and inadequate data acquisition resolution and speed can cause errors in unsteady-state measurements. With probe permeameters, probe positioning accuracy and sealing force repeatability are also important.

6.5.1.6.1 Leaks and Sample Bypass

All types of permeameters are subject to leaks in tubing, fittings, valves, transducers, flow meters, and core holder. Bypass of fluid between a core plug and rubber sleeve of the sample holder, or between a sample and the probe seal of a probe permeameter, also constitutes a leak. The magnitude of error caused by a leak generally increases as permeability decreases. For example, a small leak in the measurement of a 3 darcy plug would barely affect its calculated permeability, whereas the same leak rate with a 0.001 millidarcy plug would be disastrous.

Large leaks can be located in gas permeameters with the aid of a soap solution that forms bubbles when applied to a leak. Small leaks are most easily located by means of pressure measurements. Various portions of the flow system are pressurized with gas, then monitored by the system's pressure transducer. The leak is located through a process of elimination. It may be necessary to load an impermeable steel plug in the sample holder to isolate the upstream and downstream portions of the apparatus. Some systems incorporate automated leak-detection routines.

Sample bypass usually can be eliminated by increasing confining stress and/or reducing the hardness (durometer) of the rubber.

6.5.1.6.2 Pressure Transducer Error

Accuracy of a pressure transducer is usually specified as the root sum of squares (RSS) of deviations of its output from actual pressures throughout its range as a percent of its full scale (FS) reading. RSS accuracies of 0.5 percent FS or better are generally available. Most transducers are provided with at least two adjustments—zero and span. The zero adjustment is an offset to permit zero electrical output at zero applied pressure. The span adjustment changes the transducer's gain or multiplier that converts its electrical output to a pressure. Some transducers also have one or more internal linearity adjustments. Pressure transducers are also affected by temperature changes, and are provided with temperature compensation. Price is often nearly proportional to the quality of the temperature compensation.

The user should keep in mind consequences of the "full scale accuracy" specification. For example, a 0.5 percent transducer, used at 10 percent of full scale, can have a 5 percent error in the value of its pressure reading, and be within specification. In view of the RSS definition, the particular error at a given point could be greater than the given RSS percent of the full scale reading.

With proper calibration techniques that allow for frequent mathematical rezeroing capability and nonlinear curve fitting, as outlined in 6.61, a transducer's repeatability is among its most important characteristics. High quality transducers with 0.01 or 0.02 percent FS repeatability are available. To minimize hysteresis effects, a transducer should always be cali-

brated in the direction of its anticipated use. For example, for pressure falloff measurements, a transducer should be calibrated to its full scale pressure first, followed by a sequence of descending pressures.

Unsteady-state techniques place extra demands on pressure measurements. Pressure-time derivatives are calculated. Therefore noise, or small voltage fluctuations must be minimized, and resolution of the reading must not be truncated by an analog-to-digital voltage converter (ADC) with too few bits. Noise is minimized by starting with a good quality transducer with low noise output and shielded cables. An analog, low-pass filter can be used in conjunction with digital filtering. With a reasonably fast ADC (1,000 conversions per second) multiple readings can be taken, then averaged. A slow, integrating ADC (60 conversions per second) performs averaging automatically. A 15 bit ADC (0.0031 percent of full scale resolution) minimum is recommended for unsteady-state measurements to minimize truncation error.

High quality timer cards are available, with accuracies in the part per million range. It is imperative to query the timer card immediately either before or after reading the ADC, with no intervening commands in the operating program, so that pressure intervals correspond exactly to time intervals for the calculation of pressure-time derivatives. Furthermore, pressure intervals must be sufficiently large that noise in the measurements does not destroy accuracy of the derivative calculation. Optimum intervals may have to be determined by trial and error for a particular system.

A non-smooth plot of pressure-time derivatives vs. geometric mean pressures from pressure falloff data indicates either excessive noise or truncation of time or pressure readings. The problem is usually eliminated by increasing the pressure and/or time intervals, increasing the resolution (number of bits) of the ADC, or by improving the quality of the transducer. The sampling routine should be written in assembly language for maximum speed.

6.5.1.6.3 Flow Meter Error

Steady-state techniques depend on accurate flow rate determination. Direct reading volumetric devices in combination with time readings can yield accuracies of better than 1 percent. Indirect devices (calibrated orifices, mass flow meters and controllers) can normally be adjusted to yield a precision better than 1 percent, but accuracy is totally dependent upon calibration. Several flow meters are required to cover the entire permeability range. Each one should be used only within its linear working range. See 6.6 for mass flow meter calibration.

6.5.1.6.4 Ambient Temperature Variation

Changes in temperature affect both volumetrics and fluid viscosity. Temperature should be kept constant during a permeability measurement. Never place the apparatus near a heating or cooling vent or near a window where the sun's radiation can cause large temperature changes. In unsteady-state equipment, the rate of temperature change within fluid reservoirs must be such that temperature-induced pressure changes are small relative to pressure decreases that are caused by flow of fluid from a reservoir.

In field applications of probe permeameter measurements, temperature changes affect fluid viscosity, flow rate and pressure measurements.

6.5.2 Part 2

6.5.2.1 Error Statements in Reporting

Presentation of measured data with associated error limits in a core analysis report can be based on two different approaches: (a) measured values are used to calculate and report a mean value with associated error limits as calculated below, and (b) measured values are given together with a statement of accuracy, which the laboratory can base on repeated measurements of internal standards.

6.5.2.1.1 Calculated Error Limits

Suppose that a large number of repeated measurements (normally more than 100) are performed on a single sample. If the measurements are only subject to stochastic variation, it is generally believed that data have a normal or gaussian distribution with a mean value, $\bar{X}$:

$$\bar{X} = \frac{1}{n}\sum_{i=1}^{n} X_1$$

and standard deviation, *s*:

$$s = \sqrt{\frac{\sum_{i=1}^{n}(X_i - \bar{X})^2}{n-1}}$$

Where $(X_i - \bar{X})$ is the deviation of a single measurement from the mean and n is the total number of measurements. The result is reported as $\bar{X} \pm s$. The standard deviation is a measure of the spread or variation of data about the mean. A normal distribution has a probability that 68.3 percent of the observations will fall within the limits of plus or minus one standard deviation from the mean. The coefficient of variation, *CV,* is the relative standard deviation expressed as a percentage:

$$CV = 100(s/\bar{X})$$

Often, only a limited number of measurements are recorded. This means that we are dealing with small sample statistics and the *t*-distribution should be applied. Often it is more relevant to define confidence intervals about the mean than to quote the deviation. The confidence interval is an esti-

mate of the limits within which the true value lies with a stated probability. When reporting core analysis results it is therefore recommended to quote results as:

$$\bar{X} \pm t(s/\sqrt{n-1})$$

which is the confidence interval. Values of *t* are found in most statistical textbooks or in statistical tables, e.g., Fisher and Yates[31]. These values are given for various "degrees of freedom," *n*–1, and for different "confidence levels" or probabilities. A 95 percent confidence level is often used (corresponding to standard deviations, adjusted for the small number of repeat measurements).

The following example illustrates these concepts. Three repeat measurements were made on a sample. Calculated permeabilities are 3.35, 3.18, and 3.42 millidarcys, from which the mean and standard deviation are calculated:

$$\bar{X} = 3.32 \text{ md}$$

$$s = 0.12 \text{ md}$$

The value of *t* for 2 degrees of freedom (= *n*–1, where *n* = 3 measurements) and a 95 percent confidence level is 4.303. Therefore the confidence interval is:

$$3.32 \pm 4.303(0.12/\sqrt{3-1}) = 3.32 \pm 0.37 \text{ md}$$

In other words, these calculations imply that with a probability of 95 percent, the true permeability lies within the interval from 2.95 to 3.69 millidarcys, provided that the measurements were not biased.

Klinkenberg permeability is calculated from gas permeability measurements by performing linear regression on permeability vs. reciprocal mean pore pressure values. The goodness of fit of the regression line can be assessed from the coefficient of correlation, *r*, which should be reported with the permeability value. If we still want to have a confidence level of 95 percent, *r* must be greater than or equal to 0.95 for a 4 point regression line, and at least 0.997 for a 3 point line. Samples giving values of *r* lower than these should be inspected for fractures, vugs, or other irregularities, and if present this should be reported in the data listing. Also the measurements should be checked for the possibility of non-negligible inertial resistance at the higher mean pore pressures.

6.5.2.1.2 Laboratory Reported Error Limits

In routine core analysis, a large number of determinations on internal standard plugs will be accumulated over a period of time. It is valid that a laboratory prepare a table showing the accuracy and precision obtained for different permeability ranges based on standard measurements.

An impression of the accuracy that can be obtained in permeability measurements was reported by Thomas and Pugh[32]. Using conventional steady-state measurements performed by many laboratories all over the world on sets of standard plugs, they quoted the statistically derived confidence intervals with a probability (confidence level) of 99 percent from which the other confidence intervals were calculated in the table below.

	Confidence interval, %, with probability of:		
k_g range, millidarcys	68.3% (= mean CV)	95%	99%
0.01-0.1	±8%	±16%	±21%
0.1-1.0	8	16	21
1-50	5	10	13
50-1,000	3	6	8

The numbers shown in this table can be interpreted as follows. Suppose a certain core plug has a true permeability of 10.0 millidarcys, which lies in the 1-50 range above. Thus, if a total of one thousand measurements were made on this plug by laboratories throughout the world, approximately 683 of the reported permeabilities (68.3 percent of the total) would be expected to lie within the range from 9.5 to 10.5 millidarcys (±5 percent of the true value). Similarly 950 of them would be expected to lie within 9.0 to 11.0 millidarcys, and 990 within the range from 8.7 to 11.3 millidarcys (with 10 measurements lying outside this range).

6.5.2.1.3 Number of Significant Figures

The number of figures given in a report should respect the error associated with the measured value. For example, 4.2357, ±0.0327 md should be reported as 4.24, ±0.03 md; i.e., the error is understood to occur on the last decimal in the rounded number. In view of the table above, probably only two significant figures are justified (i.e., 4.2 md), unless a particular laboratory produces significantly better results than those upon which the table is based.

6.6 CALIBRATION OF INSTRUMENTS

6.6.1 Pressure Transducer Calibration

This discussion applies to a gauge pressure transducer, which should read zero when it is exposed to ambient atmospheric pressure. It also applies to an absolute transducer, provided that a high vacuum is applied for "zero (absolute) pressure" readings. Any pressure transducer must be calibrated periodically. Even though a transducer is normally calibrated at the factory within stated tolerances, it should be recalibrated in its flow system configuration, with the system's analog-to-digital converter (ADC). This is accomplished with a high quality primary standard, such as a dead weight tester or precision manometer and cathetometer, or with a secondary standard pressure transducer (preferably

accurate to 0.01 to 0.02 percent full scale), traceable to a primary standard. A known pressure is applied, and the resulting voltage or ADC output is read. This is repeated at several, nearly equally spaced intervals throughout the range to be calibrated, including zero pressure. A 21 point calibration (intervals of 5 percent of full scale), minimum, is desirable.

6.6.1.1 Calculations

These data (if reasonably linear—to within 0.1 to 0.5 percent of full scale) are fit with a polynomial, using least squares techniques. A good rule of thumb for choosing the polynomial order is: the number of data points taken in the calibration must be at least double the number of coefficients calculated from the polynomial fit. For 21 points or more, a fourth-order polynomial is a good choice.

In general, it is nearly impossible to set the transducer's zero-adjust potentiometer so that the transducer outputs exactly zero voltage at zero pressure at the time of calibration. Furthermore, some ADCs read only positive voltages (negative voltages are output as zero). For these ADCs, the transducer's zero offset must be sufficiently positive that zero drift will never cause a negative zero-pressure voltage. A fourth-order polynomial, with which calibration data are fit, has the form:

$$p = a_0 + a_1 v + a_2 v^2 + a_3 v^3 + a_4 v^4 \quad (37)$$

Where p is the actual pressure (determined from the standard), and v is the voltage read from the transducer for each data point. If the zero-pressure voltage is positive, then a_0, from the least-squares fit, will be negative.

It is critical to have calibrations that pass through zero at zero pressure. To illustrate, suppose that a zero-pressure voltage shift of 0.5 percent of the full scale voltage occurred—with time and temperature changes—from the zero-pressure voltage at the time of calibration. This magnitude of zero shift is not uncommon, and does not appear to be too bad. However, even with a perfect span adjustment, this represents a 5 percent error for a pressure reading at 10 percent of full scale, or a 25 percent error for a pressure reading at 2 percent of full scale.

To avoid this problem, operating software should have provision to "mathematically zero" all transducers. Unless a_0, from the calibration, is identically equal to zero in Equation 37, zero drift cannot be corrected by simply subtracting the current zero-pressure voltage from all subsequent voltage readings, then using these voltage differences in Equation 37. Instead, Equation 37 must first be mathematically transformed to:

$$p = A_0 + A_1(v - v_0) + A_2(v - v_0)^2 + A_3(v - v_0)^3 + A_4(v - v_0)^4 \quad (38)$$

To accomplish this transformation, the "best fit" zero-pressure voltage, v_0, at the time of calibration, must be found. This voltage is calculated from all points in the calibration, not just the value read when zero pressure was applied. A first estimate is calculated from:

$$v_0 \approx -a_0 / a_1 \quad (39)$$

This value of v is inserted into Equation 37 to calculate the "best fit" pressure (p_0) that corresponds to this voltage. If the value of v_0 were correct, then p_0 would be exactly equal to zero. The estimate is refined using:

$$v_0[new] = v_0[old] - p_0 / a_1 \quad (40)$$

This new value of the zero-pressure voltage is inserted into Equation 37 to calculate a refined value of p_0. Then these values of v_0 and p_0 are used in Equation 40 to produce a further refinement of v_0. This process is continued until $p_0 \leq 1 \times 10^{-10}$ psig.

Next, the coefficients $A_0 \ldots A_4$ are determined by a least-squares fit of the original calibration data using Equation 38 with the final value of v_0 as determined above. Equations 37 and 38 yield exactly the same calibration, except Equation 38 (after its coefficients have been determined) permits elimination of zero drift by inserting the current value of v_0.

The coefficients $A_0 \ldots A_4$, for a particular transducer, must be installed in an appropriate file in that transducer's operating program. The current value of v_0 must be periodically determined by the operating program of a particular instrument. It is found by pressurizing the transducer to its maximum pressure normally used in the instrument. It is then vented to atmospheric pressure and allowed to stabilize (usually for 30 seconds), after which v_0 is read and stored for subsequent computations with Equation 38.

The fit by a fourth-order polynomial reduces the average absolute deviation by a factor of 4 to 20, compared to that of a *linear* least-squares fit. Equation 38 also eliminates zero drift. The overall improvement (especially at low pressures) is significant compared to a calibration that has not been corrected for zero drift. The coefficient A_0 in Equation 38 should theoretically be equal to zero. It is usually less than 1×10^{-8}, and is calculated and reported only to assure that the transformation of Equation 37 to Equation 38 was successful. For purposes of calculating pressure from an ADC-output, A_0 should be deleted from Equation 38.

6.6.2 Calibration of Mass Flow Meters

Gas mass flow meters respond to mass flow rate, not volumetric flow rate. The volumetric flowrate at some reference condition of temperature and pressure (usually the current ambient temperature and atmospheric pressure), of gas flowing through the mass flow meter, is measured by some independent device (such as a soap bubble meter for low flow rates, which is connected in series with the mass flow meter).

Alternatively, a floating piston with mercury seals in a precision bore volumetric displacement device may be used. Voltage (or some scaled reading) from the mass flow meter is often calibrated against this volumetric flow rate, thereby providing an equation from which volumetric flow rate can be calculated from a voltage acquired. This is not the correct procedure, and unnecessarily limits the calibration! This volumetric flow rate calibration is valid only if the mass flow meter is used at exactly the calibration temperature and pressure. Changes in either pressure or temperature cause errors in the calibration, which could be avoided by simple procedural changes.

Because the mass flow meter's output is proportional to the mass flow rate of a gas, the volumetric flow rates through the bubble meter during a calibration must be multiplied by the absolute pressure in the standard meter (not in the mass flow meter). This is normally the current atmospheric pressure, which can be read from a good quality mercury barometer, with appropriate corrections. This pressure must be converted to the same units that are used in the calculation of permeability (e.g., psia, Pa, or atm). In addition, the volumetric flow rates must be divided by the absolute temperature, (459.67+°F), or (273.15+°C), in the standard meter, and by z, the gas deviation factor, calculated at the temperature and pressure within the standard meter. We will define the result of this manipulation as the "mass flow factor," y_{mf}:

$$y_{mf} = \frac{qP_{cal.}}{z_{cal.}T_{cal.}} \tag{41}$$

The subscripts, *cal.*, above refer to the absolute pressure and temperature that correspond to the volumetric flow rates measured during the calibration.

The advantage of this procedure is that the calibration is not restricted to exactly the same run-time pressure and temperature. Except for small instrumental shifts, the mass flow meter could be operated at any pressure and temperature within its specifications. This often, for example, permits the flow meter to be used at upstream conditions. Upstream operation has advantages: (a) no sand or grit from the core plug enters the flow meter, degrading its accuracy, (b) there is virtually no flow resistance between the mass flow meter and the core plug, allowing faster attainment of steady-state conditions.

Because of instrumental shifts, the mass flow meter should be calibrated at several different pressures and temperatures for most accurate results. Remember that, even though calibrations are reported for conditions in the mass flow meter, the pressure and temperature in the bubble meter (or other volumetric device) are used to calculate the mass flow factors. Normally, effects of temperature and pressure changes are small, and can be interpolated easily.

6.6.2.1 Calculations

For purposes of fitting calibration data, the same procedure should be used as described for pressure transducers, to produce a calibration equation that can be corrected for zero drift. In Equations 37, 38, and 39, the p and p_0 are replaced by y_{mf} and y_{mfo}, respectively. If only 6 to 8 flow rates are calibrated at each pressure, then the fourth-order polynomial should be replaced by a second order polynomial. A minimum of 11 equally spaced points should be obtained for the fourth-order polynomial. Whatever polynomial is used, it should be carefully tested by calculating 50 to 100 points over the entire range to make sure that the final equation does not produce unexpected results between calibration points.

Voltage (or the instrument's reading) at zero mass flow rate is determined at run time by closing valves on both the upstream and downstream ends of the mass flow meter (at its current operating pressure) and ensuring that there is absolutely no flow through it. After 30 seconds for stabilization, output from the ADC, v_0, should be recorded and subtracted from the flowing voltage output, in the flow-equivalent of Equation 38.

Mass flow factors are used in the calculation of gas permeability as follows:

$$k_g = \frac{29392\mu z_m y_{mf} T}{G_f(P_1 - P_2)(P_1 + P_2)} \tag{42}$$

Where:

k_g = non-slip-corrected gas permeability, millidarcys.
μ = gas viscosity, cp.
z_m = gas law deviation factor, calculated at the mean pore pressure and current temperature.
y_{mf} = mass flow factor, (cm^3/s)(psia/°R), or (cm^3/s)(psia/°K).
T = absolute temperature at run time; same units as $T_{cal.}$, °R, or °K.
G_f = dimensional geometric factor for a particular flow configuration, cm.
P_1 = absolute upstream pressure, psia.
P_2 = absolute downstream pressure, psia.

In other words, $y_{mf}T$ is used in place of $q_r P_r / z_r$ in the calculation of permeability. If other units than the above are used in Equation 42, the 29392 is replaced by $2C_2 / C_1$ (see Table 6-1).

6.6.3 Reference Volume Calibration

Unsteady-state gas permeameters contain gas reservoir volumes and dead volumes that must be accurately calibrated. One convenient method is to use gas expansion in conjunction with Boyle's Law, using seven or eight cylindrical steel plugs, each of which contains an accurately-measured axial hole that extends through its entire length. The length and diameter of each hole should be measured to the nearest 0.0001 inch. These plugs must fit inside the sample holder, and must be tightly sealed by the holder's rubber sleeve. The

holes should range in volume from nearly zero to about the volume of the reservoir to be calibrated. The hole volume changes, from one plug to the next, should all be approximately equal. The pressure transducer must be carefully calibrated prior to the volumetric calibration, and it must read zero at zero psig. Ambient temperature changes during the volumetric calibration should be minimized.

6.6.3.1 Calculations

Because the configuration of each system may vary slightly, only the principles of a Boyle's Law calibration will be illustrated. Boyle's Law states that the total of the masses of gas in each part of the system before an expansion is equal to the total mass after the expansion; i.e., no gas enters or leaves the system. Under isothermal conditions, the group (VP/z) is proportional to the mass of gas in volume V, where P is the absolute pressure, and z is the gas deviation factor at the pressure and temperature of the gas contained in volume V.

Referring to Figure 6-26, suppose that one of the several steel plugs has been loaded into the sample holder and is sealed by its rubber sleeve. Then the gas reservoir of volume V_0, that includes the bore of the isolation ball valve; the upper dead volume V_1, which includes the volumes inside the transducer and upper end plug; the known hole volume in the steel plug, V_H; and the lower dead volume, V_2, are filled with helium or nitrogen to pressure p_1 (a gauge pressure). For the most accurate work, this gas should be vented from the system to remove air. The fill-vent cycle should be repeated, and the system filled a third time. Then the isolation valve is closed, and V_0 is vented to atmospheric pressure, after which the vent valve is closed. After pressure equilibration, the final pressure, p_1, is read and recorded.

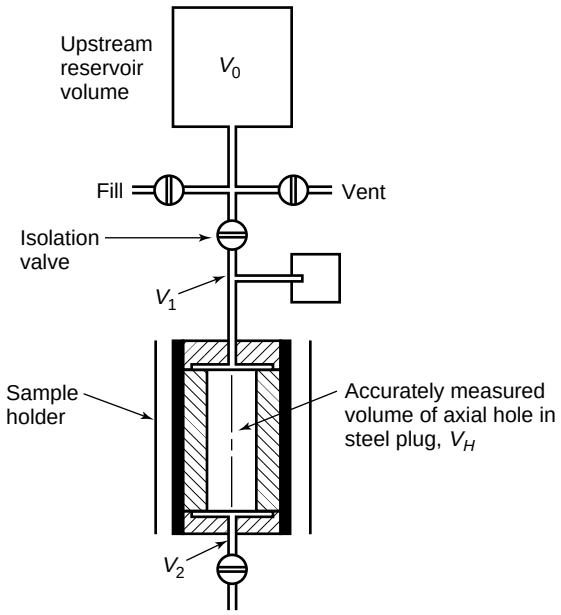


Figure 6-26—Schematic for Calibration of Volumes in Unsteady-State Permeameters

The isolation valve is now opened, expanding gas from the lower portions of the system into V_0. After pressure equilibrium is attained, the final pressure in all parts of the system, p_2, is read and recorded. Each of the remaining steel plugs is loaded into the sample holder and the procedure repeated. Atmospheric pressure, p_a, must be read from an accurate mercury barometer with appropriate corrections.

Now the mass of gas in each part of the system before an expansion is entered on the left hand side of the equation below, and the masses after the expansion are on the right hand side:

$$(V_1 + V_2 + V_{H_i})\left(\frac{p_1 + P_a}{z_1}\right) + (V_0)\left(\frac{P_a}{z_a}\right) = (V_1 + V_2 + V_{H_i} + V_0)\left(\frac{p_2 + P_a}{z_2}\right) \quad (43)$$

A similar equation is obtained for each of the steel plugs. These equations are rearranged to:

$$V_{H_i} = V_0\left[\frac{\frac{p_2 + P_a}{z_2} - \frac{P_a}{z_a}}{\frac{p_1 + P_a}{z_1} - \frac{p_2 + P_a}{z_2}}\right]_i - (V_1 + V_2) \quad (44)$$

The volumes V_0 and $-(V_1 + V_2)$ are now found as the slope and intercept, respectively, from linear regression, where V_H is the y-variable, and the quantity inside the brackets is the x-variable for each plug.

The upper dead volume, V_1, is found by inserting a solid steel plug into the core holder. Gas is expanded from this volume into the now known volume, V_0. An equation similar to Equation 43 is written for the new configuration, and solved for V_1. The reservoir volume, V_T, for a transient permeability measurement is equal to V_1 when the isolation valve is closed, or to $(V_0 + V_1)$ when it is open.

6.7 REFERENCES

1. Darcy, Henry, *Les Fontaines Publiques de la Ville de Dijon*, Victor Dalmont, Paris, 1856.

2. Forchheimer, P., "Wasserbewegung durch Boden," *Zeits. V. Deutsh. Ing.*, 1901, 45, 1782-88.

3. Klinkenberg, L. J., "The Permeability of Porous Media to Liquids and Gases," *Drilling and Production Practice*, 1941, 200-213

4. Kundt, A. and Warburg, E., *Poggendorfs Ann. Physik.*, 1875, 155, 337 and 525.

5. Kozeny J., "Uber Kapillare Leitung des Wassers im Boden, Sitzungsber," *Akad. Wiss. Wien, Math. Naturw Klasse, Abt.II* A, 1927, 136, 271-306.

6. Carman, P. C., "Fluid Flow through Granular Beds," *Trans. Inst. Chem. Eng.*, London, 1937, 15, 150-156.

7. Wyllie, M. R. J. and Rose, W., "Some Theoretical Considerations Related to the Quantitative Evaluation of the Physical Characteristics of Reservoir Rock for Electrical Log Data," *Trans.*, AIME, 1950, 189, 105-118.

8. Swanson, B. F., "A Simple Correlation Between Permeabilities and Mercury Capillary Pressures," *J. Pet. Tech.*, 1981, 33, 2498-2504.

9. Katz, A. J. and Thompson, A. H., "Quantitative Prediction of Permeability in Porous Rock," *Phys. Rev. B*, 1986, 34, 8179-81.

10. Archie, G. E., "The Electrical Resistivity Log as an Aid in Determining Some Reservoir Characteristics," *Trans.*, AIME, 1942, 146, 54-62.

11. Ondracek, G., "Effect of Microstructure on Conductivity of Composite Materials," *Zeits. fuer Metallkunde*, Sept. 1986, 77, No. 9, 603-610.

12. Biot, M. A. and Willis, "Elastic Coefficients of the Theory of Consolidation," *J. Appl. Mech.*, 1957, 57, 594-601.

13. Moran, J. H. and Papaconstantinou, C. M., "A Novel Dynamic Measurement of Permeability," *SPEJ*, December 1981, 670-8.

14. Hougen, O. A. and Watson, K. M., *Chemical Process Principles, Part Two, Thermodynamics*, John Wiley & Sons, New York, fifth printing, Sept. 1949, 482-3.

15. Prutton, C. F., and Maron, S. H., *Fundamentals of Physical Chemistry*, The Macmillan Company, Revised Edition, 1951, 23 and 49.

16. Jones, S. C., "Using the Inertial Coefficient, β, to Characterize Heterogeneity in Reservoir Rock," paper SPE 16949 presented at the 1987 SPE Annual Technical Conference and Exhibition, Dallas, Sept. 27-30.

17. Geertsma, J., "Estimating the Coefficient of Inertial Resistance in Fluid Flow Through Porous Media," *SPEJ*, October 1974, 445-50.

18. Jones, S. C., "Two-Point Determinations of Permeability and PV vs. Net Confining Stress," *SPE Formation Evaluation*, March 1988, 235-41.

19. Goggin, D. J., Thrasher, R. L., and Lake, L. W., "A Theoretical and Experimental Analysis of Minipermeameter Response Including Gas Slippage and High Velocity Flow Effects," *In Situ*, 12, No. 1 & 2, 1988, 79-116.

20. Collins, R. E., "Determination of the Transverse Permeabilities of Large Core Samples from Petroleum Reservoirs," *J. of App. Physics* 23, No. 6, June 1952, 681-4.

21. Jones, S. C., A Rapid Accurate Unsteady-State Klinkenberg Permeameter," *SPEJ*, Oct. 1972, 383-97; *Trans*. AIME, 253.

22. Jones, S. C., "A New, Fast, Accurate Pressure-Decay Probe Permeameter," *SPE Formation Evaluation*, Sept. 1994, 193-99.

23. Dicker, A. I. and Smits, R. M., "A Practical Approach for Determining Permeability from Laboratory Pressure-Pulse Decay Measurements," paper SPE 17578 presented at the 1988 SPE International Meeting on Petroleum Engineering, Tianjin, China, Nov. 1-4.

24. Brace, W. F., Walsh, J. B., and Frangos, W. T., "Permeability of Granite Under High Pressure," *J. Geophysical Research*, 1968, 73, 2225-36.

25. Bourbie, T. and Walls, J., "Pulse Decay Permeability: Analytical Solution and Experimental Test," *SPEJ*, Oct. 1982, 719-21.

26. Hsieh, P. A. et al., A Transient Laboratory Method for Determining the Hydraulic Properties of 'Tight' Rocks—I Theory," *Intl. J. Rock Mech. Min. Sci. and Geomech. Abstr.*, 1981, 18, 245-52.

27. Chen, T. and Stagg, P. W., "Semilog Analysis of the Pulse-Decay Technique of Permeability Measurement," *SPEJ*, Dec. 1984, 639-42.

28. Haskett, S. E., Narahara, G. M., and Holditch, S. A., "A Method for the Simultaneous Determination of Permeability and Porosity in Low-Permeability Cores," *SPEFE*, Sept. 1988, 651-88.

29. Kamath, J., Boyer, R. E., and Nakagawa, F. M., "Characterization of Core-Scale Heterogeneities Using Laboratory Pressure Transients," *SPEFE*, Sept. 1992, 219-27.

30. Amaefule, J. O., et al., "Laboratory Determination of Effective Liquid Permeability in Low-Quality Reservoir Rocks by the Pulse Decay Technique," paper SPE 15149 presented at the 1986 SPE California Regional Meeting, Oakland, April 2-4.

31. Fisher, R. A., and Yates, F., *Statistical Tables for Biological, Agricultural and Medical Research*, Oliver and Boyd, London, 1963.

32. Thomas, D. C., and Pugh, V. J., "A Statistical Analysis of the Accuracy and Reproducibility of Standard Core Analysis," *The Log Analyst*, March-April 1989.

33. Ruth, D. and Ma, H., "On the Derivation of the Forchheimer Equation by Means of the Averaging Theorem," *Transport on Porous Media*, Kluwer Academic Publishers, The Netherlands, 1992, 7, 255-64.

34. Gracki, J. A., Flynn, G. P. and Ross, J., "Viscosity of Nitrogen, Helium, Hydrogen, and Argon from -100 to 25 up to 150-250 atm," *J. Chem. Phys.*, November 1969, 59 (9), 3856-63.

35. Montgomery, R. B., *J. Meteorology*, 1947, 4, 193.

36. Birge, R. T., *Am. J. Phys.*, 1945, 13, 63.

37. Licht, Wm., Jr. and Stechert, D. G., *J. Phys. Chem.*, 1944, 48, 23.

38. Chapman and Cowling, *The Mathematical Theory of Nonuniform Gases*, Cambridge U. Press, London, 1939, 223-5.

39. C. E. Reistle, Jr., *History of Petroleum Engineering*, American Petroleum Institute, New York, 1961, 827 ff.

40. Fancher, G. H; Lewis, J. A; and Barnes, K. B, "Some Physical Characteristics of Oil Sands," *Pennsylvania State College Mineral Industries Experiment Station Bulletin* No. 12, 65, 1933.

41. Wyckoff, R. D; Botset, H. G; Muskat, M; and Reed, D. W., "The Measurement of the Permeability of Porous Media for Homogeneous Fluids," *Review of Scientific Instruments*, 4, 394, 1933,.

42. Lohman, S. W., Ground-Water Hydraulics, *Geological Survey Professional Paper*, 1972, 708, 4-6.

43. *Ground Water*, 31, No. 3, May-June 1993, 519.

44. Jones, S. C., "A Technique for Faster Pulse-Decay Permeability Measurements in Tight Rocks," paper SPE 28450 presented at the 1994 SPE Annual Technical Conference and Exhibition, New Orleans, LA, Sept. 25-28, 907-14.

6.8 APPENDICES

A.6.8.1 Derivation of Equations to Estimate the Maximum Δp for Darcy's Law to Apply

The "condition of Stokes flow," which is a requirement for the legitimate use of Darcy's equation, is met when inertial flow resistance is negligible relative to viscous flow resistance. The purpose of this appendix is to present equations for estimating the maximum allowable Δp to be used in axial flow permeability measurements. If this maximum is not exceeded, Darcy's equation can be safely used in most instances. Equations will first be derived for gases, followed by an equation for the flow of liquids.

A.6.8.1.1 Gases

When Equation 17 (see 6.2.1.2), the differential Forchheimer equation for steady-state flow of gases, corrected for gas slippage, is integrated with respect to length it becomes:

$$\frac{C_1 \Delta p}{L} = \frac{C_2 \mu P_r q_r z_m}{A k_\infty z_r (P_m + f_{Fo} b)} + \frac{C_3 \beta M P_r^{\,2} q_r^{\,2} z_m (P_m + b)}{A^2 R T z_r^{\,2} P_m (P_m + f_{Fo} b)} \quad \text{(A-1)}$$

When the second term on the right hand side is small relative to the first term, f_{Fo} is virtually equal to 1.0. If we assume this value, set all gas deviation factors to 1.0 (which is a good assumption for low pressures), and choose the arithmetic mean pressure, P_m, to be the reference pressure, P_r, for which q_r is calculated, then Equation A-1 simplifies to:

$$\frac{\Delta p}{L} = \frac{C_2 \mu \, v_m}{C_1 k_\infty \left(1 + \frac{b}{P_m}\right)} + \frac{C_3 \beta M P_m v_m^{\,2}}{C_1 R T} \quad \text{(A-2)}$$

where v_m, the Darcy velocity, is equal to q_m / A. This can alternatively be written:

$$\frac{\Delta p}{L} = \frac{C_2 \mu v_m}{C_1 k_\infty \left(1 + \frac{b}{P_m}\right)} \left[1 + \frac{C_3 k_g \beta M P_m v_m}{C_2 \mu R T}\right] \quad \text{(A-3)}$$

In the numerator of the second term inside the brackets, k_g replaces the group $k_\infty(1 + b / P_m)$, per Equation 21. This entire second term is dimensionless, and can be thought of as a "Reynolds number for flow through porous media." Ruth and Ma[33] suggest that this number be called the "Forchheimer number:"

$$N^*_{Fo} = \left(\frac{C_3}{C_2}\right)(k_g \beta \phi)\left(\frac{M P_m}{R T}\right)\left(\frac{v_m}{\phi \mu}\right) \quad \text{(A-4)}$$

The factors on the right hand side of Equation A-4 are grouped into the various components of the Forchheimer number, which is the ratio of inertial to viscous resistance. The first factor contains constants (see Table 6-1) to make the dimensions consistent. The next factor is the characteristic length, analogous to pipe diameter in a Reynolds number for fluid flow through pipes. The next factor can be recognized as gas density at the mean pore pressure. The final factor contains fluid velocity divided by its viscosity. The velocity is obtained by dividing Darcy velocity by rock porosity. Thus it is based upon the average actual area available for flow, not the superficial area. The product of this velocity and gas density is the mass flux of gas through the plug. The porosity that was included in the characteristic length cancels porosity in the final factor.

Unlike pipe flow, where there is a sudden transition from laminar to turbulent flow over a narrow range in the Reynolds number, inertial resistance associated with flow through porous media increases gradually and smoothly as the Forchheimer number increases. This difference can be explained

easily. In laminar pipe flow, fluids are in parallel, streamline flow with virtually no accelerations. Suddenly, at some critical velocity the flow becomes unstable and suffers numerous changes in direction, thus undergoes many accelerations. By contrast, flow through a porous medium is tortuous at any velocity, and involves many accelerations, which increase in magnitude smoothly as mass flux increases. Also, there is a whole spectrum of equivalent pipe sizes in a porous medium. Therefore, sudden transitions from laminar to turbulent flow, with a large increase in the pressure gradient, are not observed.

When the Forchheimer number is zero, all flow resistance is due to viscous shear, and Darcy's law is completely applicable. When it is equal to one, half of the total flow resistance is due to viscous shear. If the Forchheimer number is nine, only 10 percent of the total flow resistance is due to viscous resistance, and the other 90 percent to inertial resistance.

An asterisk is used in Equation A-4 to differentiate the Forchheimer number from the one defined by Equation B-5, which contains k_∞ instead of k_g. It is correct as used in Equations B-6 and B-7, but N^*_{Fo} is needed for the following calculations.

If a Δp value could be found for a core plug such that N^*_{Fo} were equal to 0.005 (i.e., the value inside the brackets in Equation A-3 equals 1.005), then Darcy's equation (or equivalently Equation A-3 with a value of 1.000 inside the brackets) would underestimate k_∞ by only 0.5 percent. We will find this Δp by first setting N^*_{Fo} in Equation A-3 to 0.005, then solving for v_m:

$$v_m = \frac{C_1 k_\infty \left(1 + \frac{b}{P_m}\right)\Delta p}{C_2 \mu L (1.005)} \qquad \text{(A-5)}$$

This and the chosen value of N^*_{Fo} are substituted into Equation A-4, which is solved for Δp, keeping in mind that $P_m = P_2 + \frac{1}{2}\Delta p$:

$$\Delta p_{max} = (P_2 + b)\left(\sqrt{1 + \frac{2C_2^2 RT\mu^2 L(0.005)(1.005)}{C_1 C_3 M k_\infty k_g \beta (P_2 + b)^2}} - 1\right) \qquad \text{(A-6)}$$

Now the task remains to find appropriate values of β (in view of the large amount of scatter in a β vs. k_∞ plot). A slightly conservative approach was taken: values of β lying on the upper dashed curve in Figure 6-3 were divided by 10. This corresponds to a curve that lies above the central solid curve by about a factor of 3 for low values of k_∞, and gradually falls below the central curve at the highest permeabilities. Thus actual β's for the majority of core plugs would be expected to be somewhat less than the values chosen, and the error incurred from calculating permeability using the Darcy equation (provided that Δp_{max} as calculated from Equation A-6 is not exceeded during the permeability measurement) would be less than 0.5 percent. On the other hand, if a plug's actual β happened to lie on the upper dashed line, use of the same Δp_{max} would result in a calculated permeability that is 5 percent low. If the plug's actual β were twice the value shown by the upper dashed curve, the error would be 10 percent, etc.

The calculation of Δp_{max} is not completely straightforward. Equation A-6 contains b, k_g, and k_∞. For purposes of preparing Figure 6-4, b was found from a correlation proposed by Jones[16] for data on a large number of core plugs using helium:

$$b_{He} = 16.4 k_\infty^{-.0382} \qquad \text{(A-7)}$$

where b_{He} is the Klinkenberg gas slippage factor for helium, psi, and k_∞ is the slip-corrected permeability, millidarcys. To calculate b_{air}, the constant 16.4 in Equation A-7 becomes 5.71, using the technique illustrated by Equation 31. Values for C_1, C_2, C_3, and R for desired units are found from Table 6-1.

The required k_{air} is calculated from:

$$k_{air} = k_\infty \left(1 + \frac{b_{air}}{P_2 + \frac{1}{2}\Delta p_{max}}\right) \qquad \text{(A-8)}$$

Because Δp_{max} is unknown, use k_∞ as the first guess for k_g in Equation A-6, and solve for Δp_{max}. Insert this value into Equation A-8 to solve for k_{air}. Now use this value in Equation A-6 for k_g, and iteratively continue to refine the estimates until there is no further change in Δp_{max}. Convergence is usually achieved in 4 to 10 iterations.

Results of these calculations are shown in Figure 6-4 for downstream pressures of 14.7, 50, 100, and 200 psia for air at room temperature. The $\Delta p / L$ values are reported as "psi per inch of core plug length." Although these ratios are slightly dependent upon length, and were calculated for two-inch long plugs, results are close enough for the intended purpose for plugs ranging in length from 1 to 3 inches.

Starting with the highest permeabilities for the 14.7 psia downstream pressure, the maximum allowable pressure drop increases with decreasing permeability, as expected. However, for permeabilities below about 0.3 millidarcys, $\Delta p_{max} / L$ decreases with decreasing permeability. This unexpected result is a consequence of gas slippage. Slippage is greatest at the lowest permeabilities, causing higher gas flow rates than would occur without slippage. It is decreased when the mean pore pressure is increased at the higher back pressures.

At the higher permeabilities, $\Delta p_{max} / L$ values are reduced with increasing back pressure. This is mainly a consequence of increased gas density, which increases mass flux, and consequently inertial resistance, for a given velocity.

A.6.8.1.2 Liquids

Liquids are nearly incompressible and are not subject to slippage. Therefore the derivation for the maximum pressure drop to maintain the condition of Stokes flow is simpler than that for gases. Again, choosing a value of 0.005 for the Forchheimer number and the same beta vs. permeability relationship as for gases, the equation for liquids is:

$$\frac{\Delta p_{max}\rho_L}{L\mu_L^2} = \frac{5.764\text{E}12}{\beta k_L^2} \quad \text{(A-9)}$$

Where:

Δp_{max} = maximum pressure drop for Stokes flow, psi.
L = length of core plug, inches.
μ_L = viscosity of liquid, cp.
ρ_L = density of liquid, g/cm^3.
β = coefficient of inertial resistivity, ft^{-1}.
k_L = permeability of plug to liquid, millidarcys.

For a given β and k_L the left hand side of Equation A-9 applies for a core of any length. Results are plotted in Figure A.6-1. Use of this figure is illustrated by an example: Suppose we determined permeability of a 2.50 inch long core plug with a pressure drop of 35.8 psi and a liquid with viscosity of 1.53 cp. and density of 0.816 g/cm^3. A permeability of 99.8 millidarcys was calculated using Darcy's law. Is it likely that Darcy's law was valid to use?

From Figure A.6-1, a permeability of 99.8 yields a value of about 6.36, which is equal to the left hand side of Equation A-9:

$$\frac{\Delta p_{max}\rho_L}{L\mu^2} = 6.36$$

therefore:

$$\Delta p_{max} = \frac{6.36 \times 2.50 \times 1.53^2}{0.816} = 45.6 \text{ psi}$$

Because the Δp actually used for the measurement, 35.8 psi, was less than the maximum allowable value, we conclude that Darcy's equation probably gave valid results.

B.6.8.2 Calculation of Permeability From Pressure-Falloff, Axial Gas Flow Measurements

The equation for axial flow, used with the pressure falloff method proposed by Jones,[21] starts with the steady-state Forchheimer equation for isothermal axial flow, corrected for gas slippage. It is obtained by integration of Equation 17 (see 6.2.1.2), which yields:

$$\frac{(P_1 - P_2)(P_m + f_{Fo}b)z_r}{P_r q_r z_m} = A_1 + A_2\left(\frac{P_r q_r (P_m + b)}{z_r P_m}\right) \quad \text{(B-1)}$$

Where:

$$A_1 = \frac{C_2\mu L}{C_1 A k_\infty} \quad \text{(B-2)}$$

$$A_2 = \frac{C_3\beta M L}{C_1 A^2 R T} \quad \text{(B-3)}$$

$$A = \frac{\pi D^2}{4} \quad \text{(B-4)}$$

$$N_{Fo} = \frac{q_r P_r A_2}{z_r A_1} \quad \text{(B-5)}$$

$$E = \frac{bN_{Fo}}{1 + N_{Fo}} \quad \text{(B-6)}$$

and

$$f_{Fo} = \frac{\left(1 - \frac{E}{P_1 - P_2}\ln\left[\frac{P_1 + E}{P_2 + E}\right]\right)\left(\frac{P_1 + P_2 + 2E}{P_1 + P_2}\right) + N_{Fo}}{(1 + N_{Fo})} \quad \text{(B-7)}$$

The Forchheimer number, N_{Fo} (Equation B-5), which is the dimensionless ratio of inertial to viscous resistivities, is a "Reynolds number for flow through media." It is discussed more fully in Appendix A. The "Forchheimer interaction factor," f_{Fo} (Equation B-7), is a dimensionless number that has a maximum value of 1.0 (whenever b or N_{Fo} goes to zero). Its minimum value for any practical case is about 0.95.

The instantaneous gas flow rate entering the sample is determined from the total connected reservoir and manifold volume upstream of the inlet core face, V_T, and the pressure-time derivative:

$$q_{1,t} = \frac{V_T f_{z,t}}{P_{1,t}}\left(\frac{-dP_{1,t}}{dt}\right) = V_T f_{z,t}\left(\frac{-d\ln[P_{1,t}]}{dt}\right) \quad \text{(B-8)}$$

Where:

$$f_{z,t} = 1 - \frac{P_{1,t}}{z_{1,t}}\left(\frac{dz_{1,t}}{dP_{1,t}}\right) \quad \text{(B-9)}$$

is a correction for non-ideal gases. The subscript 1 in these equations refers to conditions just upstream of the inlet face of the sample. Because all flow rates, pressures (except atmospheric pressure), and gas deviation factors refer to this location, this subscript will be dropped in equations below. Subscripts n will refer to time, not position.

In general, it is more useful to measure a gauge pressure than the absolute pressure required by Equation B-8. Because the sample is vented directly to atmospheric pressure, the gauge pressure is equal to the Δp across the sample at any instant. If we multiply and divide Equation B-8 by p and realize that $dp = dP$, we obtain:

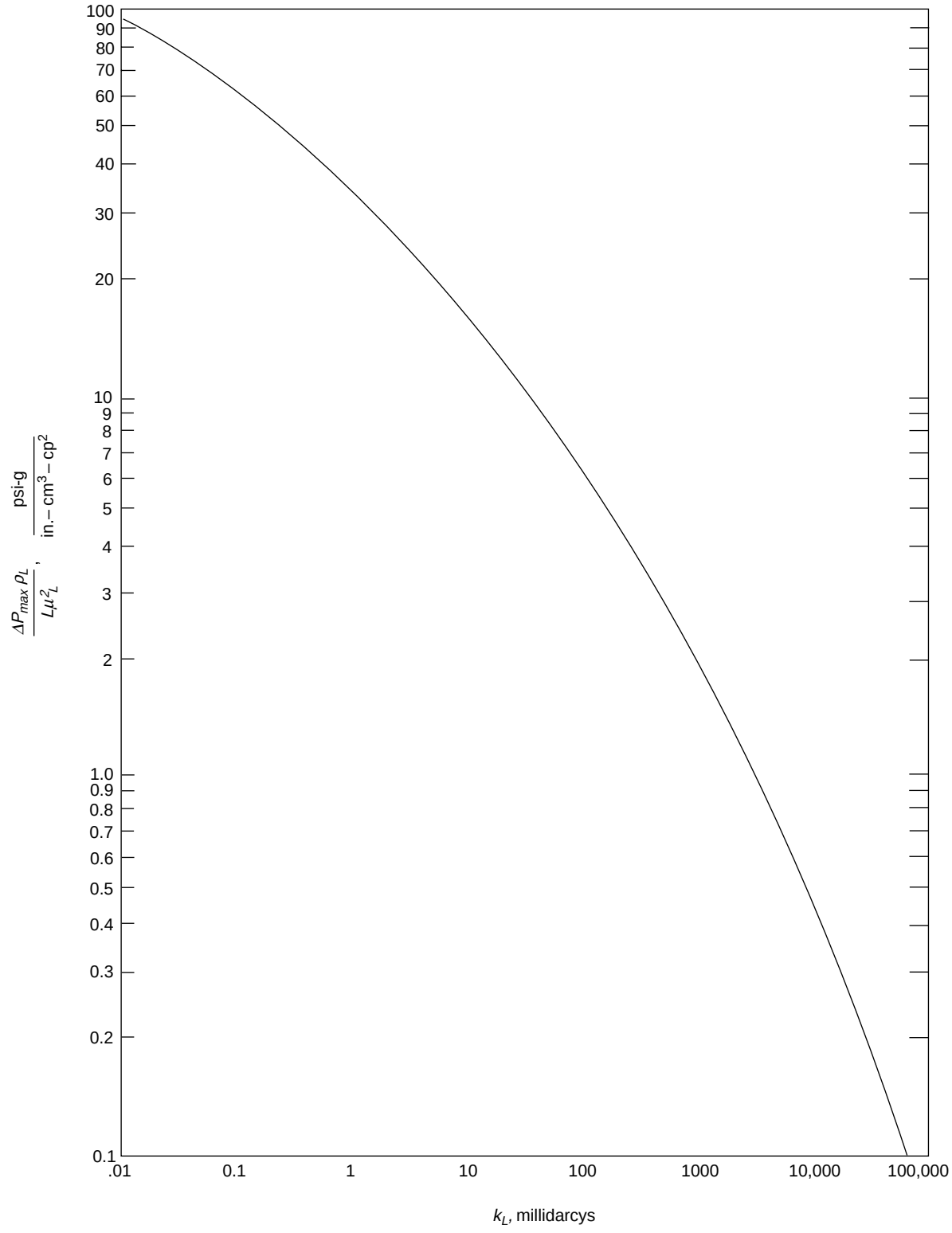


Figure A.6-1—Plot for Determining Maximum Allowable Δp in Axial Flow Liquid Permeability Measurements

$$q = \frac{-pV_T f_z}{pP}\left(\frac{dp}{dt}\right) = \frac{-pV_T f_z}{P}\left(\frac{d\ln p}{dt}\right) \qquad \text{(B-10)}$$

We can now define an instantaneous flow rate function, y:

$$y \equiv -V_T\left(\frac{d\ln p}{dt}\right) = \frac{qP}{f_z p} \qquad \text{(B-11)}$$

The derivative in Equation B-11 is accurately approximated from any pair of adjacent pressure-time points:

$$y_n = \frac{V_t \ln\left[\frac{p_{n-1}}{p_n}\right]}{t_n - t_{n-1}} \qquad \text{(B-12)}$$

This approximation most closely equals the true slope (derivative) of the ln[p] vs. time curve (see Figure B.6-1) at the pressure-midpoint of the interval, which is the geometric mean of the two pressures:

$$p_{g_n} = \sqrt{p_{n-1}p_n} \qquad \text{(B-13)}$$

Thus y_n is calculated for the instant when the upstream pressure is p_{g_n}. The mean pore pressure in the sample at this time is:

$$P_{m_n} = \tfrac{1}{2}p_{g_n} + P_a \qquad \text{(B-14)}$$

Now, except for one complication, any of the several sets of y_n, p_{g_n}, P_{m_n}, and f_{z_n} values obtained from the pressure falloff could be used to calculate the flow variables in Equation B-1. With steady-state flow, where the pressure at any point in the sample is invariant with time, the mass flow rate is constant throughout the length of the sample. The complication is that this is not true for unsteady-state flow.

Whenever pressure at any point in the sample decreases with time, gas density there decreases proportionally, with a resultant mass-depletion of gas. Because the upstream pressure decreases continually during a pressure falloff, the pressure throughout the length of the sample also decreases, except at the outlet end, which always remains at atmospheric pressure. The rate of mass depletion of resident gas in the sample is accompanied by an equal increase in the mass rate of flowing gas. Therefore, at any given time during a transient pressure decline, the mass flow rate of gas leaving a sample is

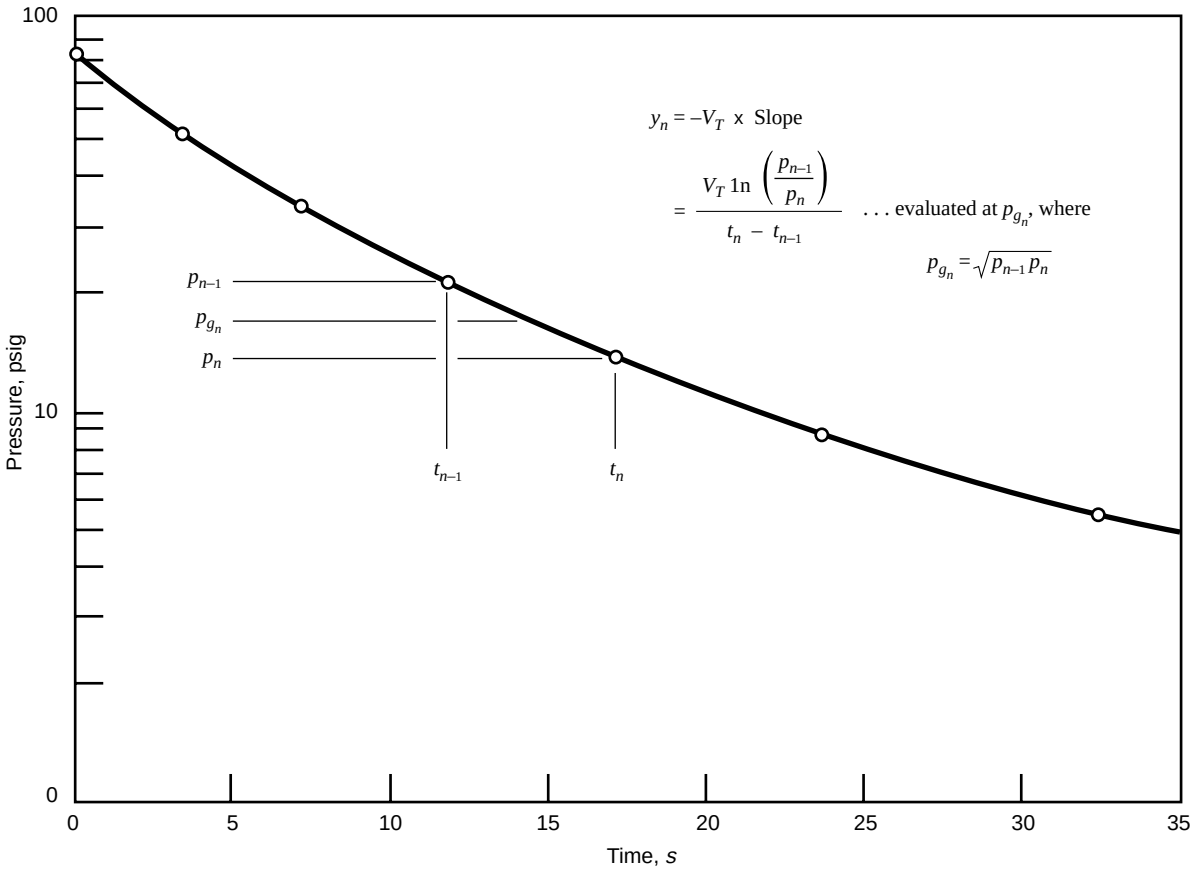


Figure B.6-1—Construction of Flow Variables y_n and p_{gn}

greater than the mass rate entering the sample. If the gas reservoir volume, V_T is large compared to the pore volume of the sample, V_p, then the relative increase is small. If these volumes are comparable, the increase is significant. In the limit, an infinite reservoir volume yields steady-state flow—no increase. At the opposite extreme, when the reservoir volume is zero, all the gas exiting from the sample originates from within the sample itself.

Because of the non-constant mass flux throughout the length of a sample at any instant during the pressure transient, we cannot use the steady-state solution directly. The correct solution can be achieved, however, by an iterative technique in which the continuity equation is decoupled from the flow equation, but where both equations are ultimately satisfied within an acceptable tolerance: the partial derivative of gas density with respect to time at any length, taken from the steady-state solution of the flow equation, is inserted into the continuity equation from which the approximate mass flux variation with length is derived for several conditions of pressure, slip-factor magnitude, and ratios of pore volume to reservoir volume. The corrected, non-constant mass flux is inserted into the differential form of the flow equation, which is then re-integrated with respect to length. The process is repeated until any further change is negligible. Four iterations were adequate for all practical cases. The corrected flow rate that can be inserted into the integrated flow equation to allow for the weighted average increase in mass flow with length at any instant is:

$$q_{c_n} = q_n(1 + \gamma G_m[c_n, \gamma]) \qquad \text{(B-15)}$$

Where:

$$\gamma = \frac{2V_p}{3V_T} \qquad \text{(B-16)}$$

and

$$c_n = \frac{(P_a + b)^2}{2p_{g_n}(P_{m_n} + b)} \qquad \text{(B-17)}$$

The correction factors for non-constant gas mass flow, $G_m[c_n, \gamma]$, are shown as a function of c for several values of γ in Figure B6-2. We can now define a "corrected instantaneous flow rate function," γ_{c_n}:

$$y_{c_n} \equiv \frac{y_n f_{z_n}}{z_n}(1 + \gamma G_m[c_n, \gamma]) = \left(\frac{q_r P_r}{z_r p_g}\right) \qquad \text{(B-18)}$$

where f_{z_n} (Equation B-9) is evaluated at pressure P_n, where $P_n = p_{g_n} + P_a$. This function can be inserted into Equation B-1 to produce the integrated form of the slip-corrected Forchheimer equation for transient flow:

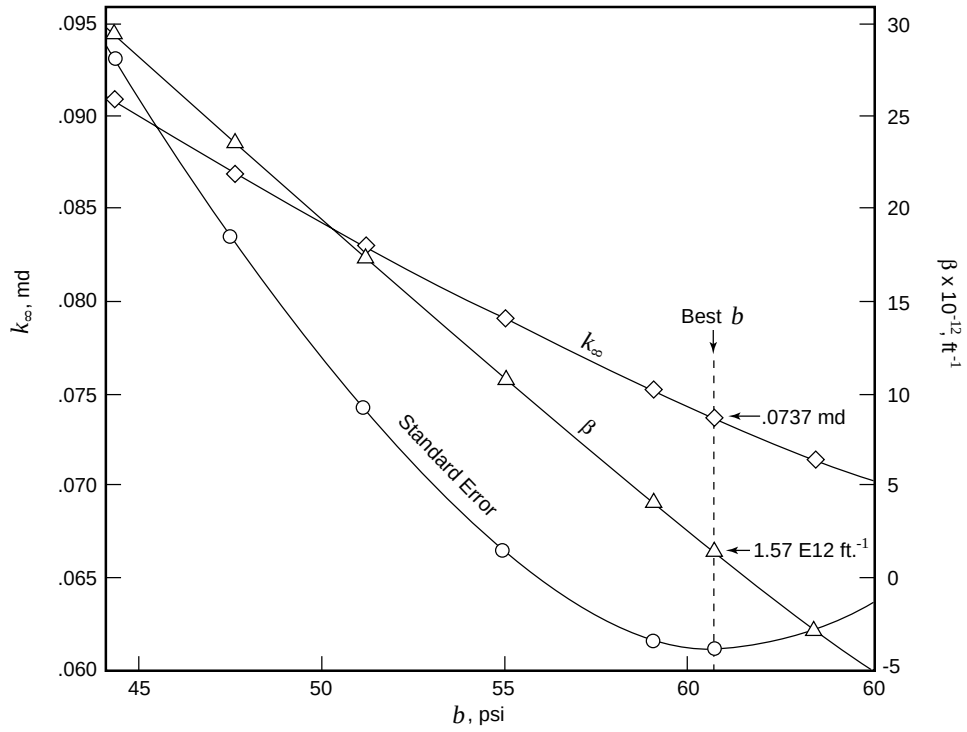


Figure B.6-2—Dimensionless Correction Factors for Non-Constant Mass Flux in Pressure-Falloff Permeability Measurements

$$\left(\frac{P_m + f_{Fo} b}{y_c z_m}\right)_n = A_1 + A_2\left(\frac{y_c p_g (P_m + b)}{P_m}\right)_n \qquad \text{(B-19)}$$

The Forchheimer number (Equation B-5) must be calculated for each f_{Fo_n} in the equation above. In terms of the corrected flow function, this is:

$$N_{Fo_n} = \frac{y_{c_n} p_{g_n} A_2}{A_1} \qquad \text{(B-20)}$$

E_n (Equation B-6) must also be calculated for each data point obtained in order to calculate f_{Fo_n} (Equation B-7).

The coefficients A_1 and A_2 in Equation B-19, which yield k_∞ and β, respectively, are determined from the set of equations corresponding to each data point, n, using linear regression as follows:

1. Calculate an initial guess for the slip factor, *b*. For helium, this is:

$$b[\text{first guess}] = 0.803\left(\frac{D^2}{\mu_{He} L y_{c_m}}\right)^{0.467} \qquad \text{(B-21)}$$

where y_{c_m} is the flow function that corresponds to the lowest p_g value obtained.
2. Set f_{Fo} for all *n*.
3. Calculate c_n (Equation B-17) and y_{c_n} (Equations B-12 and B-18) for each *n*.
4. Calculate the left-hand and right-hand terms in Equation B-19 for each *n*.
5. Perform a linear regression to obtain A_1 and A_2.
6. From these values, calculate N_{Fo} (Equation B-20), *E* (Equation B-6), and f_{Fo} (Equation B-7) for each *n*.
7. Repeat steps 4 through 6 until the change in any f_{Fo} does not exceed 0.001 from its value in the previous iteration. Three iterations are usually adequate.
8. Calculate a Standard Error, SE, from the last linear regression.
9. Choose a new *b* that is 10 percent higher than the first-guessed *b*.
10. Using the most recently calculated set of values for f_{Fo} and the new *b*, repeat steps 3 through 8.
11. If the new SE is lower than the previous one, increase *b* by 10 percent. Otherwise, reduce the original *b* by 10 percent. Repeat step 10.
12. Continue to increase or decrease *b*, repeating step 10, until the minimum SE is found. This corresponds to the best values of *b*, A_1 and A_2, in a least-squares sense. Calculate k_∞ and β from A_1 and A_2 (Equations B-2 and B-3), respectively.

Typical changes in k_∞, β, and SE with *b* are illustrated by Figure B.6-3. (The scale for the SE has been omitted for clarity.) As *b* increases, calculated values of both k_∞ and β decrease. The correct *b* for this example, which occurs at the minimum SE, is 60.6 psi. The calculated value of β becomes

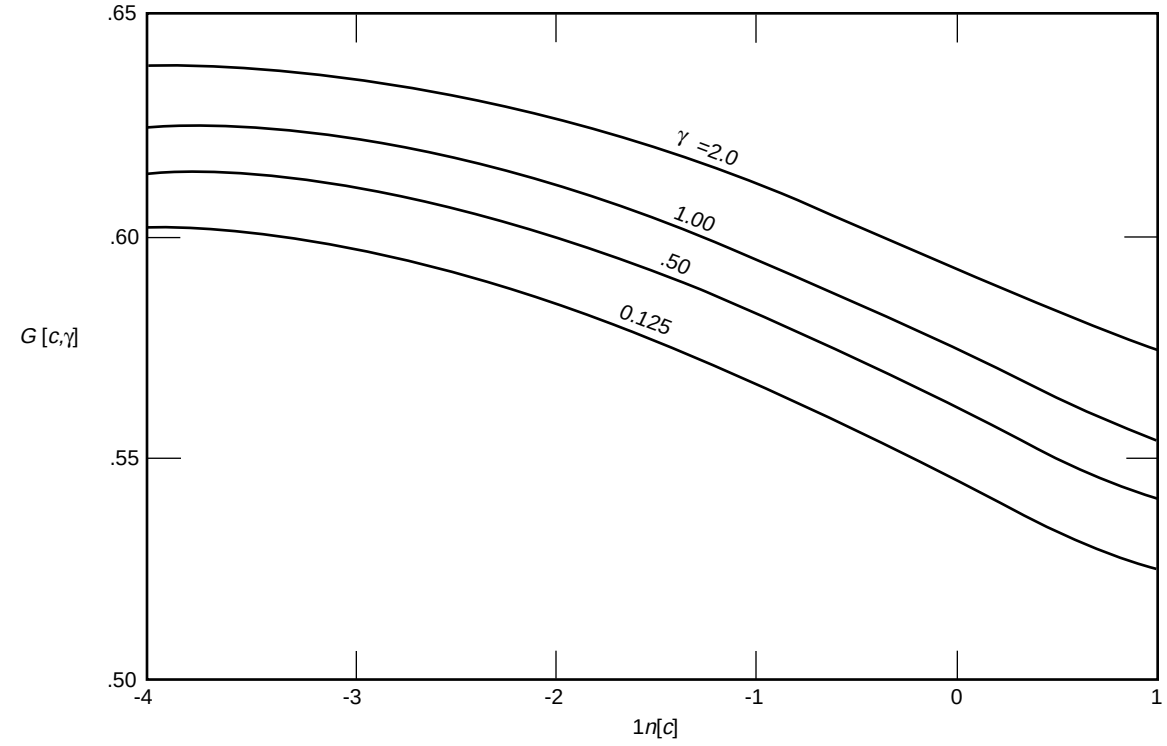


Figure B.6-3—Variation of k_∞, ß, and Standard Error with *b* for Pressure-Falloff Data

zero at a slip factor of 61.5 psi, and is negative for larger *b*s. A negative calculated β usually indicates that b is too high. However, extreme sensitivity of permeability to small changes in net stress can also cause negative βs. Changes in k_∞ and β with b, as shown in Figure B.6-3, are general, in the sense that they always decrease with increases in b.

The computational effort for the pressure falloff technique is considerably greater than for steady-state calculations. However, the entire procedure outlined above, with 25 y and p_g values each, is accomplished nearly instantaneously with a small computer.

Low-permeability rocks at small pressure differentials often exhibit little inertial resistance. In these cases, the term in Equation B-19 that is multiplied by A_2 is small, f_{Fo} is nearly equal to 1, and Equation B-19 can be approximated by:

$$y_{c_n} z_{m_n} = \frac{1}{A_1}(P_{m_n} + b) \tag{B-22}$$

For the approximation to be valid, a plot of $y_{c_n} z_{m_n}$ vs. P_{m_n} must be linear. Equation B-22 is the Darcy equation, corrected for gas slippage, for pressure-falloff measurements. Its slope and intercept are equal to $1 / A_1$ and b / A_1, respectively. Therefore k_∞ and b are calculated from:

$$k_\infty = \frac{C_2 \mu L(\text{slope})}{C_1 A} \tag{B-23}$$

and

$$b = \frac{(\text{intercept})}{(\text{slope})} \tag{B-24}$$

Departure from linearity, especially at the higher P_{m_n} values, indicates that inertial resistance is not negligible at these conditions.

C.6.8.3 Calculation of Permeability From Pressure-Falloff, Probe Gas Flow Measurements

Instantaneous flow rate functions, y_n, and geometric mean pressures, p_{g_n}, are calculated from Equations B-12 and B-13, respectively. These are inserted into:

$$\frac{\frac{1}{2}p_{g_n} + P_a + b^*}{y_n} = A_3 + A_4\left(\frac{y_n p_{g_n}(\frac{1}{2}p_{g_n} + P_a + b^*)}{\frac{1}{2}p_{g_n} + P_a}\right) \tag{C-1}$$

Where:

$$A_3 = \frac{C_2 \mu}{C_1 (G_0 r_i) k_\infty} \tag{C-2}$$

and

$$A_4 = \frac{C_3 \beta M}{C_1 G_{f_{fo}} RT} \tag{C-3}$$

The dimensional geometric factor for the Forchheimer term above, $G_{f_{Fo}}$, must be determined by numerical modeling. If this geometric factor can be determined, then β could be calculated from Equation C-3. Regardless, use of Equation C-1 corrects for inertial resistance, whether or not β can be calculated.

Note that except for the different geometrical factors contained in A_3 and A_4, Equation C-1 is virtually identical to Equation B-19 (for axial flow), except for certain simplifications in the probe-permeameter equations. These simplifications are: (a) gas deviation (z) and Forchheimer interaction (f_{Fo}) factors are omitted. Pressures are so low that these factors are nearly unity, and (b) y_n values are not corrected for non-constant (spatial) mass flow. With commonly used probe-seal dimensions, the rock pore volume that influences permeability measurement is so small, relative to the volume of even the smallest gas reservoir, that the steady-state assumption of constant mass flow through any isopotential surface at any instant does not introduce any appreciable error in the calculation of permeability. For example, the error with an interior probe seal radius of 3.8 millimeters, and reservoir volume of 5.2 cm^3 is less than 0.1 percent.

The b^* that appears in Equation C-1 is a gas slippage factor taken from a correlation. Although b could theoretically be determined directly from the pressure falloff data, the range of mean pore pressures measured (with a fill pressure of 10 psig or less) is too small to determine b reliably. The correlation b^* is obtained using an iterative technique. Its first approximation (for nitrogen, in psi) is calculated from:

$$b^*_{N_2} = 0.22\sqrt{\frac{(G_0 r_i)}{\mu_{N_2} y_m}} \tag{C-4}$$

where y_m corresponds to the lowest value of p_g obtained from the pressure-falloff data. This first-guess value of b^* is used in Equation C-1 to calculate its left and right hand terms for each y_n and p_{g_n} value obtained. Next, the coefficients A_3 and A_4 are found from Equation C-1 using linear regression. The first estimate of the Klinkenberg permeability is obtained from rearrangement of Equation C-2:

$$k_\infty = \frac{C_2 \mu}{C_1 (G_0 r_i) A_3} \tag{C-5}$$

Using this k_∞, an improved estimate of $b^*_{N_2}$ is calculated from the correlation:

$$b^*_{N_2} = 6.9 k_\infty^{-0.382} \tag{C-6}$$

This new value of b^* is reinserted into Equation C-1, and the process is repeated until the change in b^*, from one iteration to the next, is less than 0.1 psi. Convergence should be obtained within 2 to 4 iterations.

Inertial resistance virtually disappears for samples with permeabilities of less than 0.1 millidarcy for starting pressures of less than 10 psig. For these samples, the A_4 term in Equation C-1 can be ignored, and the Forchheimer equation reduces to the slip-corrected Darcy equation:

$$k_\infty = \frac{C_2 \mu y^*}{C_1 (G_0 r_i)(\frac{1}{2} p_g^* + P_a + b^*)} \qquad \text{(C-7)}$$

This equation can be used when as few as one to three $y - p_g$ points are obtained for low permeability samples. The y^* and p_g^* refer to any one of these points. The calculation is again iterative. The initial estimate of b^* is calculated from Equation C-4. The estimate is refined by calculating k_∞ (Equation C-7), which is then used in Equation C-6. The process is continued until the change in b^*, from one iteration to the next, is less than 0.1 psi. Convergence is usually achieved in 2 to 4 iterations.

Gas permeability, k_g, for any desired gas at any mean pore pressure can be calculated from k_∞ and b^* as outlined in 6.4.1.1.5 for the axial flow case.

D.6.8.4 Calculation of Permeability From Pulse Decay, Axial Gas Flow Measurements

The initial pressure difference across the sample, $\Delta p[0]$, which is slightly less than Δp_1 (see 6.4.1.3.2), must be calculated from a gas mass balance:

$$\Delta p[0] = \Delta p_1 \left(\frac{V_1 - V_d}{V_1} \right) \qquad \text{(D-1)}$$

V_1, the total upstream volume, includes: internal volumes of the upstream reservoir; the upstream chamber of the differential pressure transducer; connecting lines to the transducer and valves 1, 2, and the fill valve; and the upstream dead volume, V_d, (the volume inside valve 1); the upstream end plug, and the line connecting them. The total downstream volume, V_2, includes the volumes in the downstream reservoir, transducer, and end plug; the downstream chamber of the differential transducer; and the lines connecting them, including the line to valve 2. The volumes V_1, V_2, and V_d must all be found from careful calibration using Boyle's Law techniques (see 6.6.3.1).

The differential pressure and downstream pressure are monitored as functions of time. Data are analyzed from the solution of the diffusivity equation, that is derived by combining the differential form of Darcy's Law with the continuity equation. The sample is presumed to be initially at a uniform pore pressure throughout. Then at time $t = 0$, a pulse of slightly higher pressure, $\Delta p[0]$, is applied to its upstream end from the upstream reservoir. As gas flows from V_1 into the sample, the pressure in V_1 declines. The pressure in V_2 remains constant for a short period of time until the pressure pulse has traversed the length of the sample. Then the pressure in V_2 rises. Because $P_1[t]$ declines and $P_2[t]$ rises, $\Delta p[t]$ continues to diminish and gradually approaches zero as the upstream and downstream pressures become equal. The rate of pressure decay depends on permeability: the lower the permeability the slower the decay. The general solution for the pressure difference as a function of time as presented by Dicker and Smits,[23] following the original work of Brace et al.,[24] the error-function solution of Bourbie and Walls,[25] the general analytical solution of Hsieh et al.,[26] Chen and Stagg,[27] Haskett et al.,[28] and others is:

$$\frac{\Delta(P[t])^2}{\Delta(P[0])^2} = 2\sum_{m=1}^{\infty} \frac{a(b^2 + \theta_m^2) - (-1)^m b\sqrt{(a^2 + \theta_m^2)(b^2 + \theta_m^2)}}{\theta_m^2(\theta_m^2 + a + a^2 + b + b^2) + ab(a + b + ab)} \cdot \exp[-\theta_m^2 t_D] \qquad \text{(D-2)}$$

Where θ_m are roots of the equation:

$$\tan\theta = \frac{(a+b)\theta}{\theta^2 - ab} \qquad \text{(D-3)}$$

a, the ratio of the compressive storage (the product of volume and compressibility) of the sample's pore volume to that of the upstream reservoir, is:

$$a = \frac{V_P(c_g + c_{pv})}{V_1(c_g + c_{v_1})} \qquad \text{(D-4)}$$

b, the ratio of the compressive storage of the sample's pore volume to that of the downstream reservoir, is:

$$b = \frac{V_P(c_g + c_{pv})}{V_2(c_g + c_{v_2})} \qquad \text{(D-5)}$$

and t_D, dimensionless time, is:

$$t_D = \frac{C_1 k_g t}{C_2 \mu \phi (c_g + c_{pv}) L^2} \qquad \text{(D-6)}$$

where C_1 and C_2 are conversion constants, given in Table 6-1, required to make t_D dimensionless. The c_g, c_{pv}, c_{v_1}, and c_{v_2} are compressibilities of gas, the sample's pore volume, and upstream and downstream reservoirs, respectively. Gas compressibility is:

$$c_g = \frac{1}{P}\left(1 - \frac{d\ln[z]}{d\ln[P]}\right) \equiv \frac{f_z}{P} \qquad \text{(D-7)}$$

In Equation D-7, we have defined f_z as the value of the group within parentheses that accounts for deviation from ideal gas behavior. At pressures of interest, this deviation is not negligible. Values of f_z for nitrogen at 72°F are given by Jones.[44] If the system is built rigidly, i.e., with thick-walled vessels, and if displacement volumes of the transducers are

small relative to V_1 and V_2, then c_{V_1} and c_{V_2} are small compared to c_g, and can be ignored. Pore-volume compressibility varies considerably among samples of different lithologies. However, it is minimized by using high net confining stresses, and is usually less than 2×10^{-5} psi^{-1} for net stresses of 5,000 psi or more. A c_{pv} of 1×10^{-5} psi^{-1} is equal to 1 percent of the gas compressibility at a gas pressure of 1000 psia, or 2 percent at 2,000 psia.

The squared pressure differences (that apply to gases only) that appear on the left hand side of Equation D-2 are:

$$\frac{\Delta(P[t])^2}{\Delta(P[0])^2} \equiv \frac{P_1[t]^2 - P_2[t]^2}{P_1[0]^2 - P_2[0]^2} = \frac{\Delta p[t](P_2[t] + \frac{1}{2}\Delta p[t])}{\Delta p[0](P_2[0] + \frac{1}{2}\Delta p[0])} \quad \text{(D-8)}$$

Equation D-2 applies for all values of a, b, and t_D, provided that all roots of Equation D-3 are used, and all compressibilities are constant. Haskett et al.[28] minimize the effect of the restriction by incorporating time-varying compressibility into an "adjusted pseudo time." However, with the proper experimental conditions, neither of the restrictions poses a serious problem, as will be discussed below. The use of the squared pressure differences in Equation D-2 is nearly equivalent to using pseudo-pressures as suggested by Haskett et al. Because the change in the mean pore pressure is so small in the technique outlined here, changes in the *z*-factor and gas viscosity are small. Their ratios between the numerator and denominator of Equation D-9 are virtually unity. For experimental conditions other than outlined below, where variations in the mean pore pressure are greater, the use of pseudo-pressures is recommended.

The choice of reservoir sizes is not limited directly by Equation D-2. Some investigators have used large upstream vessels ($a \cong 0$) or large downstream vessels ($b \cong 0$). Others have advocated reservoir volumes that are similar in magnitude to the pore volume of the sample (a or $b \cong 0.2$ to 5). In general, to keep measurement times reasonably short with low-permeability samples, at least one of the reservoirs must be small.

Dicker and Smits[23] present compelling reasons to make the upstream and downstream volumes identical and fairly small. The equal volumes create a desirable situation of symmetry. Following the period when the pressure pulse traverses the length of the sample, the pressure decrease in the upstream vessel is offset by an equal downstream pressure increase, thereby keeping the mean pore pressure constant and reducing the overall pore volume compressional changes nearly to zero. Because the average pressure in the upstream half (of a homogeneous sample) decreases with time, while the average pressure in the downstream half increases by the same amount, the mass flow rate of gas entering the sample is equal to that leaving the sample at the same instant. The maximum rate at any instant occurs about midway along the length of a reasonably homogeneous sample.

Another consequence of symmetry (where $a = b$) is that all even roots of Equation D-3 cancel out in Equation D-2. The first root of Equation D-3, θ_1, is slightly less than the square root of $(a + b)$ for small values of a and b (large reservoirs). If we define f_1 as:

$$f_1 \equiv \frac{\theta_1^2}{a + b} \quad \text{(D-9)}$$

then $f_1 = 1.000$ when $a + b = 0$, and gradually decreases as $a + b$ becomes larger, as illustrated in Figure D6-1. In this figure, V_s is the smaller of V_1 or V_2, and V_L is the larger of the two reservoir volumes.

The square of the first root of Equation D-3 is found from:

$$\theta_1^2 = f_1(a + b) \quad \text{(D-10)}$$

The higher roots of Equation D-3 are equal to:

$$\theta_m = (m - 1)\pi, \text{ for } (a + b) = 0 \quad \text{(D-11)}$$

and increase gradually as $(a + b)$ increases.

Except for early times when t_D is small, only the first term in the summation (Equation D-2) is significant. The second term is zero when a equals b. At a t_D of 0.1, all higher terms add only 0.16 percent to the contribution of the first term when $a = b = 1$, and less for smaller values of a and b. Therefore, if early times are not used, Equation D-2 reduces to a single exponential, which can be written as:

$$\ln\frac{\Delta(P[t])^2}{\Delta(P[0])^2} = \ln f_0 - \left(\frac{\theta_1^2 k_g}{(c_g - c_{pv})\mu\phi L^2}\right)t \quad \text{(D-12)}$$

Where:

$$f_0 = \frac{2[a(b^2 + \theta_1^2) + b\sqrt{(a^2 + \theta_1^2)(b^2 + \theta_1^2)}]}{\theta_1^2(\theta_1^2 + a + a^2 + b + b^2) + ab(a + b + ab)} \quad \text{(D-13)}$$

Thus, if experimentally obtained values of $\ln[\Delta(P[t])^2 / \Delta(P[0])^2]$ were plotted vs. time, a straight line would result (except for early times), which has an intercept of $\ln[f_0]$, and a negative slope of absolute value:

$$|slope| = \frac{C_1\theta_1^2 k_g}{C_2(c_g + c_{pv})\mu\phi L^2} = \frac{C_1 f_1(a + b)k_g}{C_2(c_g + c_{pv})\mu\phi L^2} \quad \text{(D-14)}$$

where C_1 and C_2 are constants from Table 6-1, used to make the units consistent. If c_{v_1} and c_{v_2} are negligible compared to c_g, then:

$$|slope| = \frac{C_1 f_1 A k_g P_m}{C_2 \mu L f_z}\left(\frac{1}{V_1} + \frac{1}{V_2}\right) \quad \text{(D-15)}$$

The mean pore pressure in Equation D-15, P_m, is an absolute pressure. When $a = b$, and the rock sample is homogeneous (or if a and b are small, e.g., less than 0.3), P_m should not change during the linear portion of the semi-log decay. It is calculated from:

$$P_m[t] = P_2[t] + \tfrac{1}{2}\Delta p[t] \qquad \text{(D-16)}$$

Calculation of the slope can be performed during the measurement, using the latest two time-pressure readings:

$$|slope| = \frac{\ln\left(\dfrac{\Delta p[t_{n-1}](P_2[t_{n-1}] + \frac{1}{2}\Delta p[t_{n-1}])}{\Delta p[t_n](P_2[t_n] + \frac{1}{2}\Delta p[t_n])}\right)}{(t_n - t_{n-1})} \qquad \text{(D-17)}$$

The absolute value of this slope will be high initially (if $a \cong b$), and will decrease fairly quickly, becoming constant with increasing time. After the slope has become constant for several readings, the intercept and slope of the linear portion of the decay can be obtained by linear regression of:

$$\ln\left(\frac{\Delta p[t_n]P_m[t_n]}{\Delta p[0]P_m[0]}\right) = A_0 + A_1 t_n \qquad \text{(D-18)}$$

where A_0 is the intercept and A_1 is the slope. Only data points yielding the constant slope (from Equation D-17) are to be used in the regression. From Equation D-12, we see that f_o can be obtained from the intercept:

$$f_0 = \exp[A_0] \qquad \text{(D-19)}$$

Equation D-13 shows that f_0 is a function of a and b, as is θ_1. Remarkably, when $a = b$, f_1 is only slightly greater than f_0: 0.0055 percent for $a = 0.1$; 0.12 percent for $a = 0.5$; and 0.44 percent for $a = 1.0$. Values of θ_1^2, f_1, and f_0 are listed in Table D.6-1 for various values of a, when $a = b$. These can be calculated from Equations D-3, D-10, and D-13 when the reservoirs are not of equal volume (more precisely, of equal compressive storage).

Figure D.6-2 shows f_1 as a function of f_0 for different ratios of V_2 / V_1. Because V_1 and V_2 are both determined by calibration, f_1 can be determined uniquely from f_0, and permeability calculated from:

$$k_g = \frac{-A_2 C_2 \mu L f_z}{C_1 f_1 A P_m\left(\dfrac{1}{V_1} + \dfrac{1}{V_2}\right)} \qquad \text{(D-20)}$$

Using the value of f_0 obtained, the pore volume of the sample can be estimated from:

$$V_p = \frac{aV_1}{1 + P_m c_{pv} / f_z} \qquad \text{(D-21)}$$

where a is found from Table 6-10, or from Figure D.6-1. If c_{pv} is estimated to equal 8×10^{-6} psi^{-1}, which may be too high or too low by a factor of 2 to 4 compared to the true value at a high net effective stress (4,000 to 5,000 psi), the maximum error from the assumption is about 1.6 percent for $P_m = 1000$ psia, or 3.2 percent for $P_m = 2{,}000$ psia. At lower net stresses, c_{pv} can be expected to be higher and more variable.

The differential pressure transducer should be electrically or mathematically zeroed just before valve 2 is closed (when the true differential pressure is zero at $P_2[0]$). If the transducer maintains its linearity, even though its gain or sensitivity may shift slightly in going from a low to a higher reference pressure (i.e., when P_2 is raised from atmospheric pressure to 1,000-2,000 psia), no error is incurred (except in the calculation of P_m) because all the Δp values are relative; they occur as ratios.

Table D.6-1—Pulse Decay Constants When $a = b$ ($V_1 = V_2$, or $S_1 = S_2$)

$a = b =$	θ_1^2	$f_1 = \theta_1^2/(a + b)$	$f_0 = \exp[\text{intercept}]$	f_1 / f_0
0.0	.000000	1.000000	1.000000	1.000000
0.1	.196711	.983553	.983500	1.000055
0.2	.387016	.967539	.967334	1.000212
0.3	.571166	.951944	.951500	1.000466
0.4	.749404	.936755	.935999	1.000808
0.5	.921963	.921963	.920826	1.001235
0.6	1.089064	.907553	.905980	1.001737
0.7	1.250923	.893516	.891458	1.002309
0.8	1.407744	.879840	.877255	1.002947
0.9	1.559725	.866514	.863366	1.003646
1.0	1.707053	.853527	.849789	1.004399
1.1	1.849910	.840868	.836516	1.005202
1.2	1.988469	.828529	.823544	1.006053
1.3	2.122896	.816499	.810866	1.006947
1.4	2.253350	.804768	.798477	1.007878

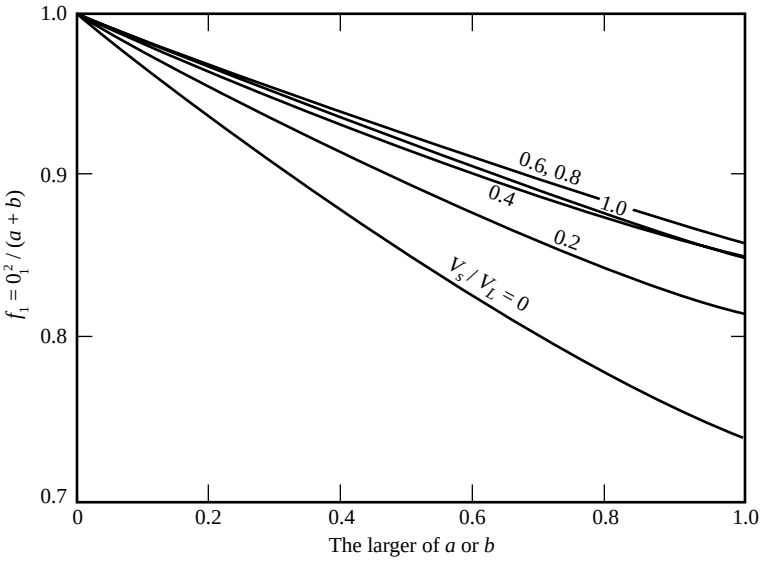


Figure D.6-1—First Root Relationship for Pulse-Decay Equation

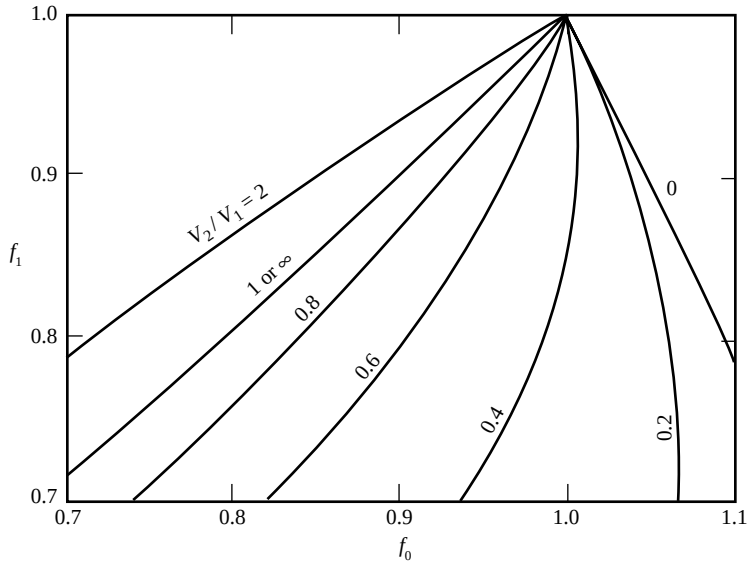


Figure D.6-2—Pulse-Decay Plot for Finding f_1, from f_0 with Known Ratios of V_2 / V_1 (for Gases) or S_2 / S_1 (for Liquids)

The practical range of permeability for gas pulse-decay measurements is about 0.1 millidarcy to 0.01 microdarcy (1 E-4 to 1 E-8 μm^2). The pulse-decay time is nearly proportional to sample length and inversely proportional to permeability and mean pore pressure. The time also depends strongly on the how much of the total pulse decay is measured.

As a general guideline, the reservoir volumes, V_1 and V_2, should be at least as large as the largest pore volume anticipated, and thereby 2-10 times the pore volume of the majority of samples to be run. The onset of the linear semi-log portion of the pulse decay occurs at about the same elapsed time regardless of the *PV*-reservoir volume ratio. The work of Kamath et al.[29] shows that the sensitivity to permeability heterogeneity is also reduced as *a* and *b* become smaller (larger reservoirs).

The upper permeability limit can be extended by increasing the reservoir volumes. However the sensitivity (thus the accuracy) of the pore volume determination (Equation D-21) is reduced with larger volumes.

Perhaps the most time-consuming part of this technique is allowing the system to reach true pressure equilibrium prior to generation of the pressure pulse. As pressures become uniform everywhere in the system, driving forces approach zero. If the pore volume of a sample is determined independently, the pressure equilibration step can be eliminated as suggested by Jones.[44]

E.6.8.5 Calculation of Permeability From Pulse Decay, Axial Liquid Flow Measurements

E.6.8.5.1 Apparatus and Measurement of Compressive Storage

One convenient method of measuring compressive storages in apparatus for liquid pulse decay measurements is to include a needle valve with a micrometer screw (as illustrated in Figure 6-25), which has a total calibrated displacement volume (cross-sectional area of the plunger at the stem seal multiplied by the length of its available stroke) of about 0.02 to 0.05 cm^3. Valves 1 and 2 should be of the type that have no internal volume change when opened or closed. To measure the storages, load a solid steel cylinder into the sample holder and apply confining stress. With valves 1 and 2 open, apply a vacuum to the fill/vacuum valve to remove all air from the apparatus. Then fill the entire system with degassed liquid to about 80 percent of the full scale pressure of the downstream pressure transducer. After the pressure has stabilized, close valves 1 and 2. The needle valve, valve 3, should be near fully-open. The differential pressure transducer should read zero, and the p_2 reading should not change when valves 1 and 2 are closed. Record the micrometer reading. Then screw in the micrometer, decreasing the upstream system volume until the differential pressure transducer reads full scale, *e.g.*, 100 psi. This pressure rise from the initial zero reading is Δp_1, and the volume decrease, calculated from the micrometer readings, is ΔV_1.

Because the volume between valve 1 and the upstream end of the steel plug in the sample holder (the "upstream dead volume") was excluded from the compression measurement, the "upstream compressive storage," S_1, less the "dead volume compressive storage," S_d, is calculated from:

$$S_1 - S_d = \frac{\Delta V_1}{\Delta p_1} \tag{E-1}$$

To determine S_d, open valve 1. After the differential pressure reading stabilizes to Δp_2, calculate the storage for the upstream dead volume from:

$$S_d = \Delta V_1 \left(\frac{1}{\Delta p_2} - \frac{1}{\Delta p_1} \right) \tag{E-2}$$

and the upstream compressive storage from:

$$S_1 = \frac{\Delta V_1}{\Delta p_2} \tag{E-3}$$

To determine the downstream storage, first unscrew the needle valve just past—then back to—the initial reading. As a check, the differential transducer should again read zero, and the downstream transducer should display its initial reading. Open valve 2. Read the downstream transducer pressure, $p_2[1]$. Screw in the needle valve until the stabilized downstream pressure, $p_2[2]$, is nearly equal to the full-scale reading of this transducer. Calculate the volume decrease, ΔV_2, from the micrometer readings, and the downstream compressive storage from:

$$S_2 = \frac{\Delta V_2}{p_2[2] - p_2[1]} - \frac{\Delta V_1}{\Delta p_1} - S_d \tag{E-4}$$

These calibrations of compressive storage must be performed for each different liquid used.

E.6.8.5.2 Procedure for Permeability Measurement

For a pulse decay liquid permeability measurement, a completely liquid-saturated rock sample is loaded into the sample holder, which is then pressurized, preferably to a fairly high (usually hydrostatic) confining stress. The system is filled with liquid, as described above, and pressurized to the initial pore pressure. The fill valve is left open long enough to allow the high pressure liquid to diffuse into the sample. With valves 1 and 2 still open, the fill valve is closed, and the downstream transducer is monitored until no further change in pressure is observed. Then valves 1 and 2 are closed and the pressure pulse is generated by screwing in the needle valve. After all pressures have stabilized, $S_1 - S_d$ is calculated from Equation E-1. This should have the same value as that obtained from the calibration.

The pulse decay is initiated by opening valve 1. The initial pressure difference across the sample, $\Delta p[0]$, will be slightly less than $\Delta p[1]$, which was generated with the needle valve with valve 1 closed, thereby excluding the upstream dead volume from the upstream volume. Therefore, $\Delta p[0]$ must be calculated, using the $\Delta p[1]$ and ΔV_1 that correspond to the run-time pressure-pulse generation, and the S_d that was determined from the calibration:

$$\Delta p[0] = \frac{1}{\frac{S_d}{\Delta V_1} - \frac{1}{\Delta p_1}} \tag{E-5}$$

E.6.8.5.3 Pulse Decay Calculations

Equations D-2 and D-3 apply to liquid, as well as gas, pulse decay measurements. However, the squared pressure differences on the left hand side of Equation D-2 must be replaced by first-power pressure differences for liquids:

$$\frac{\Delta p[t]}{\Delta p[0]} = 2\sum_{m=1}^{\infty} \frac{a(b^2 + \theta_m^2) - (-1)^m b\sqrt{(a^2 + \theta_m^2)(b^2 + \theta_m^2)}}{\theta_m^2(\theta_m^2 + a + a^2 + b + b^2) + ab(a + b + ab)} \cdot \exp[-\theta_m{}^2 t_d] \tag{E-6}$$

The a and b now refer to the ratios of liquid compressive storages:

$$a = \frac{V_p(c_L + c_{pv})}{V_1(c_L + c_{v_1})} \equiv \frac{S_{pv}}{S_1} \tag{E-7}$$

and

$$b = \frac{V_p(c_L + c_{pv})}{V_2(c_L + c_{v_2})} \equiv \frac{S_{pv}}{S_2} \tag{E-8}$$

and k_g and c_g in Equation D-6 are replaced by k_L and c_L, respectively.

After the early-time portion of the pressure decay, a plot of the natural logarithm of $\Delta p[t]/\Delta p[0]$ vs. time results in a straight line:

$$\ln\left[\frac{\Delta p[t_n]}{\Delta p[0]}\right] = \ln[f_0] - \left[\frac{C_1 f_1 kA}{C_2 \mu L}\left(\frac{1}{S_1} + \frac{1}{S_2}\right)\right] t_n \tag{E-9}$$

or, equivalently:

$$\ln\left[\frac{\Delta p[t_n]}{\Delta p[0]}\right] = A_0 + A_1 t_n \tag{E-10}$$

A_0 and A_1, which are the intercept and slope of the straight line, are found from Equation E-10 using linear regression on the linear portion only of the pressure decay data. The value of f_0, defined by Equation D-14, is calculated from the intercept:

$$f_0 = \exp[A_0] \tag{E-11}$$

The parameter f_1, which is needed for the calculation of permeability, is found from f_0 and the ratio of the calibrated downstream and upstream storages, S_2 / S_1, with the aid of Figure 6.D-2. Permeability is calculated from:

$$k_L = \frac{-A_1 C_2 \mu L}{C_1 f_1 A\left(\frac{1}{S_1} + \frac{1}{S_2}\right)} \tag{E-12}$$

Amaefule, et al.[30] used pore pressures ranging from 500 to 4,500 psi. Kamath, et al.[29] recommend pore pressures of nearly 1000 psi and pulse sizes of about 20 percent of the pore pressure, or more preferably, about 10 percent of the net confining stress. Volumes of the upstream and downstream reservoirs should be equal if possible, for reasons given in the gas pulse-decay section. They should be slightly smaller or about the same volume as the pore volume of the rock to be measured if heterogeneity is to be investigated, or 5 to 10 times larger, or more, if average permeabilities, comparable to steady-state values, are desired. See Kamath, et al.,[29] regarding “early time solutions” for investigation of heterogeneity, which is beyond the intended scope of this document.

SECTION 7—SUPPLEMENTARY TESTS

CONTENTS

Page

7 SUPPLEMENTARY TESTS 7-1
7.1 General 7-1
7.2 Petrographic Characterization 7-1
7.3 Grain Size Distribution 7-1
7.4 Oil Gravity 7-8
7.5 Oil Characterization 7-11
7.6 Acid Solubility 7-13
7.7 Core Water Salinity Determination 7-14
7.8 References 7-22
7.9 Bibliography 7-23

Figures
7-1 Examples of Graphical Forms for Reporting Sieve Analysis Data 7-3
7-2 Refractive Index (RI) Difference Versus Salinity (RI of Solution—RI of Distilled Water 7-19
7-3 Conversion Chart for Salinity Determination Resistivity Versus Temperature and Salinity 7-21

Tables
7-1 Commonly Available Screen Sizes 7-2
7-2 Example of Sieve Analysis Data 7-2

7 Supplementary Tests

7.1 GENERAL

Acid solubility or calcimetry, oil gravity, core water salinity, grain size distribution, basic oil characterization, and petrographic characterization are supplementary data frequently obtained in conjunction with core analyses. Accordingly, these tests have been included because the data are useful in understanding and interpreting the core analysis results.

7.2 PETROGRAPHIC CHARACTERIZATION

Petrographic characterization is an important part of core analysis. Together, petrographic information and basic core analysis measurements provide an integrated framework for reservoir evaluations, including log interpretation. With geologic data, the results of core analyses can be extended to parts of a reservoir where there are no core measurements. Petrographic analysis also provides a basis for interpreting core analysis results, especially those that are unexpected or anomalous. Unexpected results may be valid and caused by variations in rock texture and/or mineralogy.

7.2.1 Petrographic characterization is best performed on the core plug used for routine core analysis so that the data are directly applicable. In those cases in which the plug cannot be consumed, the end sections obtained in preparing the core test samples or the core material from the same bedding plane as the test samples can be used. A minimum of one cubic inch (approximately 40 grams) of core should be saved for petrographic characterization.

7.2.2 Description of core analysis samples provides vital information on sample quality and character. Descriptions should be performed on extracted samples with the aid of a good binocular microscope. Visual porosity and pore type (e.g. intergranular, vuggy, moldic), drilling—or coring—induced features (e.g., drilling fluid invasion or induced fractures), and natural heterogeneity (clay laminae, lithologic variation normal to bedding, mineralized fractures, etc.) should be recorded. Petrographic characterization should include textural information and major mineralogical components. Textural data should include information on grain size, sorting, rounding, and grain shape. Mineralogical observations should include information on grain types and abundance. Approximate amounts of cement and matrix components can also be estimated with a binocular microscope in many cases.

7.2.3 Quantitative or semi-quantitative petrographic information can be obtained from thin sections, x-ray diffraction (XRD), bulk rock chemical analysis (usually by x-ray fluorescence, XRF), and Fourier transform infrared spectroscopy (FTIR). Valuable information regarding the spatial distribution of clay minerals within a sample can be obtained from scanning electron microscopy (SEM).

7.3 GRAIN SIZE DISTRIBUTION

Grain size distribution data have: (a) engineering application in well completion programs in friable and unconsolidated sediments, (b) geological application in assessing sand heterogeneity and depositional environment interpretation in both consolidated and unconsolidated clastic sediments, and (c) petrophysical application in various phases of formation evaluation to effect an understanding of log responses. Grain size distribution data are commonly utilized in sidewall core analysis to derive permeability. Methods commonly employed include wet and dry sieving, settling tube particle size analysis, thin section grain size analysis, and laser diffraction particle size analysis.

7.3.1 Sieve Analysis (Mechanical Shaker)

7.3.1.1 Principle

A known mass of extracted, disaggregated sample should be mechanically vibrated through stacked screens with progressively smaller openings. The partial sample weights retained on individual screens are used to produce a sieve analysis report of screen opening size versus percent sample retained.

7.3.1.2 Apparatus

The following equipment is recommended:

a. Mechanical sieving device.
b. U.S. or Tyler sieves (the sieves must conform to ASTM Specification E-11, *Specification for Wire Cloth Sieves for Testing Purposes*).[1]
c. Balance (0.01 gram).
d. Mortar and soft-tipped pestle.
e. Soft bristle brush.
f. Drying oven.

7.3.1.3 Procedure

The screens should be cleaned, dried, weighed, and nested from the largest to the smallest screen size. A pan is placed at the bottom of the screen assembly. Table 7-1 illustrates the commonly available screens and the size equivalents.

A representative core sample is extracted to remove hydrocarbons and salt. The sample is then dried to weight equilibrium to remove solvent and water. The sample is gently disaggregated, weighed to the nearest 0.01 gram (sample size typically 100 grams if eight-inch diameter screens are used),

Table 7-1—Commonly Available Screen Sizes

	U.S. Standard Sieve Mesh	Size in Millimeters
Gravel	5	4.00
Gravel	6	3.36
Gravel	7	2.83
Gravel	8	2.38
Gravel	10	2.00
Very coarse sand	12	1.68
Very coarse sand	14	1.41
Very coarse sand	16	1.19
Very coarse sand	18	1.00
Coarse sand	20	0.84
Coarse sand	25	0.71
Coarse sand	30	0.59
Coarse sand	35	0.50
Medium sand	40	0.42
Medium sand	45	0.35
Medium sand	50	0.30
Medium sand	60	0.25
Fine sand	70	0.21
Fine sand	80	0.18
Fine sand	100	0.15
Fine sand	120	0.13
Very fine sand	140	0.11
Very fine sand	170	0.09
Very fine sand	200	0.07
Very fine sand	230	0.06
Silt	270	0.05
Silt	325	0.04
Silt	430	0.03

and placed into the top screen. The nest of screens is vibrated with a mechanical sieve shaker or manually. The grains travel downward through the nest of screens until retained on a screen having a mesh size smaller than the minimum grain dimensions. Each loaded screen is weighed. The empty screen weights are deducted from the loaded screen weights to obtain the weight of the material retained on individual screens. The data are generally reported as weight percents retained on individual screens and cumulative weight percents to and including each screen. The data are typically reported in both tabular and graphical form (see Table 7-2 and Figure 7-1).

7.3.1.4 Precautions

Precautions of this method include:

a. The sample must be thoroughly extracted to remove residual hydrocarbons. In some cases, salt must be extracted. Care must be taken throughout the extraction process to avoid fines loss.

b. Sample disaggregation must be sufficient to reduce the sample to individual grains without crushing grains or otherwise artificially creating fines.

Table 7-2—Example of Sieve Analysis Data

U.S. Standard Sieve Mesh	Size in Microns	Percent Retained	Cumulative Percent
10	>2000	3.72	3.72
18	>1000	12.69	16.41
35	>500	34.74	51.15
60	>250	21.74	72.89
120	>125	7.30	80.19
170	>88	2.36	82.55
230	>63	3.08	85.63
325	>44	1.55	87.18
430	>30	2.90	90.08
pan	<30	9.92	100.00

c. The screens must be microscopically inspected to ensure that they are not damaged. Puncture-type holes and tears at the screen to retainer contact are particularly troublesome in the finer mesh screens.

d. Sufficient time must be allowed in vibrating the screens to ensure that each size fraction has completely settled. The test duration will vary relative to the quantity and type of sample introduced for a given type of mechanical shaker.

e. The balance must be properly calibrated.

f. In shaly sands, static electricity can prevent charged <.044-millimeter fines from settling to the pan (see 7.3.3 for wet sieve alternates).

7.3.1.5 Calculations

Calculations for this method are as follows:

$$\%\text{Retained Each Screen} = \frac{(\text{loaded screen weight} - \text{empty screen weight})}{\text{total sample weight}} \times 100 \quad (1)$$

$$\%\text{Retained cumulative} = \text{sum of percent retained on each screen (to and including each mesh size from largest screen to pan)} \quad (2)$$

7.3.1.6 Advantages

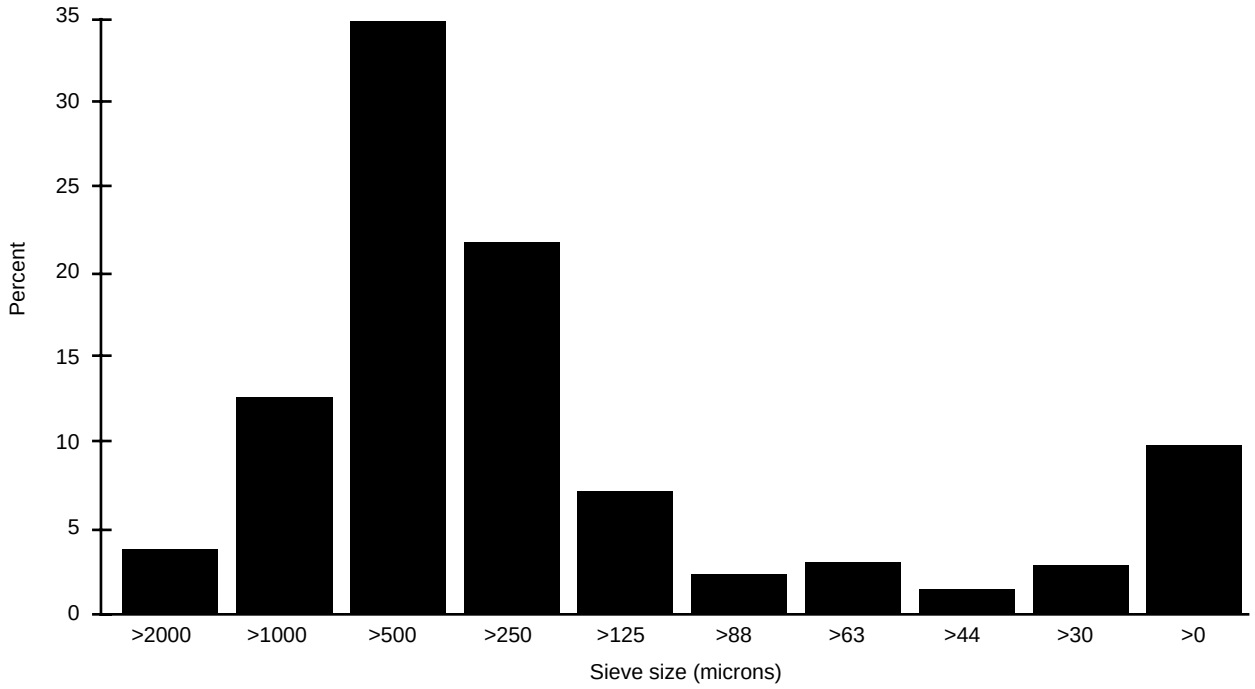
Advantages of this method include:

a. This method is acceptable for classifying grain sizes over the >.044 millimeter size range for minimally cemented sands. The method is a standard method and is accurate.

b. The sample can be recombined for reruns to ensure repeatability.

c. The sample can be recombined or maintained in separate size fractions for additional testing.

d. The test is simple and can be conducted with a minimum of equipment.



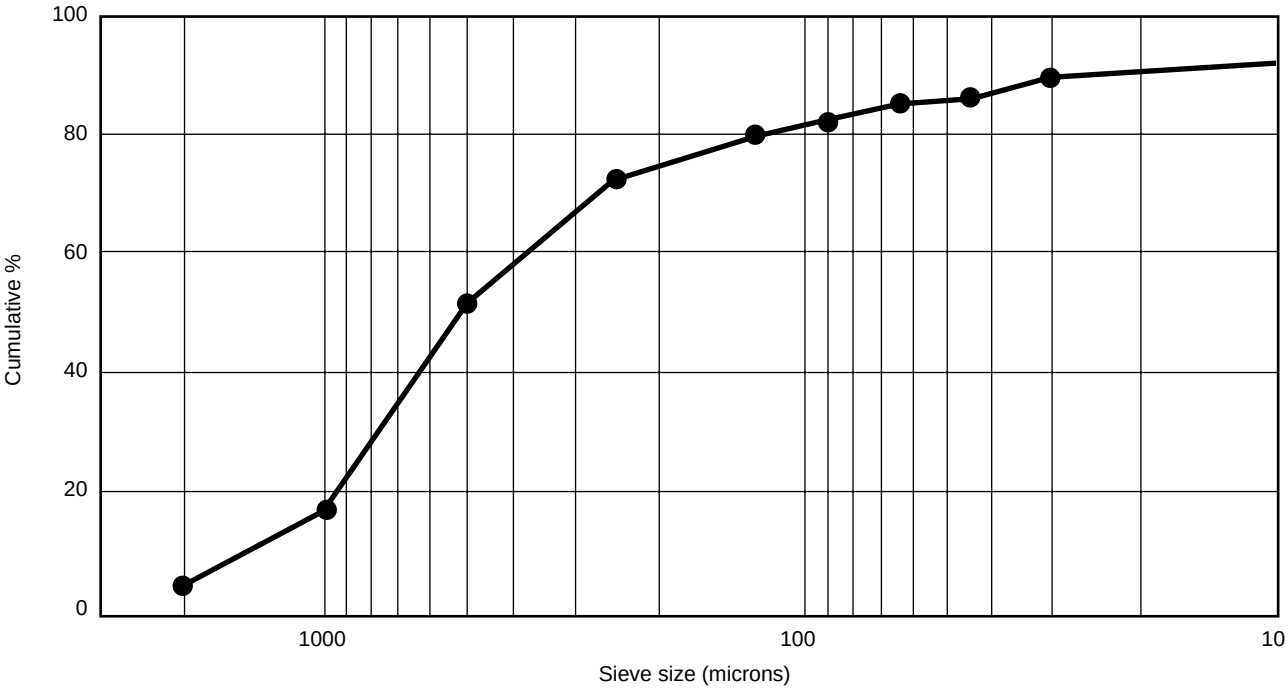


Figure 7-1—Examples of Graphical Forms for Reporting Sieve Analysis Data

7.3.1.7 Limitations

The method is relatively time consuming (one hour per sample for eight-inch diameter screens), requires a relatively large sample, and can yield inaccurate data in shaly sands and consolidated sands.

7.3.1.8 Accuracy and Precision

Provided the test is properly conducted and the precautionary measures above are observed, the expected deviation is less than 3 percent for the total sample and generally less than 0.5 percent for each screen.

7.3.1.9 Calibration

Referenced documents are ASTM C-136, *Standard Method for Sieve Analysis of Fine and Coarse Aggregates*,[2] and ASTM C-702, *Practice for Reducing Field Samples of Aggregate to Testing Size*.[3]

7.3.2 Sieve Analysis (Sonic Shaker)

7.3.2.1 Principle

Refer to 7.3.1, except that properly prepared samples are vibrated through the sieve screens with acoustic energy.

7.3.2.2 Apparatus

The following equipment is recommended:

a. Sonic sieving apparatus.
b. U.S. or Tyler sieves (see 7.3.1.2b).
c. Balance (0.001 gram).
d. Mortar and soft-tipped pestle.
e. Soft bristle brush.
f. Sample splitter.
g. Drying oven.

7.3.2.3 Procedure

Sample preparation and measurement procedures follow the material provided in 7.3.1. Five to fifteen grams of properly prepared sample are placed in the top screen. Sift and pulse amplitudes are adjusted to effectively agitate grains. Sifting time for effective sorting varies from ten to twenty minutes and is dependent upon sample size and degree of shaliness.

7.3.2.4 Precautions

Precautions for this method include:

a. Refer to 7.3.1.4, items a through f.
b. More accurate weight determinations are required than in mechanical sieve analysis because the sample sizes are smaller.

7.3.2.5 Calculations

Refer to 7.3.1.5.

7.3.2.6 Advantages

Advantages of this method include:

a. The method is acceptable for classifying grain sizes over the >0.044 millimeter size range.
b. The test is simple and can be conducted with a minimum of equipment.
c. The test can be conducted more rapidly that the mechanical shaker sieve analysis method (approximately 15 minutes per sample).
d. Less sample is required for the sonic method (5-15 grams) than for the mechanical method.
e. The sample can be recombined for reruns, retained in separate size fractions, or recombined for additional testing.

7.3.2.7 Limitations

Limitations of this method include:

a. Care must be taken to obtain a representative sample due to small sample size.
b. Charged fines retention can be particularly troublesome using this method because the screen frames are plastic. As a result, the sieving time must be increased in shaly sands. In many cases, a significant increase in sieving time may not be adequate to move the <0.044 millimeter fines to the pan.

7.3.2.8 Accuracy

Refer to 7.3.1.8.

7.3.2.9 Calibration

Refer to 7.3.1.9.

7.3.3 Sieve Analysis (Wet and Wet/Dry Combination)

7.3.3.1 Wet sieve analysis refers to the technique of washing a representative, extracted sample through individual sieve screens as opposed to the dry sieving technique, whereby the sample is mechanically agitated or sonified through a nest of sieve screens. The technique requires significantly more time than dry sieving techniques but is preferred in shaly sands.

7.3.3.2 Wet/dry combination sieve analysis, as the name implies, is a combination of both wet and dry techniques. This technique is a very good alternative in shaly sands. The sample is weighed and then wet sieved through the 325 mesh screen to remove the <0.044 millimeter fraction. The material retained on the 325 mesh screen is dried, weighed, and dry sieved in the manner prescribed in the sieve analysis (mechanical or sonic shaker) sections (see 7.3.1 and 7.3.2).

7.3.4 Particle Size Analysis (Laser Diffraction)

7.3.4.1 Principle

An extracted, disaggregated sample is placed into a suitable carrier fluid (carrier fluid may be oil-based or water-based, depending upon test criteria). The carrier fluid is typically stirred or recirculated to ensure particle dispersion. A laser beam is projected through the dispersed sample and the diffracted light patterns are analyzed by microcomputer. The principle upon which grain size distribution is computed is that of Fraunhofer diffraction theory. The angles through which the incident light is scattered are inversely proportional to particle sizes, and the intensity of light scattered by the particles is directly proportional to particle quantities.

7.3.4.2 Apparatus and Supplies

Recommended equipment for this method include:

a. Laser optics device/appropriate computer hardware.
b. Sample splitter.
c. Balance (0.0001 gram), not required for all devices.
d. Mortar and soft-tipped pestle.
e. Sonifier.
f. Deflocculant solution.
g. Drying oven.

7.3.4.3 Procedure

A representative sample is extracted to remove hydrocarbons and dried to remove solvent. The sample is sonified prior to measurement to disperse the particles. The carrier fluid reservoir is charged with carrier fluid and a background measurement is taken. The sample is introduced and surfactant (typically sodium hexametaphosphate) is added to reduce surface tension and to promote particle deflocculation. The carrier fluid/sample mixture is recirculated or stirred sufficiently to ensure uniform particle dispersion. During the measurement phase, a laser beam of known and fixed wave length is projected through the carrier fluid/sample mixture. The forward-diffracted light impacts a photodetector array and Fraunhofer diffraction theory is invoked to compute particle size distribution from the diffracted light flux. The data may be reported in sieve analysis format in tabular and graphical form to the submicron particle size range (see Table 7-2 and Figure 7-1).

7.3.4.4 Precautions

Precautions for this method include:

a. Refer to 7.3.1.4, items a and b.
b. The instrumentation must be properly calibrated (including laser alignment, photodetector array calibration, etc.).
c. The carrier fluid must be bubble-free and particle-free throughout the entire measurement range.
d. The sample must be uniformly dispersed in the carrier fluid and must be deflocculated during the measurement phase.
e. Care must be taken to obtain a representative sample.

7.3.4.5 Calculations

The algorithms to calculate grain size distribution from composite diffraction patterns are complex, manufacturer-dependent, and proprietary. However, the following equations can be utilized to illustrate the relationships between the angular extent of the diffraction pattern, particle size, scattered light intensity, and particle quantity.

The angular extent of the diffraction pattern is given by:

$$\sin\theta = \frac{1.22\lambda}{d} \tag{3}$$

Where:

θ = half-angle to the first minimum of the pattern, degrees.
λ = incident light wave length, microns.
d = particle diameter, microns.

The total intensity of the scattered light is given by:

$$I = Knd^2 \tag{4}$$

Where:

I = total diffracted light, watts.
K = instrument calibration constant, watts/micron^2.
n = number of particles.
d = particle diameter, microns.

7.3.4.6 Advantages

Advantages of this method include:

a. The grain size measurement range provides classification of particles to and including the sub-micron range (very fine silt and clay sized particles).
b. The data are highly reproducible.
c. The technique requires very little sample (typically <1.0 gram).
d. The measurement is rapid (30 seconds to several minutes).

7.3.4.7 Limitations

Limitations of this method include:

a. A size limit exists above which measurements cannot be made. This limit typically falls within the coarse sand fraction. If particle sizes exceed this limit, pre-sieving is required and the data sets must be combined.
b. The instrumentation and associated hardware is expensive.

7.3.4.8 Calibration

Calibration requirements are instrument-manufacturer dependent but generally involve accurate laser beam alignment, photodetector array calibration, and diffraction pattern calibration using a precision standard.

7.3.4.9 Accuracy

Insufficient data exist at present to establish accuracy.

7.3.5 Thin Section Measurement of Grain Size (Visual Technique)

7.3.5.1 Principle

Grain size measurement using thin sections can be undertaken on samples that range from unconsolidated sand to well-consolidated rock. It is the method of choice for consolidated rock, because such rocks cannot be disaggregated successfully without either breaking grains or failing to separate adjacent, well-cemented grains. The technique requires preparation of a thin section and measurement of the size of individual grains visually or with an image analysis system, using a petrographic microscope.

7.3.5.2 Apparatus and Supplies

Recommended equipment for this method includes:

a. Petrographic microscope equipped with an eyepiece micrometer.

b. Thin section preparation equipment.

c. Glass slides.

d. Colored epoxy resin.

7.3.5.3 Procedure

Extracted samples are usually impregnated with colored epoxy resin, mounted on a glass slide, and ground to 30 microns thickness. This thickness allows light to be transmitted through most sand grains. Grain size is measured utilizing the longest axis of individual grain cross sections in the two-dimensional thin section. In many cases, unconsolidated samples need only be dispersed in oil on a glass slide. It is customary to restrict size measurement to a single specific detrital mineral species (usually quartz in sands), although other species may be included (such as feldspar). Typically, 200 to 300 grains are measured. The actual number is dependent upon sorting; poorly-sorted sands require more measurements than well-sorted sands. Grains are selected randomly using the point count technique described by Chayes (1956).[4] Measurements are typically made in millimeters and units are subsequently converted to units of preference (inches, phi units, etc.).

7.3.5.4 Precautions

Precautions for this method include:

a. Thin sections allow for grain size measurement in only two dimensions. Because grains are irregularly shaped and non-uniform in size, and because almost all sediments composed of non-spherical particles show anisotropic dimensional fabrics (i.e., preferred grain orientation), without gross simplifying assumptions there is no unique solution for inferring true grain size distributions determined from thin section measurements (Blatt et al., 1980).[5]

b. Apparatus must be calibrated.

c. Thin sections should be representative of the reservoir interval.

d. Care must be taken to obtain a representative split of the sample because relatively few grains are measured.

e. Sufficient grains must be available for measurement in the thin section to ensure statistical reliability.

f. For laminated sands with contrasting grain size between laminae, unbiased grain size selection can be difficult.

7.3.5.5 Calculations

Grain size is measured using non-dimensional units visible through the microscope eyepiece. These units are scaled to millimeters using a conversion factor dependent upon the magnification used. Size distribution is calculated on a volume frequency basis.

7.3.5.6 Advantages

Advantages of this method include:

a. The measurement range is typically from U.S. Sieve mesh 10 to 400.

b. Thin section measurement of grain size distribution is well suited to most reservoir rocks (sandstones and coarser grained siltstones).

c. The technique allows for standardized measurement of grain size in reservoirs characterized by a range of rock consolidation.

7.3.5.7 Limitations

Limitations of this method include:

a. The technique is tedious, relatively time consuming, and limited to a two-dimensional view.

b. Equipment and instrumentation is expensive.

c. Derivation of mean grain size and the size distribution from the thin section is not straightforward and can be biased. The data are adequate in a relative sense (Kellerhals et al.).[6]

d. Image resolution (magnification and thin section thickness dependent) precludes measurement of very small particles (finer silt and clay size fractions).

7.3.5.8 Accuracy and Precision

The data are slightly biased towards smaller sizes. Precision is a function of the number of grains measured.

7.3.5.9 Calibration

Calibration of size measured in thin section with size measured by sieving and other techniques is possible (Rosenfeld et al. 1953).[7]

7.3.6 Thin Section Measurement of Grain Size (Image Analysis)

7.3.6.1 Principle

Grain size estimates from unconsolidated and consolidated sands can be obtained using automated or semi-automated image analysis procedures. The information provided in 7.3.6 primarily refers to grain size estimates in unconsolidated sands.

7.3.6.2 Apparatus and Supplies

Recommended equipment for this method includes:

a. Image analyzer.

b. Petrographic microscope with light source.

c. Thin section preparation equipment.

d. Glass slides.

e. Colored epoxy resin.

7.3.6.3 Procedure (Unconsolidated Sands)

Sand grains can be dispersed on a glass slide such that few are touching. The projection area or diameter of each grain can be obtained using a simple image analysis apparatus comprised of a videocamera attached to a microscope (Mazullo and Kennedy, 1985).[8] Grains are separated from background using the difference in intensity of the background (brighter) and the grains (darker) as a criterion for thresholding. Typically 200 to 500 grains are required which require 5 to 15 minutes to process.

7.3.6.4 Procedure (Consolidated Sands)

Grain-size distribution using image analysis techniques outlined in 7.3.6 are complicated in consolidated sands by virtue of grain contact. However, techniques exist that utilize the center-to-center distance of intergranular pores to estimate grain size.

7.3.6.5 Precautions

Precautions for this method include:

a. Refer to 7.3.5.4.
b. Inaccurate measurements can occur if the grains to be measured are in contact with one another. This can result in diameters greater than actual.

7.3.6.6 Advantages

Advantages of this method include:

a. Acquisition of raw data is reasonably fast and data are stored in a digital format.
b. Very small samples can be analyzed.

7.3.6.7 Limitations

Limitations of this method include:

a. Care must be taken such that the few hundred grains are representative. This may require careful sample splitting and a careful grain selection procedure on the grain mount.
b. The image acquisition system with software and videocamera is more expensive than the visual technique.

7.3.6.8 Calculations

Data must be converted to volume frequency. This is done by: (a) defining a number of class intervals based on grain diameter, (b) cubing the radii of all grains in each class interval and adding volumes within each interval, and (c) summing all of the class interval volumes to obtain the total volume. The volume fraction in each class interval is calculated by dividing by this total.

7.3.6.9 Accuracy and Precision

The data have been demonstrated to be accurate and deviate only slightly from sieve data (the difference is due to the fact that sieves are shape-selective as well as size-selective).

7.3.6.10 Calibration

Pixel size for a given magnification must be defined when an image analysis system is utilized.

7.3.7 Stoke's Law Particle Size Analysis

7.3.7.1 Principle

A representative, extracted sample is introduced into a liquid column, and the settling velocities of the particles are determined by monitoring the weight increase of a pan suspended in the column. The sedimentation diameter of the particles (the diameter of a sphere of equal density having an equal settling velocity) is calculated using Stoke's Law, a

modified form of Stoke's Law, or Gibbs' formula (Gibbs et al., 1971[9]). The data are typically reported in the same manner as standard sieve data.

7.3.7.2 Apparatus

Recommended equipment for this method includes:

a. Mortar and soft-tipped pestle.
b. Soft bristle brush.
c. Bottom loading balance (0.001 gram) or strain transducer.
d. Settling column and associated computer hardware.
e. Drying oven.

7.3.7.3 Procedure

Sample preparation should follow the protocol outlined in 7.3.1. The sample is precisely weighed (0.001 gram) and introduced into the settling column. The particles fall through the liquid at rates related to size, shape, and density. The particles accumulate on a pan suspended from a precision balance or strain transducer a known distance from the top of the settling column. Weight and time data are acquired by a microcomputer. These data are utilized to compute settling velocities. The test duration varies from several to twenty minutes relative to the type of equipment/instrumentation and the particle size measurement range desired. A modified form of Stoke's Law or Gibbs' formula is typically utilized to calculate sedimentation diameters. If all of the sample is not recovered at the test end point, the weight difference is classified as being finer than the equivalent sedimentation diameter of the last measurement.

7.3.7.4 Precautions

Precautions for this method include:

a. Refer to 7.3.1.4, items a, b, and c.
b. The settling column must be clean (the fluid density must be constant, and suspended particle fallout on a subsequent measurement must be negligible).
c. The particles must be evenly dispersed upon introduction into the column and must be deflocculated.
d. Very careful weighing is required as a result of the small sample quantity used.
e. Convection currents must be eliminated.

7.3.7.5 Calculations

Stoke's Law, a modified form of Stoke's Law or Gibbs' formula are typically used to calculate grain radii from the settling velocity data.

Stoke's Law:

$$V = \frac{2gr^2(\rho_g - \rho_f)}{9\mu} \quad (5)$$

Gibbs' Formula:

$$V = \frac{-3\mu + \sqrt{9\mu^2 + gr^2\rho_f(\rho_g - \rho_f)(0.015476 + 0.19841r)}}{\rho_f(0.011607 + 0.14881r)} \quad (6)$$

Where:

V = settling velocity, cm/sec.
g = acceleration of gravity, cm/sec^2.
r = grain radius, cm.
ρ_g = grain density, g/cc.
ρ_f = fluid density, g/cc.
μ = fluid viscosity, poise.

7.3.7.6 Advantages

Advantages of this method include:

a. The test is rapid (several to twenty minutes).
b. The equipment is relatively inexpensive.
c. The measurement range is relatively broad (through U.S. Sieve 500 mesh).

7.3.7.7 Accuracy and Precision

Information not available.

7.3.7.8 Calibration

The calibration procedure required is described by Gibbs *et al* (1971).[9]

7.4 OIL GRAVITY

The specific gravity of the oil must be known in order to convert oil weight to oil volume in the distillation-extraction method for determining fluid saturations (see 4.3). The oil gravity may be determined by an oil drop method, a refractive index method, a gravimetric method, a digital densitometer method, or a nuclear magnetic resonance method.

7.4.1 Oil Drop Method

7.4.1.1 Principle

The oil drop method consists of suspending a drop of the oil in a liquid medium, the gravity of which can be measured with a hydrometer or specific-gravity balance.

7.4.1.2 Apparatus

Apparatus for this method includes:

a. Glass or clear plastic cylinder.
b. API hydrometer or specific-gravity balance.

7.4.1.3 Procedure

A drop of the oil recovered from the core during the fluid saturation test should be placed in a glass or plastic cylinder

containing a solution of methyl alcohol and water. The gravity of this solution is subsequently adjusted by the addition of alcohol or water until the oil drop remains suspended; i.e., after agitation, the drop neither rises nor falls. The gravity of the solution is then measured with an API hydrometer or a specific-gravity balance. The correct procedure for measuring gravity with a hydrometer is found in ASTM D287-55, *Method of Test for API Gravity of Petroleum.*[10] The observed API gravity at the test temperature is corrected to the gravity at 60°F and converted to specific gravity by the use of Tables 5 and 3, respectively, of the ASTM-IP *Petroleum Measurement Tables* (American Edition).[11] A specific-gravity balance permits a direct reading of gravity. A simple chart for converting units of liquid gravity and density may be useful.

7.4.1.4 Precautions

Precautions for this method include:

a. Any air bubbles adhering to the surface of the oil drop must be removed before adjusting the gravity of the alcohol-water solution.

b. The solution must be agitated after each addition of alcohol or water to ensure a uniform solution and gravity.

7.4.1.5 Advantages

Elaborate equipment is not required.

7.4.1.6 Limitations

Adjustment of the alcohol-water ratio is time consuming.

7.4.1.7 Accuracy and Precision

Information not available.

7.4.2 Refractive Index Method

7.4.2.1 Principle

The refractive index method for determining API gravity is most commonly used to determine the gravity of oil recovered from core samples during the summation of fluids saturation test. The technique is also used to determine the API gravity of stock tank oil, formation test oil, drill stem test oil, etc., in those cases in which very small quantities of oil are available.

A suite of stock tank oil samples of known API gravities for the normally encountered gravity range is required. The refractive indices of the oils, before and after the summation of fluids procedure, are measured. Refractive index versus oil gravity plots are made. Oil gravity can then be determined graphically or calculated using a simple linear regression.

7.4.2.2 Apparatus

Apparatus for this method includes:

a. API hydrometers.
b. Assorted glassware.
c. Glass pipette.
d. Refractometer (refractive index range 1.30-1.90).

7.4.2.3 Procedure

The API gravity of a stock tank oil sample is determined with an API hydrometer using ASTM D287-55: *Method of Test for API Gravity of Petroleum,*[10] or with a specific-gravity balance. A representative aliquot of oil is recovered from the stock tank sample with a glass pipette and the refractometer is charged with the sample. The refractive index of the sample is determined. The stock tank oil sample is then subjected to the retorting phase of the summation-of-fluids analysis (see 4.2). (Retort method for fluid saturation determinations, both sidewall and conventional core analysis.) The refractive index of the recovered oil is determined. Plots of refractive index before and after retorting versus API gravity should be made. API gravities can then be determined graphically or calculated.

7.4.2.4 Precautions

Precautions for this method include:

a. Operation of the refractometer must be verified by measuring the refractive index of a known liquid.
b. The refractometer prism and the glass plate of the refractometer eyepiece must be thoroughly clean.
c. The refractometer prism must be securely placed against the refractometer eyepiece when making measurements.
d. Contaminants such as water and solids must be removed from the oil prior to measurement.
e. The crude must be light-colored and a thin-film refractometer must be utilized.

7.4.2.5 Calculations

A simple linear regression of the stock tank oil API gravities and refractive indices can be made. The refractive index, slope, and intercept can be utilized to calculate the API gravity.

7.4.2.6 Advantages

Advantages of this method include:

a. The technique is simple and rapid, and the data are reasonably accurate.
b. Elaborate equipment is not required.
c. The test requires very little sample (0.03 cc).

7.4.2.7 Accuracy and Precision

API gravity determined using this method should be ±1 degree API of the gravity determined by ASTM D287-55[10], if the calibration is properly made and if the precautionary measures are taken.

7.4.3 Gravimetric Method (Micro-Pycnometer Method)

7.4.3.1 Principle

The density of an oil sample is determined using a small pycnometer and the API gravity is computed.

7.4.3.2 Apparatus

Equipment for this method includes:

a. Balance (0.001 gram).
b. Calibrated pycnometers—The pycnometers can be made of thin-walled glass capillary tubing formed in a U-shape. Various sizes can be prepared by varying the length and/or the inside diameter of the tubing. The volumes should be calibrated with a liquid of known density, with the pycnometer completely full or filled to reference lines (gas-free distilled water may be used).

7.4.3.3 Procedure

A clean, dry pycnometer is weighed. A portion of the oil to be analyzed is drawn into the pycnometer. It is preferable that the largest practical amount of sample be utilized for the gravity determination. The pycnometer is re-weighed. The oil weight is calculated and divided by the capillary volume to determine the oil density. The specific gravity is calculated by dividing the oil density by the water density at the test temperature. The API gravity is calculated.

7.4.3.4 Precautions

The pycnometer weight should be minimized relative to the oil weight. This will reduce error as the weight difference becomes more accurate.

7.4.3.5 Calculations

The following are the calculations for this method:

$$\text{Oil Density (g/cc)} = \frac{\text{charged pycnometer wt (g)} - \text{initial pycnometer wt (g)}}{\text{pycnometer volume (cc)}} \quad (7)$$

$$\text{Specific Gravity} = \frac{\text{oil density (g/cc)}}{\text{water density (g/cc)}} \quad (8)$$

$$\text{API Gravity (deg)} = \frac{141.5}{\text{specific gravity}} - 131.5 \quad (9)$$

7.4.3.6 Advantages

Advantages of this method include:

a. Accurate data can be obtained if the pycnometer has been properly calibrated.
b. The determination is rapid.

7.4.4 Digital Densitometer Method

7.4.4.1 Principle

The measurement principle of the digital densitometer is based on the change of the natural frequency of a hollow oscillator when fluid-filled. The oscillator frequency change is related to the mass change of the oscillator subsequent to sample introduction, and as a result, to the sample density.

7.4.4.2 Apparatus

A digital densitometer is required.

7.4.4.3 Procedure

Instrument calibration constants are determined by measurement of the air-filled and distilled water-filled oscillator frequencies. The sample is injected into the oscillator and the oscillator frequency is determined. The API gravity is calculated as indicated in 7.4.3.5.

7.4.4.4 Advantages

The technique is rapid and accurate.

7.4.4.5 Limitations

Relatively large amounts of oil are required (1 to 2 cc).

7.4.5 Nuclear Magnetic Resonance (NMR) Method

7.4.5.1 Principle

The NMR method for determining the viscosity of crude oils is based on Stoke's relationship between viscosity and molecular translational and rotational diffusion that cause NMR spin-lattice relaxation. Either ^{1}H or ^{13}C NMR can be used. A major advantage is that the method can be applied to bulk crude oil and/or oil/water emulsions, crude oil inside core plugs or whole cores, or crude oil dissolved in solvent extracts.

7.4.5.2 Apparatus

Fourier Transform NMR Spectrometer with ^{1}H chemical shift or ^{13}C capabilities. For NMR diffusion measurements, a pulsed gradient NMR probe is required with field gradients above 100 gauss/cm.

7.4.5.3 Procedure

The ^{1}H or ^{13}C spin lattice relaxation time (T1) is measured with an inversion recovery or saturation recovery pulse sequence. Viscosity and API gravity are then determined from published correlations. ^{13}C NMR is preferred for analysis of oils inside cores because chemical shift separation between oil and water phases in the core is not required.

The application of a large, steady-pulsed magnetic field gradient enables direct ^{1}H NMR measurements of translational diffusion in liquids. The pulse sequence is normally a 90°-T-180°-spin echo with magnetic field gradient pulses applied after the 90° and 180° RF pulses. Diffusion in an applied field gradient causes an irreversible loss in the spin echo amplitude because only the nuclei that do not diffuse to a region with a different magnetic field will be refocused. The interpulse time (T) is kept very short (approximately 1 millisecond) to prevent restricted diffusion at the pore walls, and the water and oil signals are separated either by chemical shift spectroscopy or by doping the water with Mn-EDTA to eliminate the water signal. The oil diffusion coefficient can be related to viscosity via Stoke's Law and API gravity can be estimated from published correlations between viscosity and API gravity.

7.4.5.4 Advantages

Advantages of the NMR method include:

a. The technique is rapid, noninvasive, and can be automated.
b. The technique is applicable to bulk oil, oil in plug cores, or whole core, and liquid extracts.
c. Minimal sample preparation is required.
d. Measurements can be made on oil/water emulsions.
e. Very small amounts of oil are required for ^{1}H NMR (<0.1 cc).

7.4.5.5 Limitations

Limitations of the NMR method include:

a. The NMR spectrometer is expensive.
b. The T1 method is applicable for oils only up to approximately 1,000 cp (approximately 15 degrees API) because of the flattening of the T1 versus viscosity correlation. This limitation does not apply to NMR diffusion measurements, which are only limited by the strength of the applied field gradient.

7.5 OIL CHARACTERIZATION

7.5.1 Oil Characterization (Chromatographic Method/Hydrocarbon Vaporization)

Residual hydrocarbons entrained in drill cuttings and core samples can be analyzed to quantify hydrocarbon volume and to evaluate the compositional characteristics of the oil. These data can be interpreted to provide information in the following areas:

a. Alteration processes—The crude oil quality is dictated by one or more of the alteration processes that affect the oil during, or subsequent to, its accumulation in a reservoir. The changes resulting from alteration processes are reflected in the composition of the residual hydrocarbon found in the rock pore spaces.
b. Type of hydrocarbon production—The composition of the residual hydrocarbon in the rock matrix at the surface reflects the original reservoir composite less components that have escaped through pressure depletion. The distribution of intermediate and heavier components can be used to predict the type of hydrocarbon production (non-productive, gas, condensate, or oil).
c. Changes in rock characteristics (permeability)—The escape of components during pressure depletion is limited by molecular size and hydrocarbon volatility, but is also greatly influenced by permeability. Compositional contrast can often be related to permeability and permeability variation.
d. Contamination (drilling fluids and/or additives)—Drilling fluids and/or additives can be analyzed by the same procedure as the rock samples. Compositional contrast and hydrocarbon ratio techniques can be utilized to distinguish indigenous hydrocarbons from drilling contaminants.
e. Correlation of produced oil to drill cuttings or core sample oil.

7.5.1.1 Principle

This method involves vaporization of residual hydrocarbons in drill cuttings or core samples directly onto a chromatographic column. The components separated by the column are detected by a flame ionization detector (FID). Peak heights and areas are obtained from the recorded output.

7.5.1.2 Apparatus

Apparatus recommended for this method includes:

a. Chromatograph—The chromatograph should contain a large bore capillary column (0.5 millimeter ID) containing a stationary phase that is suitable for separation and a flame ionization detector.
b. Vaporization cell (low dead volume, accurate temperature control, and even temperature distribution).
c. Sample boat (quartz tube).
d. Integrator.
e. Balance (0.001 gram).

7.5.1.3 Procedure

A representative sample (0.01 to 1.0 gram) should be selected and placed in a tared quartz sample boat (drill cuttings must be rinsed with water to remove drilling fluid and

air dried for two to four hours prior to charging the boat). The quantity of sample used is dependent on the oil saturation, the detector capacity, and the column limitations. The charged sample boat is weighed and placed in the vaporization cell. Human skin oil is a possible contaminant and care should be taken to avoid directly handling the sample.

The hydrocarbons are vaporized at a temperature that will not induce cracking. Temperatures above 320°C will cause cracking of larger molecules. The gas evolved is passed directly onto the column without splitting. Resolution can be enhanced by increasing the column temperature at a programmed rate. Output of the flame ionization detector can be converted to millivolts (height) and millivolts x seconds (area). Specific peaks are identified by their retention time.

7.5.1.4 Precautions

Precautions for this method include:

a. The sample must be representative.

b. Large samples may increase reproducibility but will restrict peak resolution.

c. High bound-water saturation may cause a suppression of detector response, particularly in the lighter midrange components.

d. Accurate weights are required (±0.001 gram).

e. Care must be taken to avoid heating during sample drying to prevent lighter component losses.

7.5.1.5 Calculations

7.5.1.5.1 Quantification of residual hydrocarbon volume:

$$\text{Normalized total area } (A_{nt}) = \frac{\text{total area } (A_t)}{\text{sample weight } (g)} \quad (10)$$

7.5.1.5.2 Alteration processes—Indices can be tabulated to distinguish changes in composition.

Following are several indices that are typically used to determine type, origin, and alteration processes:

a. n-C15+ (%) = $\dfrac{> n\text{-C15 area}}{A_t} \times 100$ (11)

b. n-alkane (%) = $\dfrac{n\text{-c components area}}{A_t} \times 100$ (12)

c. ratio n-C17 + n-C18 to pristane+phytane =

$$\frac{(\text{height n-C17} + \text{height n-C18})}{(\text{height pristane} + \text{height phytane})} \quad (13)$$

d. pristane/phytane ratio = $\dfrac{\text{height pristane}}{\text{height phytane}}$ (14)

e. Carbon preference index =

$$\left|\frac{\Sigma\text{odd heights n-C17-C31}}{\Sigma\text{even heights n-C16-C30}}\right| + \left|\frac{\Sigma\text{odd heights n-C17-C31}}{\Sigma\text{even heights n-C18-C32}}\right| \quad (15)$$

7.5.1.5.3 Type of hydrocarbon production:

The area percent within each carbon group (light, intermediate and heavy) is calculated as follows:

a. % light components = $\dfrac{< \text{C8 area}}{A_t} \times 100$ (16)

b. % intermediate components = $\dfrac{< \text{C13 area}}{A_t} \times 100$ (17)

c. % heavy components = $\dfrac{> \text{C18 area}}{A_t} \times 100$ (18)

Prediction of hydrocarbon production can be based on the following general guidelines:

a. Non-hydrocarbon productive—relatively high percentage of lighter components, relatively low total area.

b. Gas productive—relatively high percentage of lighter components with lower percentages of intermediate components, relatively high total area.

c. Oil productive—relatively high percentage of intermediate and heavy components, high total area.

7.5.1.6 Advantages

Advantages of this method include:

a. Extremely small samples (0.01-1.0 gram) can be utilized.

b. Solvent extraction and sample splitting are not required.

c. Oil composition and residual hydrocarbon content can be monitored simultaneously.

7.5.1.7 Limitations

Bound water may suppress detector response, particularly in the lighter midrange components.

7.5.1.8 Accuracy

Accurate sample weights and careful control of operating conditions should yield oil volumes reproducible to within 3 percent. Reproducibility of component distribution is dependent on maintenance of operating conditions and column condition and can be verified by repeat runs.

7.5.1.9 Calibration

Equipment calibration is essential for consistent results. The following areas are critical:

a. Component identification—Identification of individual peaks is based in part on retention time. Retention time is dependent on carrier gas flow rate, column temperature, and the condition of the column stationary phase. Carrier gas flow rate should be calibrated and the system should be leak tested routinely. Retention times for specific peaks can be measured. A sample can be spiked with specific components, such as the normal alkanes for calibration purposes.
b. Calibration of detector response—A standard should be used to check peak resolution and system noise. Relative areas should be consistent. Relative standard deviation of 2 to 3 percent is typical.
c. Reproducibility—Reproducibility can be evaluated by making repeated runs of the same sample. Specific indices used should be checked along with total area normalized by weight.

7.6 ACID SOLUBILITY

7.6.1 Principle

Acid solubility tests on drill cuttings or core samples are typically used to assess HCl and/or mud acid (HCl/HF mixture) reactivity with reservoir rock. These data are used to evaluate the potential success of acid stimulation, near wellbore damage removal, to assess formation damage sensitivity, to determine calcite/dolomite ratios in carbonates, and to determine quartz content in carbonates. Test procedures can be adapted to determine acid volumes required for field treatment and to determine acid response coefficients.

The acid solubility test is a gravimetric test performed to determine formation sensitivity to acid. A known mass of extracted and dried sample is placed in excess acid for a specific residence period. Subsequently, the sample is dried and reweighed. The weight difference is used to calculate the percent acid-soluble material.

7.6.2 Apparatus and Supplies

Equipment for acid solubility determination includes:

a. Balance (0.001 gram).
b. Assorted glassware for HCl solubility (beakers, funnels, graduated cylinders, etc.).
c. Assorted polyethylene or polypropylene beakers, funnels, graduated cylinders, etc. for mud acid solubility.
d. Mortar and pestle.
e. U.S. Sieve, 80 mesh screen.
f. Drying oven.
g. Acid-resistant filter paper.
h. HCl, 15 weight percent.
i. Mud acid (HCl, 12 weight percent—HF, 3 weight percent).
j. Appropriate safety equipment (face shield, apron, etc.).

7.6.3 Procedure (HCl Solubility, Calcite Content)

A representative sample is extracted to remove hydrocarbons and dried to constant weight to ensure removal of solvent and water. The sample is disaggregated sufficiently to produce several grams of material finer than U.S. Sieve 80 mesh. Approximately 1 gram of sample is weighed and placed in 150 cc of 15 percent HCl for a minimum of 60 minutes and a maximum of 65 minutes without stirring. The filtration apparatus is prepared and the filter paper is weighed. The acid/sample mixture is filtered and rinsed with deionized water. The remaining sample is dried and weighed. The weight difference is used to compute the percent acid soluble material.

7.6.4 Procedure (HCl Solubility, Calcite Plus Dolomite Content)

Dolomite is also soluble in HCl at 80°C. Applying the procedure in 7.6.3 at this temperature yields a value referred to as the total carbonate content (calcite plus dolomite).

7.6.5 Procedure (Mud Acid Solubility, Total Acid-soluble Content)

Carbonates and siliceous materials such as feldspars and clays dissolve in a mixture of HCl and HF. This mixture is generally called mud acid. Although various concentrations are in use, a mixture of 12 weight percent HCl and 3 weight percent HF is most commonly applied. Applying the procedure above, using HF tolerant apparatus, with mud acid results in the total acid solubility (i.e., carbonates, feldspars, iron oxides, clays, etc.).

7.6.6 Precautions

The procedure is gravimetric and requires strict adherence to procedures and good laboratory technique to provide reproducibility.

CAUTION: In all procedures outlined in 7.6.3, 7.6.4, and 7.6.5, appropriate safety precautions for handling acids must be employed.

7.6.7 Calculations

The following are the calculations for this method:

Calcite Content (HCl, room temperature):

$$\% \text{ Acid soluble} = \frac{\text{initial sample weight} - \text{final sample weight}}{\text{initial sample weight}} \times 100 \quad (19)$$

Total Carbonate Content (HCl, 80°C.):

$$\% \text{ Acid soluble} = \frac{\text{initial sample weight} - \text{final sample weight}}{\text{initial sample weight}} \times 100 \quad (20)$$

$$\% \text{ Dolomite} = \% \text{ total carbonate content} - \% \text{ calcite content} \quad (21)$$

Total Acid Soluble Content (Mud acid):

$$\% \text{ Acid soluble} = \frac{\text{initial sample weight} - \text{final sample weight}}{\text{initial sample weight}} \times 100 \quad (22)$$

7.6.8 Advantages

The technique is uncomplicated and requires simple equipment.

7.6.9 Limitations

Limitations of this method include:

a. The procedure is indicative of maximum rock reactivity, as the sample is disaggregated and the test is conducted in excess acid.

b. Siderite is soluble in HCl at room temperature and, as a result, can distort the calculated calcite content.

7.6.10 Accuracy and Precision

Information not available.

7.6.11 Calibration

The balance must be properly calibrated.

7.7 CORE WATER SALINITY DETERMINATION

A salinity determination on the water present in the core can be desirable because it could aid in core analysis data interpretation and in electric log evaluation.[12,13] Salinity or total dissolved solids (TDS) is defined as the sum of all dissolved materials in terms of milligrams of salt per kilogram of solution (mg/kg) which is equivalent to parts per million (ppm). Salinity is often calculated from the amount of chloride ion present in the core water and is expressed as the equivalent amount of sodium chloride. However, if the salinity is calculated from a resistivity measurement, it represents an NaCl equivalent of the contribution from all soluble electrolyte ions. Measurements of the core water salinity are based on the assumption that all of the chemical ions and gases in the core formation water at reservoir conditions are still contained in the core water at ambient conditions. This may not be the case for brines with ion concentrations near the solubility limits, i.e., soluble at reservoir temperature and pressure but insoluble at ambient conditions.

During the coring operation, the coring fluid (mud) filtrate may invade the core and dilute the formation water. There are three factors to minimize mud filtrate diluting the formation water. The core material should be obtained using a low invasion coring bit, the coring fluid should have a low API fluid loss, core samples for analysis should be taken at well site right after the core surfaces.[14,15,16] The core plugs for core water salinity should be taken from the center of the core, farthest from the perimeter of the core where filtrate invasion will be the greatest (see 4.3.7).[15] To avoid evaporation of water from the core/plug material, the core/plug should be sealed immediately after retrieval (see 2.5). Freezing the whole core after removal from the core barrel to prevent tracer migration and counter-current imbibition is not a recommended alternative to taking a plug sample at well site from the middle of the core material. A salinity front develops as the core freezes which concentrates salts ahead of the freeze line.[17]

If the salinity of the formation water and the mud filtrate are known, the degree of flushing by the coring fluid can be estimated by the core water salinity (but may be affected by nonuniform distributions). Even if the salinity of formation water is not known, the degree of flushing by the water based coring fluid may be estimated if the coring fluid contains a tracer chemical; such as tritiated water, bromide, or iodide, that can be assumed to be at a low concentration in the formation water (see 4.3.7).[18]

If the coring fluid contains a high concentration of potassium, because potassium chloride is the main source of salinity in the coring fluid, then the potassium ion can be used as the tracer. In formation waters, the ratio of sodium to potassium is much greater than one. A cation analysis (sodium and potassium) of the core water provides a method to determine if the core is relatively free of coring fluid. The use of potassium ion as a tracer can only provide a qualitative indication of the extent of coring fluid filtrate invasion compared with the more quantitative assessment using coring fluid tracer chemicals; such as tritiated water, bromide, or iodide. These anions are not chemically attenuated by the rock matrix as is possible for cations like potassium.

7.7.1 Core Water Recovery

To measure salinity of the core water, it must first be separated from the core. This can be achieved by mechanically expelling the core water from the core using an immiscible fluid flush or by centrifugation. When the core water is near irreducible saturation, mechanical expulsion may be unsuccessful. When no brine is expelled by mechanical means, then the salts can be water-extracted from a dried core. The salinity can then be estimated from the amount of salt extracted and the amount of water initially present in the core.

7.7.1.1 Centrifugation

7.7.1.1.1 Principle

Core fluids, including the formation water, may be expelled by centrifuging the sample.

7.7.1.1.2 Apparatus

Recommended apparatus for this method includes:

a. Centrifuge and sample holders.
b. Assorted glassware.

7.7.1.1.3 Procedure

The centrifuge should be of such a size and so driven that a force equal to at least 1,000 times the force of gravity can be exerted on the center of gravity of the core sample. The centrifuge sample holder should be an airtight unit having a perforated bottom with a receiving chamber. Unconfined core samples are placed in weight-matched centrifuge sample holders. The samples are centrifuged for at least one hour, preferably overnight if the sample permeability is less than 100 md. A sample of the produced brine is separated from the fluid mixture centrifuged from the core, with the use of a separator funnel or by pipetting a sample from the brine layer. This sample should be saved for salinity determination.

7.7.1.1.4 Precautions

Precautions for this method include:

a. The temperature of the centrifuge should be controlled to reduce the potential for water loss from the sample holders.
b. Care should be taken to avoid evaporation of water during all steps, starting with handling the core material to transferring of the brine sample to the sample container for salinity determination.

7.7.1.1.5 Advantages

Advantages of this method include:

a. The core sample does not have to have a cylindrical shape or be consolidated.
b. The presence of reservoir crude oil in the core sample does not interfere with this procedure or the validity of the results.

7.7.1.1.6 Limitations

Limitations of this method include:

a. The time required to centrifuge out the brine may be long for low permeability samples.
b. The procedure only works if the core contains a high enough brine saturation such that a portion can be removed at the highest available centrifuge speed.
c. The centrifugal force on the unconfined sample may irreversibly alter the matrix of the rock, changing porosity and permeability, such that the core may not be suitable for subsequent core analysis tests.

d. The salinity of the core water expelled may not be representative of the core water salinity at reservoir pressure and temperature conditions.

7.7.1.1.7 Accuracy/Precision

The accuracy/precision of the separation technique is limited by the ability to preserve core from well site to the laboratory and the subsequent handling of the core samples and the extracted brine sample without altering the salinity of the core water. Evaporation effects for relatively small samples or samples containing very low brine saturations, Sw=<5%, may increase salinity. Percent error will be significantly reduced as the amount of core water volume increases. Careful core handling practices may result in errors in core salinities due to core and brine handling being less than 2% of total salinity.[13]

7.7.1.2 Immiscible Fluid Flush

7.7.1.2.1 Principle

The formation water may be expelled by an immiscible displacement flush.

7.7.1.2.2 Apparatus

Apparatus for this method includes:

a. Hassler or triaxial core holder.
b. Solvent pump.
c. Assorted glassware.

7.7.1.2.3 Procedure

A core plug is mounted in a rubber sleeve Hassler or triaxial core holder cell that will permit fluid to flow through the matrix of the sample. Details of this procedure are given in 3.6.4.1. The water-immiscible fluid could be a refined mineral oil or an organic solvent like toluene. The higher the viscosity contrast between the water-immiscible fluid and water, the more likely that some formation water will be produced during the flush. A sample of the produced brine is separated from the fluid mixture flushed from the core using a separator funnel or by pipetting a sample from the brine layer. This sample should be saved for salinity determination.

7.7.1.2.4 Precautions

Precautions for this method include:

a. The fluid used to expel the formation water should have a low solubility for water so as not to change the salinity of the extracted water.
b. Care should be taken to avoid evaporation of water during handling of the core, flushing, and transferring of the brine to the sample container for salinity determination.

7.7.1.2.5 Advantages

The presence of reservoir crude oil in the core sample does not interfere with this procedure or affect the validity of the results.

7.7.1.2.6 Limitations

Limitations of this method include:

a. For low permeability samples, the time required to flush out the brine may be long.
b. This procedure only succeeds if the core contains a brine saturation which can be reduced by the available applied pressure drop, e.g., core is from water or oil-water transition zone.
c. If the net hydrostatic pressure used to confine the sample while flushing exceeds the net reservoir overburden pressure, the sample porosity may be irreversibly compacted and the sample may not be suitable for subsequent core analysis tests.
d. The core sample must have cylindrical shape.
e. The salinity of the core water expelled may not be representative of the core water salinity at reservoir pressure and temperature conditions.

7.7.1.2.7 Accuracy/Precision

See section 7.7.1.1.7.

7.7.2 Water Extraction of Core Salt

Core water salts are extracted from a dried core sample.

7.7.2.1 Apparatus

Apparatus for this method includes:

a. Mortar and pestle.
b. Balance.
c. Desiccator.
d. Assorted glassware.

7.7.2.2 Procedure

Approximately 50 grams of sample are selected. The water saturation must be known. The preferred method is to use a portion of the sample used in a previous saturation determination test. Less preferably, the sample may be selected from core material at a point as close as possible to the sample used in the saturation measurement test.

The sample must be free of hydrocarbons. Core material from a Dean-Stark toluene extraction is suitable. The sample is ground in a mortar to approximately 16-mesh size and dried in an oven to constant weight. After being cooled in a desiccator, the sample is weighed and transferred to a flask. 100 ml of distilled water is added and the mixture is stirred vigorously for several minutes. Agitation is continued intermittently for a minimum of one hour. The resulting salt solution is filtered or decanted and saved for salinity determination.

7.7.2.3 Precautions

Precautions for this method include:

a. If the core sample is suspected of containing clay material, a constant humidity oven should be used at a temperature of about 140°F (60°C) and 45 percent humidity during the drying step for determining core water content. This is done to avoid removing chemically- and physically-bound water (water that does not contribute to the salinity),[19] and thus to avoid overstating the amount of core water, which results in underestimating the core water salinity. Since constant humidity drying under these conditions does not dehydrate clays, no detectable fraction of the 100 ml of water added to the sample will be used to rehydrate the clays.
b. Care should be taken to avoid evaporation of water during all steps, starting with core preservation to transferring of the brine sample to sample container for salinity determination.

7.7.2.4 Advantages

This procedure is simple and can be conducted with very little equipment in a short time.

7.7.2.5 Limitations

Limitations of this method include:

a. The presence of hydrocarbons in the core sample interferes with the determination of core water content. The more volatile components of the hydrocarbons will be lost during the drying step, and thus the amount of water present will be overstated.
b. If the core material contains any sulfide minerals, such as pyrite that has oxidized during core handling, storage, or during extraction, the amount of salts extracted will be overstated. Oxidation of pyrite will produce water soluble iron sulfate salts.
c. If the in situ core rock matrix includes solid phase soluble salts (that are in solubility equilibrium with the formation brine), i.e., anhydrite or sodium chloride, then during drying and extraction these salts may be extracted because of the

high water to rock ratio during extraction, and the salinity of the core water will be overstated.

d. The sample is destroyed.

e. The calculated salinity of the core water may not be representative of the core water salinity at reservoir pressure and temperature conditions.

7.7.3 Determination of Core Water Salinity

The salinity of the water is determined, using either: (a) chemical determination of the chloride ion, (b) refractive index, or (c) resistivity measurement. The salinity is expressed as parts of sodium chloride per million parts of core water (although other salts are usually present), which is equivalent to milligrams of sodium chloride per kilogram of solution (sodium chloride and water).

There are several other analytical techniques for measuring the salt content of the core water. These include ion chromatography, atomic absorption, and inductively coupled plasma spectroscopy. A combination of these methods can accurately determine the cationic and anionic composition of core water, but the equipment is much more expensive than that required by procedures described in 7.7.3.1, 7.7.3.2, and 7.7.3.3.

7.7.3.1 Chemical Determination of Chloride Ion

The chloride concentration is measured in the undiluted brine solution expelled from the core or the filtrate obtained by separation of the crushed sample and the water used in the leaching process. The salinity of the water is expressed as parts per million sodium chloride in the core water (mg/kg).

7.7.3.1.1 Apparatus and Procedure

The apparatus, reagents, and procedure for chloride determination are described in detail in API RP 45,[20] *Recommended Practices for Analysis of Oilfield Waters*, and also in ASTM D512-89,[21] *Standard Test Methods for Chloride Ion in Water*. Of the four ASTM methods listed, Method A (Mercurimetric Titration), Method B (Silver Nitrate Titration), Method C (Colorimetric), and Method D (Ion-Selective Electrode), Method B is preferred. The calculation described in this reference will give the salinity in terms of chloride ion instead of sodium chloride. The calculation as sodium chloride is described in 7.7.3.1.3.

7.7.3.1.2 Precautions

Precautions are as follows:

a. Brine sample should be diluted to fall within optimum range of the titration solutions and equipment.

b. Make blank chloride determinations to correct for any chloride present in the glassware and water used in the tests.

7.7.3.1.3 Calculations

The following calculations are used:

a. The salinity of the core water expelled from the core sample can be calculated directly. The normality of salt solution measured by a chloride titration can be converted to sodium chloride concentration, mg/kg, as follows:

$$\text{Conc. of NaCl (mg/kg)} = 1{,}000 \times N_1 \times MW_{NaCl}/D_w \qquad (23)$$

Where:

$$N_1 = V_2 \times N_2/V_1 \qquad (24)$$

N_1 = normality of salt solution titrated, meq/ml.
V_1 = volume of salt solution titrated, ml.
N_2 = normality of chloride reagent (i.e., silver nitrate), meq/ml.
V_2 = volume of chloride reagent at titration endpoint, ml.
D_w = density of core water, gm/ml.
MW_{NaCl} = equivalent weight of NaCl = 58.5 gm/equ.

b. When core water cannot be expelled and the salts are water extracted, the salinity of the originally-present core water must be back-calculated. The salinity from the chemical determination method is converted to total milligrams of sodium chloride leached from the sample. This number represents the total salt from the core water of the sample. The total salt in milligrams and the weight of the volume of core water obtained by saturation tests are used to calculate the concentration of salt in the core water, and this value is expressed in parts per million of sodium chloride.

The milligrams of sodium chloride leached from the core sample determined from the chloride titration are calculated as follows:

$$\text{Weight of NaCl (mg)} = N_1 \times 58.5 \times V_w \qquad (25)$$

Where:

$$N_1 = V_2 \times N_2/V_1 \qquad (26)$$

N_1 = normality of salt solution titrated.
V_1 = volume of salt solution titrated, ml.
N_2 = normality of chloride reagent (i.e., silver nitrate).
V_2 = volume of chloride reagent at titration endpoint, ml.
V_w = volume of water used to leach or extract the sample, ml.

The weight of the core water can be obtained directly by difference between the weight of the sample before and after drying, if no oil is present. The drying should follow the directions as given in 5.3.2.2.3.6.

The salinity (mg/kg) of the core water is obtained by:

$$\text{mg NaCl/kg of core water} = \frac{\text{mg NaCl}}{\text{(gm core water + gm NaCl)}} \times 1000.0 \qquad (27)$$

If the sample contains oil, the following calculations can provide an approximate salinity value. The fluid saturations and the porosity must be determined either on this sample or an adjacent core. The amount of water contained in the sample can be calculated as follows:

$$\text{Weight of core water, gm} = V_b \times \emptyset \times S_w \times D_w \quad (28)$$

Where:

$$V_b = [(W/D_g)/1 - \emptyset)] \quad (29)$$

W = dry weight of sample, gm.
D_g = grain density, gm/cc.
$\emptyset$ = porosity, as fraction.
V_b = bulk volume of sample, cc.
S_w = water saturation, as fraction of pore volume.
D_w = density of core water, gm/ml.

If the grain density has not been determined, use a representative value for the formation of interest. The density of the core water is assumed to be 1.00 as an initial estimate. After the salinity is calculated with Equation 27, the weight of the core water, Equation 28, is recalculated using a new density based on the previously-calculated salinity; and a new salinity is recalculated using Equation 27.

7.7.3.1.4 Advantages

Advantages of this method are as follows:

a. Method is rapid and convenient.
b. Chloride determination is accurate.

7.7.3.1.5 Limitations

Limitations of this method include:

a. Result is expressed as sodium chloride. Anions other than chloride, such as sulfate and bicarbonate, present in the solution are not detected.
b. Other halides, such as bromide and iodide, are determined as chloride.

7.7.3.1.6 Accuracy/Precision

When the core water can be recovered from the core by centrifugation or an immiscible flush, the accuracy in the determination of salinity (mg/kg) is controlled by the salinity determination technique, chloride titration, on the core water sample which is about ±1 to 2% of the total salinity. API Recommended Practice 45 reports a precision of about 1% and an accuracy of about 2% of the amount present. When the core water salts have to be extracted from a dried core, the accuracy in the determination of core water salinity (mg/kg) is limited by the determination of the water content.[13] McCoy, et al.[13] report field data where chloride salinity determinations on oil-based-mud (OBM) cores intersecting the oil-water contact agreed within 1% on the average with produced water salinity. Chloride concentration measurements were obtained on the water extracts of about 2,000 OBM dried core samples (using procedures similar to 7.7.2 and 7.7.3).

7.7.3.2 Refractive Index Measurement

The total ionic constituents of water may be estimated by measuring the refractive index. The refractive index varies in a direct manner with the ionic concentration of sodium chloride and other salts. Standard graphs, which show the refractive index value for various salinities and temperatures of sodium chloride solutions, can be prepared from data in the literature (see Figure 7-2).[22,23] If the refractive index is known, the salinity of the core water, expressed as parts per million of sodium chloride (mg/kg), can be determined from such a graph using a measured refractive index value.

7.7.3.2.1 Apparatus

Apparatus for this method includes:

a. Refractometer.
b. Assorted glassware.

7.7.3.2.2 Procedure

The instrumentation should be calibrated with known concentrations of sodium chloride solutions, resulting in a graph similar to that shown in Figure 7-2. Details on apparatus and procedures may be found in ASTM D542-85, *Standard Test Method for Pore Water Extraction and Determination of the Soluble Salt Content of Soils by Refractomer.* The refractive index is measured on brine sample. With the use of a standard graph (see Figure 7-2) and suitable calculations, the refractive index value is converted to a salinity value for the core water.

7.7.3.2.3 Precautions

Refractive index value must be corrected to a standard temperature.

7.7.3.2.4 Advantages

Advantages of this method include:

a. Rapid determination.
b. The total equivalent sodium chloride concentration is determined.

7.7.3.2.5 Limitations

All ions present in the water are calculated as sodium chloride.

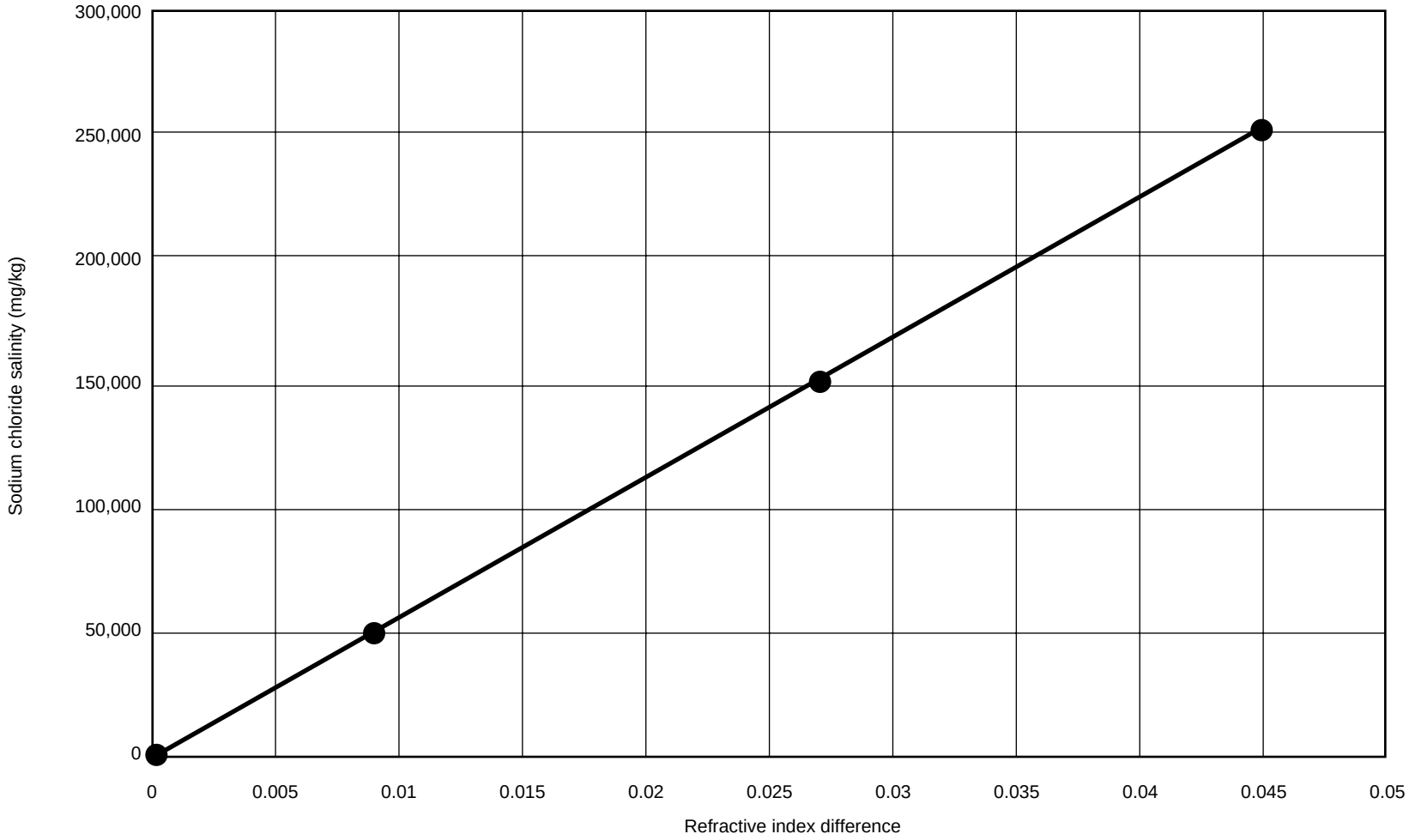


Figure 7-2—Refractive Index (RI) Difference Versus Salinity (RI of Solution—RI of Distilled Water)

7.7.3.2.6 Accuracy/Precision

See 7.7.3.1.6 for general comments. According to ASTM D4542, salinity determination using the refractometer should be accurate within ±300 mg/kg salinity.

7.7.3.3 Resistivity Measurement

The total ionic constituents of water may be estimated by measuring the resistivity. The resistivity varies in an inverse manner with the ionic concentration of sodium chloride and other salts over a wide range of concentrations. Standard graphs, illustrated by Figure 7-3, show the resistivity value for various salinities and temperatures of sodium chloride solutions. If the resistivity is known, then the salinity of the core water, expressed as parts per million of sodium chloride (mg/kg), can be determined from such a graph using a measured resistivity value.

7.7.3.3.1 Apparatus

Apparatus for this method includes:

a. Resistivity cell.
b. Resistivity meter.
c. Assorted glassware.

7.7.3.3.2 Procedure

The resistivity cell should be calibrated with known concentrations of sodium chloride solutions, resulting in a graph similar to that shown in Figure 7-3. A portion of the solution obtained by leaching the core is placed in the resistivity cell. The resistivity is measured on a suitable meter and calculated in ohmmeters. With the use of a standard graph (Figure 7-3) and suitable calculations, the resistivity value is converted to a salinity value for the core water. Additional details on apparatus and procedures may be found in ASTM D1125-82, *Method of Test for Electrical Conductivity for Water*.[24,25]

7.7.3.3.3 Precautions

Precautions for this method include:

a. Resistivity meter and cell must be calibrated.
b. Resistivity value must be corrected to a standard temperature.
c. Be certain that electrodes of the resistivity cell are clean before making a measurement.
d. Calibrate electrode with known standard salt solution over the entire range of use to determine/check cell constant as described by Worthington, et al.[25]
e. Hydrogen sulfide may cause some electrodes to change calibration.

7.7.3.3.4 Calculations

Calculations for this method are as follows:

a. The salinity of the core water expelled from the core sample can be calculated directly. The resistivity of the salt solution can be converted directly to concentration milligrams of salt per kg of solution, using a conversion chart of the type illustrated in Figure 7-3.[20]
b. When core water cannot be expelled and the salts are water extracted, the salinity of the originally present core water must be back calculated. The salinity from the resistivity measurement method is converted to total milligrams of sodium chloride leached from the sample. This number represents the total salt from the core water of the sample. The total salt in milligrams and the weight of the volume of core water obtained by saturation tests are used to calculate the concentration of salt in the core water, and this value is expressed in parts per million of sodium chloride. The salinity (mg/kg) of the core water is obtained by following the calculation outlined in 7.7.3.1.3b.

7.7.3.3.5 Advantages

Advantages of this method include:

a. Rapid determination.
b. The resistivity data relate directly to electric log measurements.
c. The total equivalent sodium chloride concentration is determined.

7.7.3.3.6 Limitations

All ions present in the water are calculated as sodium chloride.

7.7.3.3.7 Accuracy/Precision

See 7.7.3.1.6 for general comments and to ASTM D1125[24] for resistivity specific information.

7.7.3.4 Determination of Cations

The direct analysis for sodium, potassium, calcium, and magnesium allow for the estimation of core water salinity and may provide a method to determine if the core water is free of coring fluid. Other cations are present in formation waters but typically account for less than 5 percent of the salinity. Core water salinity can also be estimated from the concentrations of these four cations.

The concentration of these cations can be measured rapidly, simultaneously, and accurately on an inductively coupled plasma atomic emission spectrometer (ICPES). These cations can also by determined individually with an atomic absorption spectrometer (AAS).

Some modern drilling/coring fluids contain high concentrations of potassium. In most formations waters, the ratio of sodium to potassium is much greater than one. Thus for

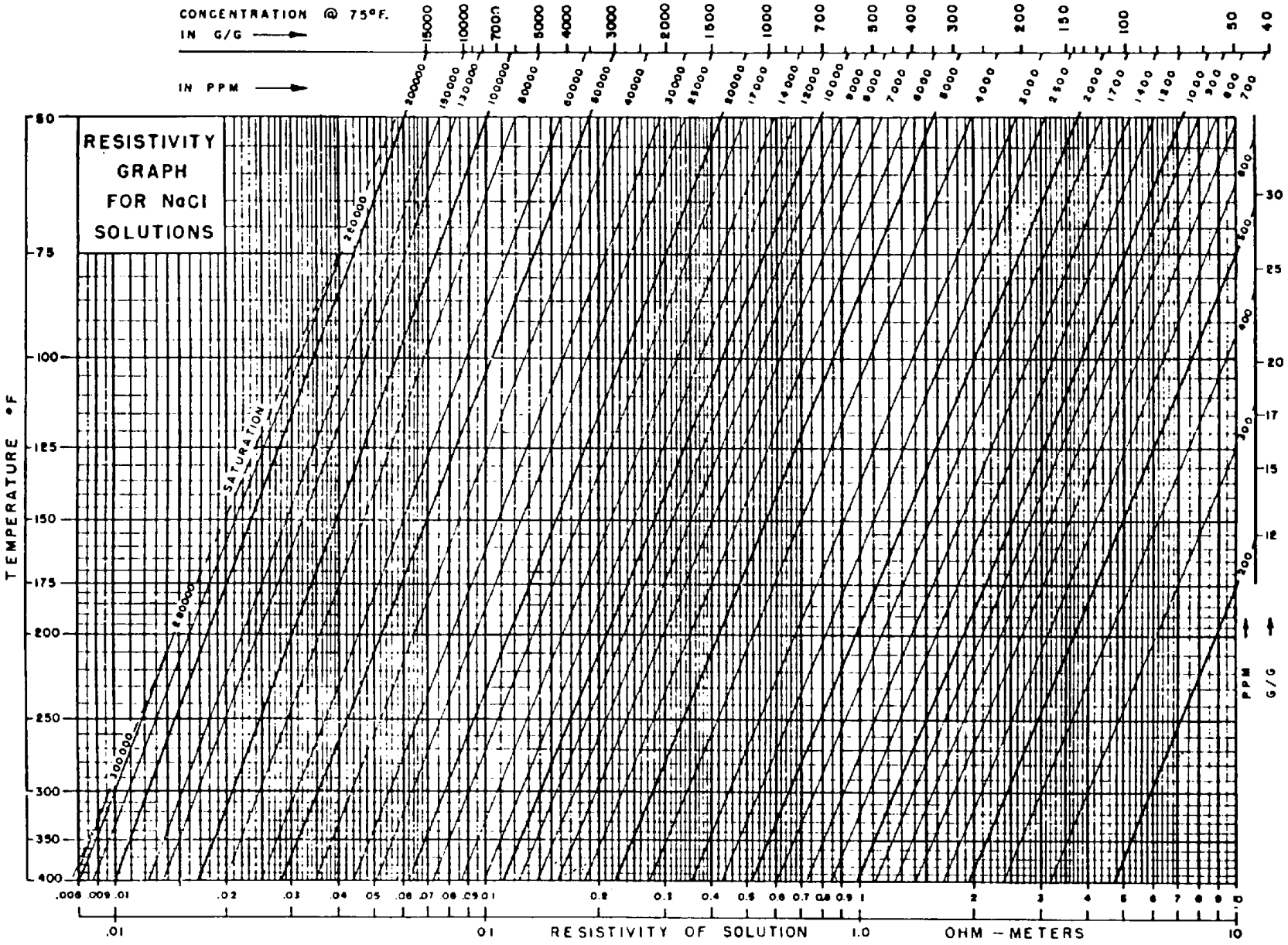


Figure 7-3—Conversion Chart for Salinity Determination Resistivity Versus Temperature and Salinity

potassium-based coring fluids, invasion can be estimated based on the ratio of potassium to sodium in the core water.

7.7.3.4.1 Apparatus and Procedure

The apparatus and procedure for cation determination using AAS are described in detail in ASTM D4191[26] for sodium, ASTM D4192[27] for potassium, and ASTM E508[28] for calcium. The apparatus and procedure for cation determination using ICPES are described in detail in ASTM D1976.[29]

7.7.3.4.2 Precautions

Precautions for this method include:

a. For salinity calculations, the anion is assumed to be chloride even though there are commonly significant sulfate and bicarbonate ions and smaller amounts of other ions.
b. Coring fluid invasion estimation using Na/K ratio requires a knowledge of the interaction of potassium with the sodium bentonite clay in the coring fluid and with the clays in the formation rock.
c. An understanding of local evaporites and their effects on sample preparation is required.
d. The analysis should be performed by personnel trained in these techniques and in a laboratory with the equipment permanently installed.

7.7.3.4.3 Calculations

The methods described in 7.7.3.1.3 for calculating sodium chloride concentration in the undiluted brine and the core leachate can be used to calculate the concentration of cations by replacing the molecular weight of sodium chloride with the atomic weight of the cation.

7.7.3.4.4 Advantages

Advantages of this method include:

a. Both ICPES and AAS are accurate analytical methods.
b. Coring fluid invasion estimates can be made with these data.
c. Cation analysis can provide a more complete picture of the brine salinity instead of assuming that sodium is the only cation present when total salinity is based only on chloride analysis.

7.7.3.4.5 Limitations

The lack of availability and high cost of ICPES and AAS analyses are a limitation.

7.7.3.4.6 Accuracy/Precision

Refer to 7.7.3.1 for general comments and to ASTM D4191, D4192, E508, and D1976 for more specifics.[26-29] In general, AAS and ICPES should be able to determine the concentration of these cations with an accuracy and precision of 1% to 2%.

7.8 REFERENCES

1. ASTM Specification E-11, *Specification for Wire Cloth Sieves for Testing Purposes*, American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19248.
2. ASTM C-136, *Standard Method for Sieve Analysis of Fine and Coarse Aggregates*, American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19248.
3. ASTM C-702, *Practice for Reducing Field Samples of Aggregate to Testing Size*, American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19248.
4. Chayes, F., *Petrographic Modal Analysis, An Elementary Statistical Appraisal*, Wiley, New York, 1956, p. 113.
5. Blatt, H., Middleton, G., and Murray, R., *Origin of Sedimentary Rocks*, Prentice-Hall, Inc., New Jersey, 1980, p. 782.
6. Kellerhals, R., Shaw, J., and Arora, V. K., "On Grain Size From Thin Sections," *Journal of Geology*, Volume 83, 1975, pp. 79-96.
7. Rosenfeld, M. A., Jacobson, L., and Ferm, J. C., "A Comparison of Sieve and Thin Section Techniques for Size Analysis," *Journal of Geology*, Volume 61, 1953, pp. 114-132.
8. Mazullo, J. and Kennedy, S., "Automated Measurement of the Nominal Sectional Diameters of Individual Sedimentary Particles," *Journal of Sedimentary Petrology*, Volume 55 (4), July 1985, pp. 593-595.
9. Gibbs, R. J., Matthews, M. D., and Link, D. A., "The Relationship Between Sphere Size And Settling Velocity," *Journal of Sedimentary Petrology*, Volume 41, No. 1, March 1971, pp. 7-18.
10. ASTM D287-55, *Method of Test for API Gravity of Petroleum*,* American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19248.
11. ASTM-IP, *Petroleum Measurement Tables* (American Edition), American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19248.
12. Coleman, M.L., Jones, M.R.O., and Cox, M.A., "Analysis of Formation Water Sample from Core," *North Sea Oil and Gas Reservoirs—II*, Graham and Trotman (1990).
13. McCoy, D.D., Warner, H.R. Jr., and Fisher, T.E., "Water Salinity Variations in the Ivishak and Sag River Reservoirs, Prudhoe Bay Field," SPE 28577, presented at the SPE 69th annual Technical Conference and Exhibition, September 25-28, 1994, New Orleans, Louisiana.

14. Wylie, G.S., Rathmell, J.J., and Tibbitts, G.A., "Coring Technology for Point McIntyre Wells," SPE 26353, presented at the SPE 68th annual Technical Conference and Exhibition, October 3-6, 1993, Houston, Texas.
15. Bloys, J.B., Rathmell, J.J., Kriel, W.A., Spence, A.P., Wylie, G.S., and Ray, C.R., "Development and Application of Mineral Oil Base Mud and Tracer for Low Invasion Coring at Point McIntyre," SPE 26326, presented at the SPE 68th annual Technical Conference and Exhibition, October 3-6, 1993, Houston, Texas.
16. Tibbitts, G.A., Reed, M.G., and McCarter, M., "New Coring System Reduces Filtrate Invasion," presented at the Society of Core Analysts, First European Core Analysis Symposium, London, U.K., May 21-23, 1990.
17. Gidman, J., and Conner, F.J., "The Effects of Freezing on Tracer Distribution in Cores," *Proceedings of the Society of Core Analysts*, Third European Core Analysis Symposium, London, U.K., September 14-16, 1992.
18. Brown, A., and Marriott, F.T., "Use of Tracers to Investigate Drilling-Fluid Invasion and Oil Flushing During Coring," *SPE Reservoir Engineering*, 1988, pp. 1317-1322.
19. Nagra, S.S., Nelson, H.W., and Gorden, H.M., "Interpretation of Water Salinities Obtained from Oil Based Cores in the Deep Basin of Western Canada," Paper No. 82-33-53, presented at 33rd Annual Technical Meeting of the Petroleum Society of CIM, Calgary, 1982.
20. API Recommended Practice 45, *Recommended Practice for Analysis of Oil-Field Waters*, Second Edition, November 1968 (reissued July 1981), American Petroleum Institute, 1220 L Street, N.W., Washington, D.C. 20005.
21. ASTM D512, *Standard Test Method for Chloride Ion in Water*, American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19248.
22. ASTM D4542, *Standard Test Method for Pore Water Extraction and Determination of Soluble Salt Content of Soils by Refractometer*, American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19248.
23. Washburn, E.W., editor, *International Critical Tables of Numerical Data, Physics, Chemistry and Technology, Volume VII*, First Edition, McGraw-Hill, New York, 1930, p. 73.
24. ASTM D1125, *Method for Test for Electrical Conductivity for Water*, American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19248.
25. Worthington, A.E., Hedges, J.H., and Pallatt, N., "SCA Guidelines for Sample Preparation and Porosity Measurement of Electrical Resistivity Samples, Part I—Guidelines for Preparation of Brine and Determination of Brine Resistivity for Use in Electrical Resistivity Measurements," *The Log Analyst*, 1990, 20-28.
26. ASTM D4191, *Standard Test Method for Sodium Water by Atomic Absorption Spectrophotometry*, American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19248.
27. ASTM D4192, *Standard Test Method for Potassium in Water by Atomic Absorption Spectrophotometry*, American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19248.
28. ASTM E508, *Standard Test Method for Calcium and Magnesium in Iron Ores by Atomic Absorption Spectrophotometry*, American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19248.
29. ASTM D1976, *Standard Test Method for Elements in Water by Inductively-Coupled Argon Plasma Atomic Emission Spectrophotometry*, American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19248.

7.9 BIBLIOGRAPHY

7.9.1 Grain/Particle Size Distribution

a. Anderson, J. B. and Kurtz, D. D., "RUASA, An Automated Rapid Sediment Analyzer," *Journal of Sedimentary Petrology*, Volume 49, 1979, pp. 625-627.

b. Anderson, J. B., Wolfteich, C., Wright, R., and Cole, M.L., "Determination of Depositional Environments of Sand Bodies Using Vertical Grain-Size Progressions," *Transactions Gulf Coast Association of Geological Societies*, Volume XXXII.

c. API RP 60, *Recommended Practices for Testing High Strength Proppants Used in Hydraulic Fracturing Operations*, First Edition, February 1989, pp. 1-21.

d. API RP 58, *Recommended Practices for Testing Sand Used in Gravel Packing Operations*, First Edition, March 1986, pp. 1-13.

e. API RP 56, *Recommended Practices for Testing Sand Used in Hydraulic Fracturing Operations*, First Edition, March 1983, pp. 1-13.

f. ASTM C136-849, *Standard Method for Sieve Analysis of Fine and Coarse Aggregates*, December 1984, pp. 76-79.

g. ASTM C702, *Standard Methods for Reducing Field Samples of Aggregate to Testing Size*, March 1975, pp. 421-424.

h. ASTM C-117-69, *Standard Method of Test for Materials Finer Than No. 200 (75 Micro-meter) Sieve in Mineral Aggregates by Washing*, October 1969 (Reapproved 1974), pp. 69-70.

i. Komar, P. D., and Chi, B., "The Analysis Of Grain Size Measurement By Sieving And Settling-Tube Techniques," *Journal of Sedimentary Petrology*, Volume 54, No. 2, June 1984, pp. 603-614.

j. Griffin, T. G., "Extended Range Particle Size Distribution Using Laser Diffraction Technology, A New Perspective," *Proceedings of the Society of Core Analysts*, Fifth Technical Conference, August 1991, No. 9126.

k. Saucier, R. J., "Considerations in Gravel Pack Design," *Journal of Petroleum Technology*, February 1974, pp. 205-212.
l. Frock, H. N. and Weiss, E. L., "Particle Size Control Using Light-Scattering Technology," *Powder and Bulk Engineering*, February 1988.
m. Weiss, E. L. and Frock, H. N., "Rapid Analysis of Particle Size Distribution by Laser Light Scattering," *Powder Technology*, 1976, pp. 287-293.

7.9.2 Oil Gravity

a. *API Manual of Petroleum Measurement Standards*, Chapter 9, "Density Determination", Section 1, "Hydrometer Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products", June 1981, pp. 1-7.
b. Tutunjian, P.N. and Vinegar, H.J., "Petrophysical Applications of Automated Proton High Resolution NMR Spectroscopy," SCA 4th Annual Technical Conference Paper, August 1990, No. 9020.
c. Vinegar, H.J., Tutunjian, P.N., Edelstein, W.A., and Roemer, P.B., "13C NMR of Whole Cores," 64th Annual Conference of the Society of Petroleum Engineers, October 1989, No. 19590, pp. 201-221.
d. Karger, J., Pfeifer, H., and Heink, W., *Principles and Application of Self-Diffusion Measurements by Nuclear Magnetic Resonance, Advances in Magnetic Resonance*, ed. John Waugh, Academic Press, New York, pp. 1-89.

7.9.3 Core-Water Salinity Determination

a. ASTM C136-849, *Standard Test Method for Pore Water Extraction and Determination of Soluble Salt Content of Soils by Refractometer*, ASTM Comm. D-18, pp. 746-748.
b. Brown, A.A., Berry IV, W.R., Coates, G.R., Fertl, W.H., Hoyer, W.A., Patchett, J.G., and Ransom, R.C.; W.H. Lang, Jr., chairman, "SPWLA Reprint Volume Shaly Sand," Society of Professional Well Log Analysts, July 1982, Section V, *Cation Exchange Models*.
c. Hill, H.J., Shirley, O.J., and Klein, G.E., edited by Waxman, M.H., and Thomas, E.C., "Bound Water in Shaly Sands—Its Relation to Qv and Other Formation Properties," *The Log Analyst*, Volume XX, No. 3, 1979.
d. API RP 45, *Recommended Practice for Analysis of Oil-Field Waters*, American Petroleum Institute, July 1981.

SECTION 8—REPORTING

CONTENTS

Page

8 REPORTING 8-1
8.1 Introduction 8-1
8.2 Tabular Report 8-1
8.3 Graphical Report 8-1
8.4 Digital Report 8-1
8.5 Quality Assurance 8-7
8.6 QA/QC Protocol 8-8

Figures
8-1 Permeability vs. Porosity 8-4
8-2 Core Data Profile 8-5
8-3 Example of DCIS Logical File 8-7

Tables
8-1 Core Analysis Wellsite Data 8-2
8-2 Basic Core Analysis Laboratory Data. 8-3
8-3 Basic Core Plug Analysis Report 8-8
8-4 Basic Full Diameter Core Analysis Report. 8-9
8-5 Basic Sidewall Core Analysis Report 8-9
8-6 Basic Oil-Wet Sponge Core Analysis Report 8-10
8-7 Basic Full Diameter Pressure-Retained Core Analysis Report 8-10
8-8 Basic Core Fracture Analysis Report 8-11
8-9 Standard Abbreviations for Lithologic Descriptions 8-12
8-10 Standard Abbreviations for Lithologic Descriptions 8-18
8-11 Units and Conversions 8-24
8-12 Nomenclature 8-26

8 Reporting

8.1 INTRODUCTION

An essential part of a core analysis program is the data documentation phase. Typical data reporting formats are tabular, graphical, and/or digital. These constitute the permanent record(s) of the testing program. Such reports are furnished to the user and are filed by the testing laboratory in accordance with sound archiving procedures.

Tables 8-1 and 8-2 are developed for data documentation at the wellsite and in the laboratory. Commentary which may serve to aid in the understanding and/or the interpretation of the data should be furnished by the way of attachments and/or footnotes. Deviations from accepted standard practices must be noted. Such deviations may include: (a) unusual coring conditions, (b) alterations to the planned coring fluid design, (c) necessary changes in planned on-site or laboratory core handling/preservation methods, and (d) unusual laboratory procedures, etc. The report should contain sufficient wording so as to leave no question or doubt as to the testing methods that were employed.

8.2 TABULAR REPORT

A tabular report should include all of the data, positively identified and tabulated, in some convenient form. Identification should include such items as listed in Tables 8-1 and 8-2. Porosity values should be reported in percent to 0.1 whereas the grain density values should be reported to 0.01 g/cm^3 accuracy. The permeability values should be reported with no less than three significant figures. The specific presentation of data may be determined between the user and the analysts. The suggested numbers of significant figures to be reported are as follows:

Porosity values to	3	(e.g. 0.251 fraction or 25.1%)
Grain density values to	3	(e.g. 2.65 g/cm^3)
Saturation values to	3	(e.g. 50.5% pore volume)
Pore volume values to	4	(e.g. 21.85 cm^3)
Permeability values to	3	(e.g. 2.55 x 10^{+3} md)

8.3 GRAPHICAL REPORT

Graphical presentations are often included to provide the user with a pictorial overview of various core data. Through the continued advances in computer graphic software a large number of pictorial formats are readily available including crossplots, histograms, and core data profiles (logs). The selection of any specific format is left to the discretion of each individual core analyst; however, two graphs have been widely accepted and are recommended for inclusion in every basic core analysis report: a *Permeability vs. Porosity* plot and a *Core Data vs. Depth (sub-surface or sub-sea)* plot.

8.3.1 Permeability vs. Porosity Crossplots

A plot of permeability vs. porosity, with porosity plotted on a linear x-axis and permeability on a logarithmic y-axis, has been included in most conventional core analysis (percussion sidewall data reports rarely contain these crossplots) reports (see Figure 8-1). Also, some best-fit lines are “estimated” while others are derived by curve-fitting procedures. The latter is not always the better approach, depending on the amount of data and the presence of out-liers or fliers that may or may not impact a mathematical approach. A standard labeling procedure should include the essential data identification as adopted through the rest of the core report, as well as any assumptions, notations, or scaling unit parameters unique to each specific plot. While the selection of axis scaling is data dependent and may vary according to the intent of the analyst, a general recommendation is made to select an appropriate scale which will allow the data to fill the entire plot while maintaining easily interpolated axis divisions such as unit divisions of 2, 5, or 10.

8.3.2 Core Data Profiles

The core profile log is a multi-plot figure sometimes referred to as a “core analysis log” (see Figure 8-2). This multi-plot provides a pictorial overview of a variety of core data as a function of sample depth. Generally included on this plot is a gamma ray log, a log of permeability, porosity, reservoir fluids saturation, and possibly a log of bulk or grain density. The recommended standard for vertical scale is 5 inches per 100 feet of depth. The horizontal scale will vary with a log and should be individually selected to allow the data to span the entire plot. A standard labeling procedure should include the essential data identification, as adopted through the rest of the core report, as well as any assumptions, notations, or scaling unit parameters unique to each specific plot.

8.4 DIGITAL REPORT

Current and emerging technologies provide means for core analysis data to be recorded automatically in a digital format. Initial (raw) and calculated (final) data storage, as a result, is being done increasingly on computer systems. Current technology offers a wide choice of data gathering and storage methods. A standardized method of data reporting within the petroleum industry could reduce the cost and effort required to translate data between the various computer platforms.

The Digital Core Analyses Interchange Standard (DCIS) defines the logical data organization on any type of physical

Table 8-1—Core Analysis Wellsite Data

Prepared By: _____ Date: M M D D Y Y Core Number _____

Operator _____ Coring Company _____ Core Analysis Company _____

Contract/Phone _____ Contact/Phone _____

Well Identification _____ Field Reservoir _____ Address _____

API Number _____ ❑ Vertical Well ❑ DeviatedWell ❑ Sidetrack _____

County/State/Country _____ Legal Location/Block _____ Elevations: _____ Ground/Mean Sea Level/ _____ Kelly Bushing ____ Derrick Floor

Persons Requesting Work _____ Phone _____ Fax _____ Address _____

Report Results To _____ Phone _____ Fax _____ Address _____

Alternate _____ Phone _____ Fax _____ Address _____

Rig Contract _____ Phone _____ Fax _____ Address _____

Coring/Drilling Fluid
Type/Contents _____ Weight _____ ppg Funnel Viscosity _____ sec Water Loss _____ cm^3/30 min Chlorides _____ ppm pH _____ Tracer _____

Type of Core
❑ Conventional _____ Inner Barrel Type _____
❑ Sponge ❑ Oriented Barrel
❑ Pressure Retained ❑ Other _____
Sidewall: ❑ Percussion or ❑ Mechanically Drilled Number Attempted _____ Number Recovered _____

Analysis Planned
❑ Plug Size or ❑ Full Diameter
Method
❑ Fluid Saturation _____
❑ Porosity _____
❑ Grain Density _____
❑ Permeability _____
❑ Surface Gamma Log _____
❑ Special Instructions _____

Transportation
❑ By Service Company _____
❑ Ground _____
❑ Air _____
❑ Other _____
Carrier _____
Date Shipped _____

Preservation
Method ❑ Plastic Laminate/Type _____
❑ Freeze ❑ Dry Ice or ❑ Liquid Nitrogen
❑ Refrigerate _____ °F _____°C
❑ Core Inner Barrel
❑ Core Wrap and/or ❑ Dip-Type ❑ Resination
❑ Other

Exposure Time/Climate _____
Notes _____

Laboratory/Long Term Preservation _____
Special Instructions _____
Post Core Analysis Instructions/Core Material Distribution _____

Formation(s)/ Zones _____
Expected Core Point _____
_____
Expected Core Point _____
_____
Expected Core Point _____

Depths (Drillers)
Interval Cut _____
Interval Recovered _____
Interval Cut _____
Interval Recovered _____
Interval Cut _____
Interval Recovered _____

Well Inclination _____ Core Diameter _____ ❑ In ❑ cm
Total Core Recovered _____ Bit Type _____
Length Cored _____ ❑ ft ❑ m Percent Recovery _____
Coring Time _____ Trip Time _____
Estimated Connate Water Salinity _____ ppm Chlorides or Estimated R_W _____ @ _____ ❑ °F ❑ °C
Estimated Production ❑ Oil _____ °API
❑ Condensate
❑ Dry Gas

Notes _____

Attach Coring Log and Core Description

Table 8-2—Basic Core Analysis Laboratory Data

General Information
Analysis Requested Date [M][M][D][D][Y][Y] API # _____
File # _____

Requested By: _____

❑ Full Diameter or ❑ Plug : Size _____ ❑ In ❑ cm

Measurement	Method/Special Instructions
❑ Saturations (O,W,G)	_____
❑ Porosity	_____
❑ Permeability	_____
❑ Grain Density	_____
❑ Other	_____

Objective of the tests as agreed by parties involved _____

_____

Transportation and Inventory

Carrier _____ Date Shipped _____ Core Arrival Date _____

Core condition on arrival:

❑ Preserved ❑ Frozen ❑ Unpreserved ❑ Cleaned ❑ Other _____

Correlation Depths: Driller's _____ Log _____ TVD _____ ❑ ft ❑ m

Allocation of the missing core dinterval: ❑ Bottom ❑ Middle ❑ Top _____

Screening

❑ CT ❑ X-ray ❑ NMR ❑ Fluroscopy ❑ Other _____

Sample Handling

Core Gramma (yes/no, type) _____

Lab Preservation (prior to analyses) _____

Sampling Method

Sample Sleeve ❑ Yes ❑ No, Type _____

Nominal Sample Diameter/Length_____ ❑ In ❑ cm

Plugging/Trimming Fluid_____

Treatment prior to testing (flushing, evaluation, resaluration, etc.) _____

_____

Core Disposition (removed samples, storage location, preservation, etc.)_____

_____

Lab Analyst _____

Other Service

❑ Slab ❑ Resination ❑ Photo: ❑ BW ❑ Color ❑ U.V. ❑ Other _____

Supplementary Tests _____

_____

Methods and Conditions

Cleaning:	Methods	Conditions
	❑ No Cleaning	Solvents _____
	❑ Dean-Stark	Temperature _____ ❑ °F ❑ °C
	❑ Soxhlet	Pressure _____ ❑ psi ❑ kPa
	❑ CO2/Solvent	Time _____
	❑ Flow Through	Volume and Rate_____ ❑ cc ❑ cc/sec
	❑ Others	Others _____
Drying:	Methods	Conditions
	❑ Convection oven	Temperature _____ ❑ °F ❑ °C
	❑ Vacuum oven	Time _____
	❑ Humidity oven	Relative Humidity % _____
	❑ Others	

Porosity: Methods

Pore Volume	Grain Volume	Bulk Volume
❑ Boyle's Law	❑ Boyle's Law	❑ Caliper
❑ Saturation	❑ Archimedes	❑ Archimedes
❑ Summation-Of-Fluids	❑ BV-GV	❑ Mercury Displacement
❑ BV-GV	❑ Other	❑ GV+PV
❑ Other		❑ Other

Conditions/Fluids

Confining Stress (Magnitude and Type)_____ ❑ psi ❑ kPa

Gas _____ Liquid_____

Pressure _____

Permeability:	Methods	Conditions
	❑ Steady State	Fluid Type _____
	❑ Unsteady State	Confining Stress _____ ❑ psi ❑ kPa
	❑ Probe	Sleeve Durometer _____
	❑ Empirical	Pore Pressure _____Units _____
	❑ Not Measured	Klinkenberg: ❑ Measured ❑ Empirical ❑ No Correction
	❑ Others	Inertial Factor: ❑ Measured ❑ Empirical ❑ No Correction
Saturation:	Methods	Conditions
	❑ Distillation Extraction (DS)	Temperature _____ ❑ °F ❑ °C
	❑ High Temperature Retort	Fluids _____
	❑ Others	Water Density_____ ❑ g/cm³ ❑ kg/m³
		Oil Density _____ ❑ g/cm³ ❑ kg/m³
	Correction for Salt:	❑ Corrected for Water Volume
		❑ Corrected for Oil Weight ❑ Not Corrected

Quality Assurance: (Page number in report where the information is provided) _____

_____

Comments/Remarks: _____

Data Anomalies: _____

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block:		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

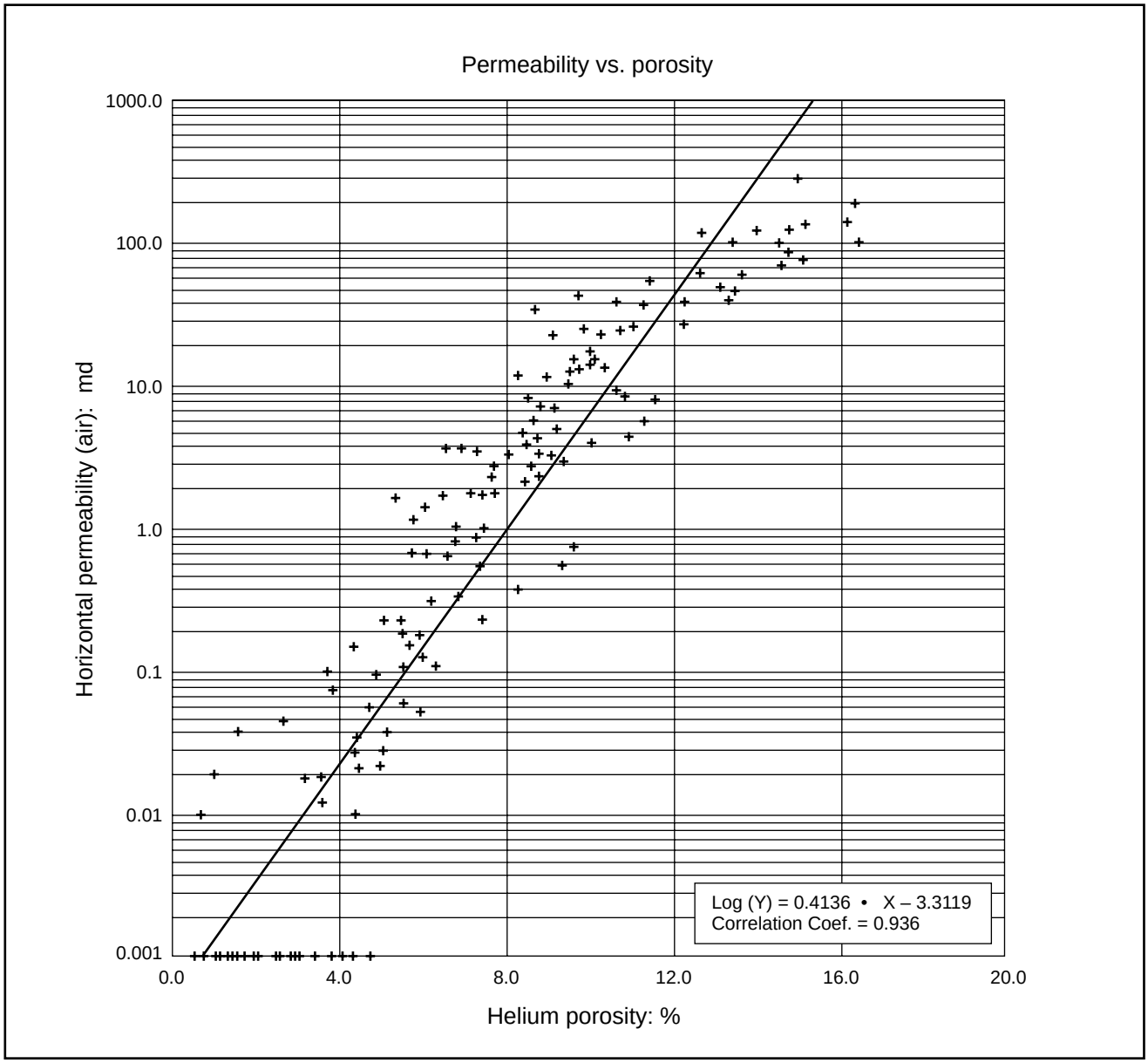


Figure 8-1—Permeability vs. Porosity

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block:		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

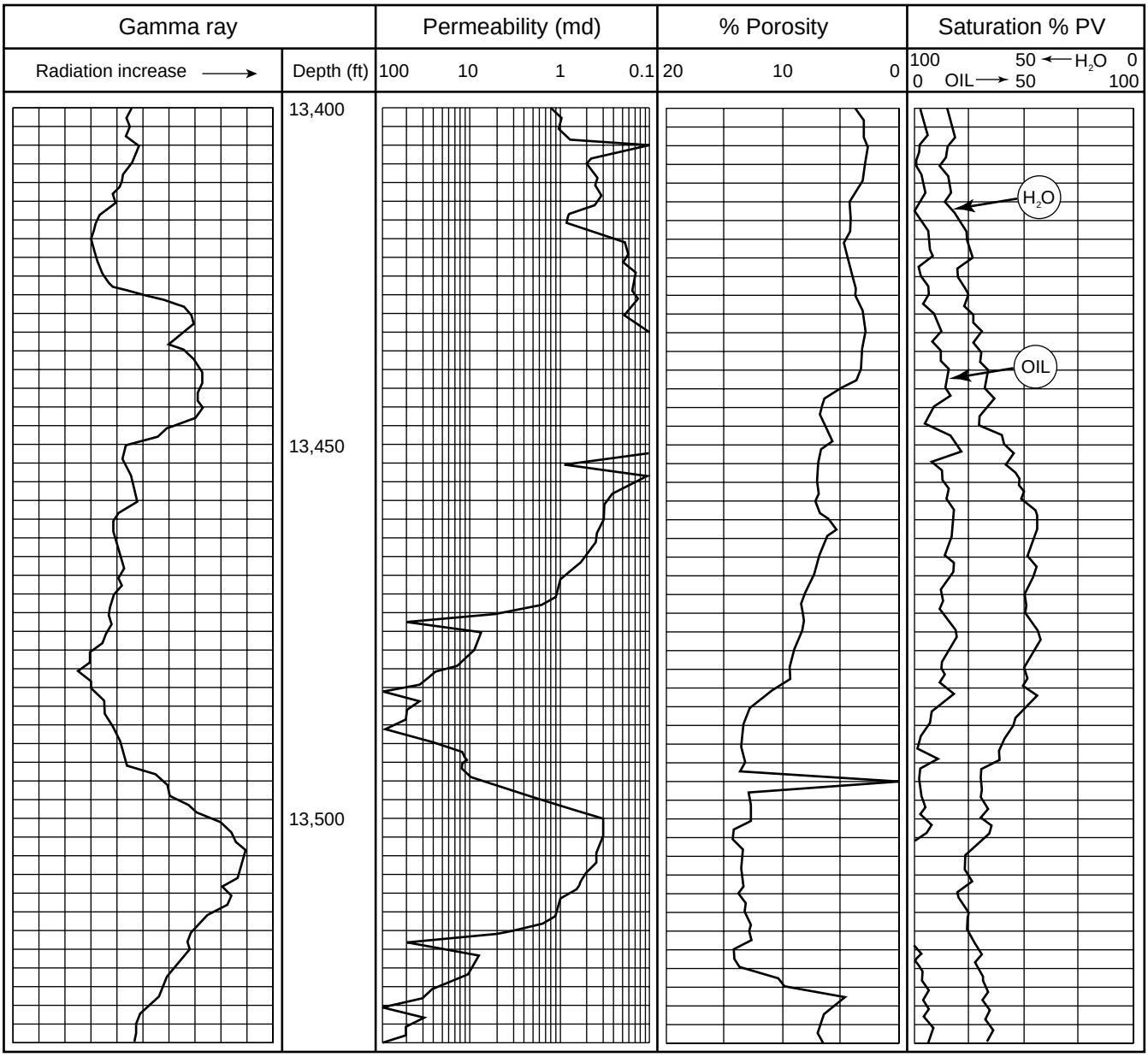


Figure 8-2—Core Data Profile

storage media (i.e., floppy disk, magnetic tape, optical disk, etc.). The physical format is the way in which stored data is located and organized on a physical medium. The specific way in which the logical and physical formats are bound depends on the medium and access mechanism and therefore are beyond the scope of this standard.

8.4.1 Digital Format

The DCIS format consists of the following elements:

a. Logical Record—A group of 8-bit bytes or data characters.
b. Logical File—A group of related Logical Records.

At the byte level, DCIS data are an ordered stream of 8-bit bytes, in which byte k precedes byte k+1. Data encryption is not allowed. No high-order bytes are allowed; all data should be represented in printable ASCII characters (ASCII 32 through ASCII 126). The only exception is ASCII 13 (carriage return) and ASCII 10 (line feed) at the end of each logical record. A combination carriage return and line feed signifies the end of the Logical Record. All records start at the byte location immediately following the carriage return-line feed combination of the previous record.

The layout of the logical file should be as follows (see example in Figure 8-3).

8.4.1.1 Logical File Header

This segment provides general information about the rest of the Logical File. Each record in this segment, as in other segments, is terminated by a carriage return, followed by a line feed character. Starting at logical location of byte 1 at record number 1, the following information should be present:

a. Record 1—Record Count: A positive integer, represented as ASCII characters, right justified, with no embedded commas, and with preceding blanks, if needed, to define the number of logical records including the record count in this logical file. This number includes records in the Logical File Header.
b. Record 2—Maximum Record Length: A non-negative integer represented as 3 ASCII characters, right justified, and with preceding blanks, if needed. A value of 0 indicates that the maximum length of a record is unknown. A positive value indicates that no record in the Logical File will exceed Maximum Record Length in size. A positive value need not correspond to the actual maximum record length; it needs only be an upper bound.

8.4.1.2 Logical File Body Header

The digital presentation of Basic Core Analysis reports should closely follow the format and layout of the hard copies. A maximum of 15 records is allowed in this section. Each record in this section is represented as ASCII characters, left justified, with trailing blanks (designated by the ASCII space character) allowed. The only exception to this is the first record in this segment, the "Number of Records In Logical Body Header," which is represented by a two character, right justified, positive integer. The Logical File Body Header should consist of the following, as a minimum, in this order:

a. Number Of Records In Logical File Body Header (this is included in the count).
b. Company.
c. Well.
d. Field.
e. Formation.
f. State.
g. County.
h. Core Type.
i. Mud Type.
j. Elevation (KB).
k. Date Of Report.
l. Analyzed by.

The first characters in each record in this segment must define the record (e.g. Company, Well, Field, etc.). A colon, :, is used as a separator between the definition and the value.

8.4.1.3 Logical File Body

This segment contains the main body of the core analyses data. One record is devoted to each sample reported in the hard copy report. This segment consists of:

a. Column Header—This is a segment giving the layout of the information to follow in the next segment. Information in this segment will provide the definitions for each column of data. A maximum of 25 records is allowed in this section. Each record in this section is represented by ASCII characters, left justified, with trailing blanks (designated by the ASCII space character) allowed. The only exception to this is the first record in this segment, the "Number Of Records In Logical File Body Column Header," which is represented by a two character, right justified, positive integer. It should be remembered that the count of records, identified as the "Number of Records in Logical File Body Header" found within the Logical File Body Header includes the line listing of the column header record count. This segment should contain, as a minimum, the following:

1. Number Of Records In Logical File Body Column Header (including this one in the count).
2. Sample Number.
3. Depth.
4. Sample type (plug, full diameter, etc.).

Additional records in this segment must specify the layout of additional data columns (e.g. permeability, oil saturation, etc.). Units of measurement must be included (%PV, gram, cm^3, etc.).

b. Body—This segment contains the main body of core analysis data, the layout of which is defined by the preceding segment. Various data columns for each sample analyzed must be separated by at least two ASCII space characters. The layout should be the same as that for the hardcopy of the report.

8.5 QUALITY ASSURANCE

Quality assurance/quality control (QA/QC) is an integral component of the core analysis process. This component is the foundation of all laboratory techniques and management. QA/QC impacts all technical decisions, training of personnel, selection of personnel and analytical equipment, use of instruments and equipment, and adherence to defined standards and quality control methodologies. In addition, operational procedures, i.e., regular procedural audits, documentation control, and quality control verifications are also impacted. QA/QC should be considered in the planning phase, before actually cutting the core. Quality assurance is the examination of the overall process and procedures used to obtain the data. Cost restraints and the purpose or use of the data may govern the testing methods and thoroughness used in obtaining data. How well the data represent the in situ formation may be quite uncertain and its assessment should be left to the evaluating engineer and/or the end user.

Quality control is the process of evaluating a particular testing method or instrument. It is a procedure which results in definable error limits as determined from the current study, or in a statement of precision which the laboratory has established based on comparable measurements of standard samples.

Quality control data should be supplied with every direct or indirect measurement along with the measurement procedures to provide the beginning of quality assurance. Quality control is the responsibility of the laboratory; and quality assurance is the responsibility of both the laboratory and the end user.

Proprietary procedures need not be divulged in detail in the report.

A high standard of QA/QC depends upon commitment by analysts, supervisor, laboratory director, and QA manager to quality production in strict accordance to established protocols. The International Organization for Standardization and National Measurement Accreditation Service are two organizations that are dedicated to setting quality assurance standards.

29	Record Count							Logical File Header
225	Maximum Record Length							
16	No. of Records in Logical File Body Header							
Company:	Gusher Oil Company							
Well:	Gusher #1							
API #:	3507123474							
Section:	18							
Township:	T-42-S							
Range:	R-18-E							
Field:	Gusher							Logical File Body Header
Formation:	Huge							
State:	California							
County:	Los Angeles							
Core Type:	Conventional With plastic Liner							
Mud Type:	Water base							
Elevation (KB):	20 ft. (6.1 meters)							
Date of Report:	June 9, 1992							
Analyzed by:	M. M. H.							
9	No. of Records in Logical File Body Column Header							
Sample Number								
Depth (ft.)								
Sample type								
Permeability (md)								Logical File Column Header
Porosity (% BV)								
Oil Saturation (% PV)								
Water Saturation (% PV)								
Lithology								
1	1234	“Whole Core”,	12.3	35.5	30.0	42.0	SS: 1t brn, slt-med gr, p cmt	Body
2	1235	or “Plug”, or	10.3	30.2	29.0	50.8	SS: 1t brn, slt-med gr, p cmt	
3	1236	“Sidewall, etc.	0.3	8.5	21.3	62.3	SS: 1t brn, slt-med gr, p cmt	

Figure 8-3—Example of DCIS Logical File

8.6 QA/QC PROTOCOL

8.6.1 Step 1

a. Ensure that the Standard Operating Procedure (SOP) is compatible with sample type and supervisor's instructions.

b. Assume custody of all or portions of the samples.

c. Set up instrument/apparatus consistent with specified SOP.

d. Perform analysis (including calibration) according to specified SOP.

e. Derive data from analyses and ensure that data is within control limits.

f. Maintain all results, instrument control, calibration checks, and documentation in accordance with SOP documentation requirements.

g. Maintain all QA/QC data control charts.

8.6.2 Step 2

a. Review procedural requirement for sample type and ensure that correct SOP was followed.

b. Review all data generated and check calculations.

c. Review QA/QC criteria and ascertain that the data are within limits.

d. Evaluate results for reanalysis if data are not within QA/QC limits and note in report when acceptance limits have been exceeded.

e. Prepare a final report and submit it to the Laboratory Director.

8.6.3 Step 3

a. Review final report data and compare each new set of data with previously completed data for consistency.

b. Verify that the data are within acceptable QA/QC criteria limits.

c. Order reanalysis of sample when necessary and if possible.

d. Review final report for analytical quality, custodial documentation, and completeness. Confirm that all laboratory procedures and documentation satisfy in-house and client requirements.

8.6.4 Step 4

a. Perform completeness audits of randomly and non-randomly selected final reports. This audit includes document verification, data reduction, and report narrative evaluations.

b. Review final reports for analytical and documentary completeness, client or contractual requirements, and timeliness.

c. Report to senior management.

d. Perform biannual system audits with blind check samples and periodic performance evaluation studies.

Tabulated data in written reports could follow the format presented in Tables 8-3 through 8-8. Standard abbreviations for lithologic descriptions could follow the nomenclature presented in Tables 8-9 and 8-10. Table 8-11 presents some of the SI units, conversion factors, and equations that are often used by core analysts. Table 8-12 presents common nomenclature.

Table 8-3—Basic Core Plug Analysis Report*

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Interval represented, ft. or m	Gas Permeability[b], md k_h $k_{h\infty}$[c] k_v $k_{v\infty}$[c]	Pore Volume[d] cm^3	Porosity[b] % BV	Grain Density g/cm^3	Saturation, % PV Oil Water Gas	Lithology[e]
_____								
_____								
_____								

*This form can also be used for reporting core analysis of rotary sidewall plugs.
[a]The indicated depth is driller's depth to tenth of the foot or hundredth of meter (0.1 ft. or 0.01 m).
[b]The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.
[c]Whether the correction for gas slippage or inertia is made (extrapolation or correlation) and the type of gas used for flow should be specified.
[d]Optional, intended for data QA/QC.
[e]See the standard abbreviation table.

Table 8-4—Basic Full Diameter Core Analysis Report

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Gas Permeability[b], md k_{max}[c] k_{90}[c] k_v[c]	Pore Volume[d] cm^3	Porosity[b] % BV	Grain Density g/cm^3	Saturation, % PV Oil Water Gas	Lithology[e]
_____							
_____							
_____							

[a]In full diameter core analysis, the depth associated with the actual top and bottom of the sample should be specified.
The indicated depth should be drillers depth to tenth of the foot or hundredth of meter (0.1 ft. or 0.01 m).
[b]The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.
[c]Whether the correction for gas slippage or inertia is made and the type of gas used for flow should be specified (See Table 8-2).
k_{max} is not the maximum permeability but the greater of two measured horizontal permeability values, whereas k_{90} is the horizontal value 90° to k_{max}.
[d]Optional, intended for data QA/QC.
[e]See the standard abbreviation table.

Table 8-5—Basic Sidewall Core Analysis Report

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Recovered length in. or cm	Depth[a] ft. or m	Permeability, md (empirical)	Porosity % BV	Pore Volume[b] cm^3	Saturation, % PV (retort) Oil Water	Saturation[c] % PV Oil Gas	Probable[d] Production	Critical Water[e] Saturation, % PV (empirical)	Gas[f] Detector Units	Lithology[g]
_____										
_____										
_____										

[a]The indicated depth is the wireline depth to tenth of the foot or hundredth of meter (0.1 ft. or 0.01 m).
[b]Optional, intended for QA/QC.
[c]Optional.
[d]Optional, expected fluid to be produced.
[e]Optional, estimated reservoir water saturation above which one should expect water production (empirical value obtained from correlation).
[f]Presence of hydrocarbon gas measured by conductivity of hot wire element inserted into each sidewall jar or suitable container used to transport each core.
[g]See the standard abbreviation table.

Table 8-6—Basic Oil-Wet Sponge Core Analysis Report

Client Company:		Date:
API Number:		Other Information: Oil density used in calculations, _____ ❑g/cm³ ❑kg/m³
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Permeability[b], md k_{max}[c] k_{90}[c] k_v[c]	Pore Volume[b] cm³	Porosity % BV	Grain Density g/cm³	Sponge Oil Saturation %PV[e]	Core Oil Saturation %PV	Total Oil Saturation %PV (Core + Sponge)	Water Saturation %PV	Lithology[f]
_____										
_____										
_____										

[a]In full diameter core analysis, the depth associated with the actual top and bottom of the sample should be specified. The indicated depth should be drillers depth to tenth of a foot or hundredth of a meter (0.1 ft. or 0.01 m).
[b]The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.
[c]Whether the correction for gas slippage or inertia is made and the type of gas used for flow should be specified (See Table 8-2). k_{max} is not the maximum horizontal permeability value, whereas k_{90} is the horizontal value 90° to k_{max}.
[d]Optional, intended for data QA/QC.
[e]Standard condition should be specified.
[f]See the standard abbreviation table.

Table 8-7—Basic Full Diameter Pressure-Retained Core Analysis Report

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Gas Permeability[b], md k_{max}[c] k_{90}[c] k_v[c]	Pore Volume[d] cm³	Porosity[b] % PV	Grain Density g/cm³	Saturation, % PV Pressure Depletion Oil Water	Saturation, % PV Total @ Stand. Cond.[e] Oil Water	Lithology[f]
_____								
_____								
_____								

[a]In full diameter core analysis, the depth associated with the actual top and bottom of the sample should be specified. The indicated depth should be drillers depth to tenth of a foot or hundredth of a meter (0.1 ft. or 0.01 m).
[b]The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.
[c]Whether the correction for gas slippage or inertia is made and the type of gas used for flow should be specified (See Table 8-2). k_{max} is not the maximum permeability but the greater of two measured horizontal permeability values, whereas k_{90} is the horizontal value 90° to k_{max}.
[d]Optional, intended for data QA/QC.
[e]Standard condition should be specified.
[f]See the standard abbreviation table.

Table 8-8—Basic Core Fracture Analysis Report

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Gas Permeability[b], md k_h, N-S[c] k_h, E-W[c] k_h, NW-SE[c] k_h, NE-SW[c]	Gas Permeability[b], md k_v[c]	Pore Volume[d] cm^3	Porosity[b] % BV	Grain Density g/cm^3	Saturation, % PV Oil Water	Lithology[e]
_____								
_____								
_____								

[a]In full diameter core analysis, the depth associated with the actual top and bottom of the sample should be specified. The indicated depth should be drillers depth to tenth of a foot or hundredth of meter (0.1 ft. or 0.01 m).
[b]The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.
[c]Whether the correction for gas slippage or inertia is made and the type of gas used for flow should be specified.
[d]Optional, intended for QA/QC.
[e]See the standard abbreviation table.

Table 8-9—Standard Abbreviations for Lithologic Descriptions (Sorted alphabetically by term.)

about	abt
above	abv
absent	abs
abundant	abd
acicular	acic
after	aft
agglomerate	aglm
aggregate	agg
algae algal	alg
allochem	allo
alter (ed ing)	alt
altered altering	alt
amber	amb
ammonite	amm
amount	amt
and	&
angular	ang
anhedral	ahd
anhydrite (ic)	anhy
aphanitic	aph
apparent	apr
appears	aprs
approximate (ly)	aprox
aragonite	arag
arenaceous	aren
argillaceous	arg
argillite	argl
arkose (ic)	ark
as above	a.a
asphalt (ic)	asph
assemblage	assem
associated	assoc
at	@
authigenic	authg
average	av
band (ed)	bnd
Barite (ic)	bar
basalt (ic)	bas
basement	bm
become (ing)	bcm
bed (ded ing)	bdg
bentonite (ic)	bent
bioclastic	biocl
bioherm (al)	bioh
biomicrite	biomi
biosparite	biosp
biostrom (al)	biost
biotite	biot
bioturbated	biotb
birdseye	bdeye
birefringence	bifg
bitumen (inous)	bit
black (ish)	blk blksh
blade (ed)	bld
block (y)	blky
blue (ish)	bl blsh
bored (ing)	bor
botryoid (al)	btry
bottom	btm
boudinage	boudg
boulder	bldr
boundstone	bdst
brachiopod	brac
brackish	brak
branching	brhg
break	brk
breccia (ted)	brec
bright	brt
brittle	brit
brown	brn
bryozoa	Bry
bubble	bubl
buff	bu
Bulbous	bulb
burrow (ed)	bur
calcarenite	clcar
calcareous	calc
calcilutite	clclt
calcirudite	clcrd
calcisiltite	clslt
calcisphere	clcsp
calcite filled fractures	ccf
calcite (ic)	CaCO3
caliche	cche
carbonate	CO3
carbonized	cb
cavern (ous)	cav
caving	cvg
cement (ed ing)	cmt
center centered	cntr
cephalopod	ceph
chalcedony (ic)	chal
chalk (y)	chk chky
charophyte	char
chert (y)	cht chty
chitin (ous)	chit
chitinozoa	chtz
chlorite (ic)	chlor
chocolate	choc
circulate (ion)	circ
clastic	clas
clay (ey)	cly
claystone	clyst
clean	cln
clear	clr
cleavage	clvg
cluster	clus
coal	coal

(continues)

Table 8-9—(Continued)

coarse (ly ness) ..... crs
coated grains ..... ctd gn
coated (ing) ..... ctd
cobble ..... cbl
color (ed) ..... col
common ..... com
compact ..... cpct
compare ..... cf
concentric ..... cncn
conchoidal ..... conch
concretion (ary) ..... conc
conglomerate (ic) ..... cgl
conodont ..... cono
considerable ..... cons
consolidated ..... consol
conspicuous ..... conspic
contact ..... ctc
contamination (ed) ..... contm
content ..... cont
contorted ..... cntrt
coquina (oid) ..... coq
coral coralline ..... cor corin
core c
covered ..... cov
cream ..... crm
crenulated ..... cren
crevice ..... crev
crinkled ..... crnk
crinoid (al) ..... crin crinal
cross x
crossbedded (ing) ..... xbd xbdg
crosslaminated ..... xlam
crossstratified ..... xstrat
crumpled ..... crpld
cryptocrystalline ..... crpxl
cryptograined ..... crpgr
crystal (line) ..... xl xln
cube (ic) ..... cub
cuttings ..... ctgs
dark (er) ..... dk dkr
dead ..... dd
debris ..... deb
decrease (ing) ..... decr
dendritic ..... dend
dense (er) ..... dns
depauperate ..... depau
description ..... descr
desiccation ..... dess
determine ..... dtrm
detrital (us) ..... dtrl
devitrified ..... devit
diabase ..... db
diagenesis (etic) ..... diagn
diameter ..... dia
difference ..... dif
disseminated ..... dism
distillate ..... dist
ditto ..... "
dolocast (tic) ..... dolc
dolomite (ic) ..... dol
dolomold (ic) ..... dolmd
dolostone ..... dolst
dominant (ly) ..... dom
drill stem test ..... DST
drilling ..... drlg
druse ..... dru
drusy ..... drsy
earthy ..... ea
east ..... E
echinoid ..... ech
elevation ..... elev
elliptical ..... elip
elongate ..... elg
embedded ..... embd
enlarged ..... enl
equant ..... eqnt
equivalent ..... equiv
euhedral ..... euhd
euxinic ..... eux
evaporite (itic) ..... evap
excellent ..... ex
expose (ed ure) ..... exp
extraclast (ic) ..... exclas
extremely ..... extr
extrusive rock, extrusive (ion) ..... extr
facet (ed) ..... fac
faint (ly) ..... fnt
fair ..... fr
fault (ed) ..... flt
fauna ..... fau
feet ..... ft
feldspar (athic) ..... fspr
fenestra (al) ..... fen
Ferromagnesian ..... Femag
ferruginous ..... Fe
fibrous ..... fibr
figured ..... fig
fine (ly) ..... f fnly
fissile ..... fis
fissures ..... fiss
flaggy ..... flg
flake (s y) ..... flk flks flky
flat ..... fl
flesh ..... fls
floating ..... fltg
flora ..... flo
fluorescence (ent) ..... flor
foliated ..... fol
foot ..... ft
foraminifera (al) ..... foram

(continues)

Table 8-9—(Continued)

formation....fm
fossil (iferous)....fos
fracture (d)....frac
fragment (al)....frag
framework....frmwk
frequent....freq
fresh....frs
friable....fri
fringe (ing)....frg
frosted quartz grains....F.Q.G.
frosted....fros
fucoid (al)....fuc
fusulinid....fus
gabbro....gab
gas....G
gastropod....gast
generally....gen
geopetal....gept
gilsonite....gil
glass (y)....glas
glauconite (itic)....glau
Globigerina (inal)....glob
gloss (y)....glos
gneiss (ic)....gns
good....gd
grade (s ing d)....grd
grain (s, ed)....gr
grainstone....grst
granite wash....grnt.w
granite (ic)....grnt
granule (ar)....grnl
grapestone....grapst
graptolite....grap
gravel....grv
gray, grey (ish)....gry
grysh, graywacke....gwke
greasy....gsy
green (ish)....gn gnsh
grit (ty)....gt
gypsum (iferous)....gyp
hackly....hky
halite (iferous)....hal
hard....hd
heavy....hvy
hematite (ic)....hem
heterogeneous....hetr
heterostegina....het
hexagonal....hex
high (ly)....hi
homogeneous....hom
horizontal....hztl
hornblend....hornbl
hydrocarbon....hydc
igneous rock, igneous....ig
impression....imp
in part....I.P.
inch....in
inclusion (ded)....incl
increasing....incr
indistinct....indst
indurated....ind
inoceramus....inoc
insoluble....insl
interbedded....intbd
intercalated....intercal
intercrystalline....intxl
interfingered....intfr
interfragmental....intfrag
intergranular....intgran
intergrown....intgn
interlaminated....intlam
interparticle....intpar
interpretation....intpt
intersticies (iitial)....intst
interval....intvl
intraclast (ic)....intclas
intraformational....intfm
intraparticle....intrapar
intrusive rock, intrusive....intr
invertebrate....invtb
iridescent....irid
iron....Fe
ironstone....Fest
irregular (ly)....irr
irridescent....irid
isopachous....iso
jasper (oid)....jasp
joint (s, ed, ing)....jt jts jtd
kaolin (itic)....kao
lacustrine....lac
lamina (tions, ated)....lam
large larger....lge
laterite (itic)....lat
lavender....lav
layer....lyr
leached....lchd
ledge....ldg
lens, lenticular....len lent
light (er)....lt
lignite (itic)....lig
limestone....ls
limonite (itic)....lim
limy....lmy
lithic....lit
lithographic....lithgr
lithology (ic)....lith
little....ltl
littoral....litt
local....loc
long....lg

(continues)

Table 8-9—(Continued)

loose ... lse
lower ... low
lumpy ... lmpy
lustre ... lustr
lutite ... lut
macrofossil ... macrofos
magnetite, magnetic ... mag
manganese, manganiferous ... mn
marble ... mbl
marine ... marn
marl (y) ... mrl mrly
marlstone ... mrlst
maroon ... mar
massive ... mass
material matter ... mat
matrix ... mtrx
maximum ... max
median ... mdn
medium ... med
member ... mbr
meniscus ... men
metamorphic (osed) ... meta metaph
metasomatic ... msm
mica (ceous) ... mic
micrite (ic) ... micr
microcrystalline ... micxl
microfossil (iferous) ... microfos
micrograined ... micgr
micromicaceous ... micmica
microoolite ... microol
micropore (osity) ... micropor
microspar ... microspr
microstylolite ... microstyl
middle ... mid
miliolid ... milid
milky ... mky
mineral (ized) ... mnrl
minimum ... min
minor ... mnr
minute ... mnut
moderate ... mod
mold (ic) ... mol
mollusc (a) ... moll
mosaic ... mos
mottled (ing) ... mot
mud (dy) ... md mdy
mudstone ... mdst
muscovite ... musc
nacreous ... nac
no show ... n/s
no ... n
no sample ... n.s.
no visible porosity ... n.v.p.
nodule (s, ar) ... nod
north ... N
novaculite ... novac
numerous ... num
object ... obj
occasional ... occ
ochre ... och
odor ... od
oil source rock ... OSR
oil ... O
olive ... olv
Olivine ... olvn
oncolite (oidal) ... onc
ooid (al) ... oo
oolicast (ic) ... ooc
oolite (itic) ... ool
oomold (ic) ... oomol
opaque ... op
orange (ish) ... orng
orbitolina ... orbit
organic ... org
orthoclase ... orth
orthoquartzite ... otz
ostracod ... ostr
overgrowth ... ovgth
oxidized ... ox
oyster ... oyst
packstone ... pkst
paper (y) ... pap
part (ly) ... pt
particle ... par
parting ... ptg
parts per million ... ppm
patch (y, es) ... ptch
pearl (y) ... prly
pebble (ly, s) ... pbl
pelecypod ... plcy
pellet (al, oids) ... pel
pelletoid (al) ... peld
pendular (ous) ... pend
permeability (able) ... perm K k
petroleum petroliferous ... pet
Phenocrysts ... phen
phlogopite ... phlog
phosphate (atic) ... phos
phreatic ... phr
phyllite, phyllitic ... phly
pink ... pk
pinkish ... pkish
pinpoint porosity ... p.p.
pisoid (al) ... piso
pisolite pisolitic ... pisol
pitted ... pit
plagioclase ... plag
plant ... plt
plastic ... plas
platy ... plty

(continues)

Table 8-9—(Continued)

polish polished ..... pol
pollen ..... poln
polygonal ..... poly
poor (ly) ..... p
porahyry ..... prphy
porcelaneous ..... porcel
porosity porous ..... por ϕ
possible (ly) ..... pos
predominant (ly) ..... pred
preserved (ation) ..... pres
pressure deformation ..... p d
primary ..... prim
prism (atic) ..... pris
probable (ly) ..... prob
production ..... prod
prominent (ly) ..... prom
pseudo ..... psdo
pseudo oolite (ic) ..... psool
pumicestone ..... pst
purple ..... purp
pyrite (itized itic) ..... pyr
pyrobitumen ..... pybit
pyroclastic ..... pyrcl
pyroxene ..... pyrxn
quartz (ose, ic) ..... qtz qtzs qtzt
radial (ate, ating) ..... rad
radiaxial ..... radax
range (ing) ..... rng
rare ..... rr
recemented ..... recem
recovery (ered) ..... rec
recrystal (lize, ed, ation) ..... rexl rexlzd rexlt
red (ish) ..... rd rdsh
reef (oid) ..... rf
regular ..... reg
remains, remnant ..... rmn
replaced (ment, ing) ..... rep
residue (ual) ..... res
resinous ..... rsns
rhomb (ic) ..... rhb
ripple ..... rpl
rock ..... rk
round (ed) ..... rnd
rounded frosted pitted ..... r.f.p.
rubble (bly) ..... rbl rbly
rudist ..... rud
rugose, ruga ..... rug
rugose coral rugosa ..... rugc
saccharoidal ..... sacc
salt and pepper ..... s & p
salt water ..... s.w.
salt (y) ..... sa
saltcast (ic) ..... sac
same as above ..... a.a.
sample ..... spl
sand (y) ..... sd sdy
sandstone ..... ss
saturation (ated) ..... sat
scales ..... sc
scaphopod ..... scaph
scarce ..... scs
scatter (ed) ..... scat
schist (ose) ..... sch
scolecodont ..... scol
secondary ..... sec
sediment (ary) ..... sed
selenite ..... sel
septate ..... sept
shadow ..... shad
shale (ly) ..... sh shy
shell ..... shl
shelter porosity ..... shlt por
show ..... shw
siderite (itic) ..... sid
sidewall core ..... S.W.C.
silica (iceous) ..... sil
silky ..... slky
silt (y) ..... slt slty
siltstone ..... sltst
similar ..... sim
size ..... sz
skeletal ..... skel
slabby ..... slb
slate (y) ..... sl
slickenside (d) ..... sks
slight (ly) ..... sli
small ..... sml
smooth ..... sm
soft ..... sft
solitary ..... sol
solution soluble ..... sln
somewhat ..... smwt
sort (ing, ed) ..... srt srtg srtd
south ..... S
spar (ry) ..... spr
sparry calcite ..... spc
sparse (ly) ..... sps spsly
speck (led) ..... spk spkld
sphalerite ..... sphal
spherule (itic, s) ..... sph
spicule (ar) ..... spic
splintery ..... splty
sponge ..... spg
spore ..... spo
spot (ted, y) ..... sp sptd spty
stain (ed, ing) ..... stn
stalactitic ..... stal
stippled ..... stip
strata (ified, tion) ..... strat
streak (ed) ..... strk

(continues)

Table 8-9—(Continued)

streaming	stmg
striae (ted)	stri
stringer	strg
stromatolite (itic)	stromlt
stromatoporoid	strom
structure	struc
stylolite (itic)	styl
sub	sb
subangular	sbang
subhedral	sbhed
sublithic	sblit
subrounded	sbrd
sucrosic	suc
sugary	sug
sulphur (ous)	S su
superficial oolite (ic)	spfool
surface	surf
syntaxial	syn
tabular (ate)	tab
tan	tn
tension	tns
terriginous	ter
texture (d)	tex
thick	thk
thin section	T.S.
thin	thn
thinbedded	t.b.
throughout	thru
tight (ly)	ti
top	tp
tough	tgh
trace	tr
translucent	trnsl
transparent	trnsp
trilobite	tril
tripoli (itic)	trip
tube (ular)	tub
tuff (aceous)	tuf
type (ical)	typ
unconformity	unconf
unconsolidated	uncons
underclay	uc
underlying	undly
unidentifiable	unident
uniform	uni
upper	u
vadose	vad
variation (able ed)	var
varicolored	vcol
variegated	vgt
varved	vrvd
vein (ing ed)	vn
veinlet	vnlet
vermillion	verm
vertebrate	vrtb
vertical	vert
very poor sample	vps
very	v
vesicular	ves
violet	vi
visible	vis
vitreous (ified)	vit
volatile	volat
volcanic rock volcanic	volc
vug (gy)	vug
wackestone	wkst
washed residue	W.R.
water	wtr
wavy	wvy
waxy	wxy
weak	wk
weather (ed)	wthr
wthrd well	wl
west	W
white	wh
with	w/
without	w/o
wood	wd
yellow (ish)	yel yelsh
zeolite	zeo
zircon	Zr
zone	z

Note: Most of the terms and abbreviations are extracted from *Sample Examination Manual*, R. G. Swanson, AAPG, 1981.

Table 8-10—Standard Abbreviations for Lithologic Descriptions
(Sorted alphabetically by abbreviations.)

ditto	"
and	&
at	@
same as above	a.a.
as above	a.a.
abundant	abd
absent	abs
about	abt
above	abv
acicular	acic
after	aft
aggregate	agg
agglomerate	aglm
anhedral	ahd
algae, algal	alg
allochem	allo
alter (ed ing)	alt
altered, altering	alt
amber	amb
ammonite	amm
amount	amt
angular	ang
anhydrite (ic)	anhy
aphanitic	aph
apparent	apr
approximate (ly)	aprox
appears	aprs
aragonite	arag
arenaceous	aren
argillaceous	arg
argillite	argl
arkose (ic)	ark
asphalt (ic)	asph
assemblage	assem
associated	assoc
authigenic	authg
average	av
Barite (ic)	bar
basalt (ic)	bas
become (ing)	bcm
birdseye	bdeye
bed (ded ing)	bdg
boundstone	bdst
bentonite (ic)	bent
birefringence	bifg
bioclastic	biocl
bioherm (al)	bioh
biomicrite	biomi
biosparite	biosp
biostrom (al)	biost
biotite	biot
bioturbated	biotb
bitumen (inous)	bit
blue (ish)	bl blsh
blade (ed)	bld
boulder	bldr
black (ish)	blk blksh
block (y)	blky
basement	bm
band (ed)	bnd
bored (ing)	bor
boudinage	boudg
brachiopod	brac
brackish	brak
breccia (ted)	brec
branching	brhg
brittle	brit
break	brk
brown	brn
bright	brt
bryozoa	Bry
bottom	btm
botryoid (al)	btry
buff	bu
bubble	bubl
Bulbous	bulb
burrow (ed)	bur
core	c
calcite (ic)	CaCO3
calcareous	calc
cavern (ous)	cav
carbonized	cb
cobble	cbl
calcite filled fractures	ccf
caliche	cche
cephalopod	ceph
compare	cf
conglomerate (ic)	cgl
chalcedony (ic)	chal
charophyte	char
chitin (ous)	chit
chalk (y)	chk chky
chlorite (ic)	chlor
chocolate	choc
chert (y)	cht chty
chitinozoa	chtz
circulate (ion)	circ
clastic	clas
calcarenite	clcar
calcilutite	clclt
calcirudite	clcrd
calcisphere	clcsp
clean	cln
clear	clr
calcisiltite	clslt
cluster	clus
cleavage	clvg
clay (ey)	cly

(continues)

Table 8-10—(Continued)

claystone ..... clyst
cement (ed ing) ..... cmt
concentric ..... cncn
center centered ..... cntr
contorted ..... cntrt
carbonate CO3
coal ..... coal
color (ed) ..... col
common ..... com
concretion (ary) ..... conc
conchoidal ..... conch
conodont ..... cono
considerable ..... cons
consolidated ..... consol
conspicuous ..... conspic
content ..... cont
contamination (ed) ..... contm
coquina (oid) ..... coq
coral coralline ..... cor corin
covered ..... cov
compact ..... cpct
crenulated ..... cren
crevice ..... crev
crinoid (al) ..... crin
crinal cream ..... crm
crinkled ..... crnk
cryptograined ..... crpgr
crumpled ..... crpld
cryptocrystalline ..... crpxl
coarse (ly ness) ..... crs
contact ..... ctc
coated (ing) ..... ctd
coated grains ..... ctd gn
cuttings ..... ctgs
cube (ic) ..... cub
caving ..... cvg
diabase ..... db
dead ..... dd
debris ..... deb
decrease (ing) ..... decr
dendritic ..... dend
depauperate ..... depau
description ..... descr
desiccation ..... dess
devitrified ..... devit
diameter ..... dia
diagenesis (etic) ..... diagn
difference ..... dif
disseminated ..... dism
distillate ..... dist
dark (er) ..... dk dkr
dense (er) ..... dns
dolomite (ic) ..... dol
dolocast (tic) ..... dolc
dolomold (ic) ..... dolmd
dolostone ..... dolst
dominant (ly) ..... dom
drilling ..... drig
drusy ..... drsy
druse ..... dru
drill stem test ..... DST
detrital (us) ..... dtrl
determine ..... dtrm
east ..... E
earthy ..... ea
echinoid ..... ech
elevation ..... elev
elongate ..... elg
elliptical ..... elip
embedded ..... embd
enlarged ..... enl
equant ..... eqnt
equivalent ..... equiv
euhedral ..... euhd
euxinic ..... eux
evaporite (itic) ..... evap
excellent ..... ex
extraclast (ic) ..... exclas
expose (ed ure) ..... exp
extrusive rock extrusive (ion) ..... extr
extremely ..... extr
fine (ly) ..... f fnly
frosted quartz grains ..... F.Q.G.
facet (ed) ..... fac
fauna ..... fau
ferruginous ..... Fe
iron ..... Fe
Ferromagnesian ..... Femag
fenestra (al) ..... fen
ironstone ..... Fest
fibrous ..... fibr
figured ..... fig
fissile ..... fis
fissures ..... fiss
flat ..... fl
flaggy ..... flg
flake (s y) ..... flk flks flky
flora ..... flo
fluorescence (ent) ..... flor
flesh ..... fls
fault (ed) ..... flt
floating ..... fltg
formation ..... fm
faint (ly) ..... fnt
foliated ..... fol
foraminifera (al) ..... foram
fossil (iferous) ..... fos
fair ..... fr
fracture (d) ..... frac
fragment (al) ..... frag

(continues)

Table 8-10—(Continued)

frequent ..... freq
fringe (ing) ..... frg
friable ..... fri
framework ..... frmwk
frosted ..... fros
fresh ..... frs
feldspar (athic) ..... fspr
feet ..... ft
foot ..... ft
fucoid (al) ..... fuc
fusulinid ..... fus
gas ..... G
gabbro ..... gab
gastropod ..... gast
good ..... gd
generally ..... gen
geopetal ..... gept
gilsonite ..... gil
glass (y) ..... glas
glauconite (itic) ..... glau
Globigerina (inal) ..... glob
gloss (y) ..... glos
green (ish) ..... gn gnsh
gneiss (ic) ..... gns
grain (s, ed) ..... gr
graptolite ..... grap
grapestone ..... grapst
grade (s ing d) ..... grd
granule (ar) ..... grnl
granite (ic) ..... grnt
granite wash ..... grnt.w
grainstone ..... grst
gravel ..... grv
gray, grey (ish) ..... gry grysh
greasy ..... gsy
grit (ty) ..... gt
graywacke ..... gwke
gypsum (iferous) ..... gyp
halite (iferous) ..... hal
hard ..... hd
hematite (ic) ..... hem
heterostegina ..... het
heterogeneous ..... hetr
hexagonal ..... hex
high (ly) ..... hi
hackly ..... hky
homogeneous ..... hom
hornblend ..... hornbl
heavy ..... hvy
hydrocarbon ..... hydc
horizontal ..... hztl
in part ..... I.P.
igneous rock, igneous ..... ig
impression ..... imp
inch ..... in
inclusion (ded) ..... incl
increasing ..... incr
indurated ..... ind
indistinct ..... indst
inoceramus ..... inoc
insoluble ..... insl
interbedded ..... intbd
intraclast (ic) ..... intclas
intercalated ..... intercal
intraformational ..... intfm
interfingered ..... intfr
interfragmental ..... intfrag
intergrown ..... intgn
intergranular ..... intgran
interlaminated ..... intlam
interparticle ..... intpar
interpretation ..... intpt
intrusive rock intrusive ..... intr
intraparticle ..... intrapar
intersticies (iitial) ..... intst
interval ..... intvl
intercrystalline ..... intxl
invertebrate ..... invtb
iridescent ..... irid
irridescent ..... irid
irregular (ly) ..... irr
isopachous ..... iso
jasper (oid) ..... jasp
joint (s, ed, ing) ..... jt jts jtd
kaolin (itic) ..... kao
lacustrine ..... lac
lamina (tions, ated) ..... lam
laterite (itic) ..... lat
lavender ..... lav
leached ..... lchd
ledge ..... ldg
lens, lenticular ..... len lent
long ..... lg
large larger ..... lge
lignite (itic) ..... lig
limonite (itic) ..... lim
lithic ..... lit
lithology (ic) ..... lith
lithographic ..... lithgr
littoral ..... litt
lumpy ..... lmpy
limy ..... lmy
local ..... loc
lower ..... low
limestone ..... ls
loose ..... lse
light (er) ..... lt
little ..... ltl
lustre ..... lustr
lutite ..... lut

(continues)

Table 8-10—(Continued)

layer	lyr
macrofossil	macrofos
magnetite, magnetic	mag
maroon	mar
marine	marn
massive	mass
material matter	mat
maximum	max
marble	mbl
member	mbr
mud (dy)	md mdy
median	mdn
mudstone	mdst
medium	med
meniscus	men
metamorphic (osed)	meta metaph
mica (ceous)	mic
micrograined	micgr
micromicaceous	micmica
micrite (ic)	micr
microfossil (iferous)	microfos
microoolite	microol
micropore (osity)	micropor
microspar	microspr
microstylolite	microstyl
microcrystalline	micxl
middle	mid
miliolid	milid
minimum	min
milky	mky
manganese, manganiferous	mn
minor	mnr
mineral (ized)	mnrl
minute	mnut
moderate	mod
mold (ic)	mol
mollusc (a)	moll
mosaic	mos
mottled (ing)	mot
marl (y)	mrl mrly
marlstone	mrlst
metasomatic	msm
matrix	mtrx
muscovite	musc
no show	n/s
no	n
north	N
no sample	n.s.
no visible porosity	n.v.p.
nacreous	nac
nodule (s, ar)	nod
novaculite	novac
numerous	num
oil	O
object	obj
occasional	occ
ochre	och
odor	od
olive	olv
Olivine	olvn
oncolite (oidal)	onc
ooid (al)	oo
oolicast (ic)	ooc
oolite (itic)	ool
oomold (ic)	oomol
opaque	op
orbitolina	orbit
organic	org
orange (ish)	orng
orthoclase	orth
oil source rock	OSR
ostracod	ostr
orthoquartzite	otz
overgrowth	ovgth
oxidized	ox
oyster	oyst
poor (ly)	p
pressure deformation	p d
pinpoint porosity	p.p.
paper (y)	pap
particle	par
pebble (ly, s)	pbl
pellet (al, oids)	pel
pelletoid (al)	peld
pendular (ous)	pend
permeability (able)	perm K k
petroleum, petroliferous	pet
Phenocrysts	phen
phlogopite	phlog
phyllite, phyllitic	phly
phosphate (atic)	phos
phreatic	phr
pisoid (al)	piso
pisolite, pisolitic	pisol
pitted	pit
pink	pk
pinkish	pkish
packstone	pkst
plagioclase	plag
plastic	plas
pelecypod	plcy
plant	plt
platy	plty
polish, polished	pol
pollen	poln
polygonal	poly
porosity, porous	por ϕ
porcelaneous	porcel
possible (ly)	pos
parts per million	ppm

(continues)

Table 8-10—(Continued)

predominant (ly) ..... pred
preserved (ation) ..... pres
primary ..... prim
prism (atic) ..... pris
pearl (y) ..... prly
probable (ly) ..... prob
production ..... prod
prominent (ly) ..... prom
porahyry ..... prphy
pseudo ..... psdo
pseudo oolite (ic) ..... psool
pumicestone ..... pst
part (ly) ..... pt
patch (y, es) ..... ptch
parting ..... ptg
purple ..... purp
pyrobitumen ..... pybit
pyrite (itized itic) ..... pyr
pyroclastic ..... pyrcl
pyroxene ..... pyrxn
quartz (ose, ic) ..... qtz qtzs qtzt
rounded frosted pitted ..... r.f.p.
radial (ate, ating) ..... rad
radiaxial ..... radax
rubble (bly) ..... rbl rbly
red (ish) ..... rd rdsh
recovery (ered) ..... rec
recemented ..... recem
regular ..... reg
replaced (ment, ing) ..... rep
residue (ual) ..... res
recrystal (lize, ed, ation) ..... rexl rexlzd rexlt
reef (oid) ..... rf
rhomb (ic) ..... rhb
rock ..... rk
remains remnant ..... rmn
round (ed) ..... rnd
range (ing) ..... rng
ripple ..... rpl
rare ..... rr
resinous ..... rsns
rudist ..... rud
rugose, ruga ..... rug
rugose coral, rugosa ..... rugc
salt and pepper ..... s & p
south ..... S
sulphur (ous) ..... S su
salt water ..... s.w.
sidewall core ..... S.W.C.
salt (y) ..... sa
saltcast (ic) ..... sac
saccharoidal ..... sacc
saturation (ated) ..... sat
sub ..... sb
subangular ..... sbang
subhedral ..... sbhed
sublithic ..... sblit
subrounded ..... sbrd
scales ..... sc
scaphopod ..... scaph
scatter (ed) ..... scat
schist (ose) ..... sch
scolecodont ..... scol
scarce ..... scs
sand (y) ..... sd sdy
secondary ..... sec
sediment (ary) ..... sed
selenite ..... sel
septate ..... sept
soft ..... sft
shale (ly) ..... sh shy
shadow ..... shad
shell ..... shl
shelter porosity ..... shlt por
show ..... shw
siderite (itic) ..... sid
silica (iceous) ..... sil
similar ..... sim
skeletal ..... skel
slickenside (d) ..... sks
slate (y) ..... sl
slabby ..... slb
slight (ly) ..... sli
silky ..... slky
solution soluble ..... sln
silt (y) ..... slt slty
siltstone ..... sltst
smooth ..... sm
small ..... sml
somewhat ..... smwt
solitary ..... sol
spot (ted, y) ..... sp sptd spty
sparry calcite ..... spc
superficial oolite (ic) ..... spfool
sponge ..... spg
spherule (itic, s) ..... sph
sphalerite ..... sphal
spicule (ar) ..... spic
speck (led) ..... spk spkld
sample ..... spl
splintery ..... splty
spore ..... spo
spar (ry) ..... spr
sparse (ly) ..... sps spsly
sort (ing, ed) ..... srt srtg srtd
sandstone ..... ss
stalactitic ..... stal
stippled ..... stip
streaming ..... stmg
stain (ed, ing) ..... stn

(continues)

Table 8-10—(Continued)

strata (ified, tion)	strat
stringer	strg
striae (ted)	strl
streak (ed)	strk
stromatoporoid	strom
stromatolite (itic)	stromlt
structure	struc
stylolite (itic)	styl
sucrosic	suc
sugary	sug
surface	surf
syntaxial	syn
size	sz
thinbedded	t.b.
thin section	T.S.
tabular (ate)	tab
terriginous	ter
texture (d)	tex
tough	tgh
thick	thk
thin	thn
throughout	thru
tight (ly)	ti
tan	tn
tension	tns
top	tp
trace	tr
trilobite	tril
tripoli (itic)	trip
translucent	trnsl
transparent	trnsp
tube (ular)	tub
tuff (aceous)	tuf
type (ical)	typ
upper	u
underclay	uc
unconformity	unconf
unconsolidated	uncons
underlying	undly
uniform	uni
unidentifiable	unident
very	v
vadose	vad
variation (able ed)	var
varicolored	vcol
vermillion	verm
vertical	vert
vesicular	ves
variegated	vgt
violet	vi
visible	vis
vitreous (ified)	vit
vein (ing ed)	vn
veinlet	vnlet
volatile	volat
volcanic rock volcanic	volc
very poor sample	vps
vertebrate	vrtb
varved	vrvd
vug (gy)	vug
with	w/
without	w/o
west	W
washed residue	W.R.
wood	wd
white	wh
weak	wk
wackestone	wkst
well	wl
weather (ed)	wthr wthrd
water	wtr
wavy	wvy
waxy	wxy
cross	x
crossbedded (ing)	xbd xbdg
crystal (line)	xl xln
crosslaminated	xlam
crossstratified	xstrat
yellow (ish)	yel yelsh
zone	z
zeolite	zeo
zircon	Zr

Note: Most of the terms and abbreviations are extracted from *Sample Examination Manual*, R. G. Swanson, AAPG, 1981 Units and Conversions.

Table 8-11—Units and Conversions

Quantity	Customary unit	Metric unit SPE preferred	Conversion factor multiply customary unit by factor to get metric unit	
t = time	d	s	8.6400	E+04
	s	d	1.1574	E-05
L = length	ft	m	3.048	E-01
	in	mm	2.54	E+01
A = area	sq ft	m^2	9.290	E-02
	sq in	mm^2	6.4516	E=02
V = volume, capacity	liter	dm^3	1.0	
	ft^3	m^3	2.831685	E-02
m = mass	lbm	kg	4.535942	E-01
	kg	g	1.0	E+03
	g	kg	1.0	E-03
T = temperature	˚F	˚C	(˚F - 32)/1.8	
	˚C	˚C	1.0	
	˚R	K	5/9	
	K	K	1.0	
P = pressure	atm (760 mm Hg at 0˚C or 14.696 lbf/in^2)	MPa	1.01325	E-01
		kPa	1.01325	E+02
	bar	MPa	1.0	E-01
	bar	kPa	1.0	E+02
	lbf/in^2 (psi)	MPa	6.894757	E-03
	lbf/in^2 mm Hg (0˚C)	kPa	6.894757	
	torr	kPa	1.333224	E-01
	$dyne/cm^2$	Pa	1.0	E-01
q = flow rate	ft^3/D	m^3/d	2.831685	E-02
	U.S. gal/min (liquids)	dm^3/s	6.309020	E-02
	ft^3/D	cm^3/s	3.277413	E-01
u = volumetric velocity (flux, or superficial)	ft/D	m/d	3.048	E-01
	ft/D	cm/d	3.048	E+01
	ft/D	mm/d	3.48	E+02
	ft/s	m/s	3.048	E-01
ρgas = Density (gases)	lbm/ft^3	kg/m^3	1.601846	E+01
	lbm/ft^3	g/m^3	1.601846	E+04
ρw, ρo = Density	lbm/U.S. gal (liquids)	kg/m^3	1.198264	E+02
	lbm/U.S. gal (liquids)	g/cm^3	1.198264	E-01
	lbm/ft^3	kg/m^3	1.601846	E+01
	lbm/ft^3	g/cm^3	1.601846	E-02
	g/cm^3	kg/m^3	1.0	E+03
	g/cm^3	kg/dm^3	1.0	
	˚API	specific gravity	141.5/(131.5+˚API)	
ρma = density (solids)	lbm/ft^3	kg/m^3	1.601846	E+01
ν = viscosity (Kinematic)	cm^2/s	mm^2/s	1.0	E+02
	ft^2/hr	mm^2/s	2.58064	E+01
	cST mm^2/s	1.0		
μ = viscosity (dynamic)	$dyne\text{-}s/cm^2$	Pas	1.0	E-01
	cP	Pas	1.0	E-03
	lbm/(ft-hr)	Pas	4.133789	E-04

Table 8-11—Units and Conversions (Continued)

Quantity	Customary unit	Metric unit SPE preferred	Conversion factor multiply customary unit by factor to get metric unit	
	cP	$(Ns)/m^2$	1.0	E-03
σs = surface tension	dyne/cm	m(milli)N/m	1.0	
γg = interfacial tension (IFT)	dyne/cm	m(milli)N/m	1.0	
k = absolute/specific permeability	darcy (d)	μm^2	9.869233	E-01
	millidarcy (md)	μm^2	9.869233	E-04
	microdarcy (μmd)	μm^2	9.869233	E-07
ω = angular velocity	rpm	radian/s	1.047198	E-01

Table 8-12—Nomenclature*

k_a	=	air permeability, md
k_h	=	horizontal permeability, md
k_v	=	vertical permeability, md
k_∞	=	equivalent liquid permeability (Klinkenberg corrected), md
k_{90}	=	permeability 90 degrees from horizontal permeability (k_{max}), md
k_{max}	=	higher of the k_h measured values in two directions 90 degrees apart
°C	=	degree Centigrade
°F	=	degree Fahrenheit
°R	=	degree Rankine
°K	=	degree Kelvin
cm^3	=	cubic centimeter
dm^3	=	cubic decimeter
ft	=	feet
ft^2	=	square feet
in	=	inch
m	=	meter
m^2	=	square meter
mm^2	=	square micrometer = square micron
μm^2	=	square micrometer
Pa	=	pascal
kPa	=	kilo pascal
kg	=	kilogram
g	=	gram
lbm	=	pound mass
lbf	=	pound force
lbm/ft^3	=	$pound/ft^3$
kg/m^3	=	$kilogram/cubic\ meter$
g/m^3	=	$gram/cubic\ meter$
g/cm^3	=	g/ml = gram/cubic centimeter = gram/milliliter
lbm/US	=	pound/US gallon
cST	=	centistokes
cP	=	centipoise
d	=	day
s	=	second
S_g	=	gas saturation, percent of pore volume
S_o	=	oil saturation, percent of pore volume
S_w	=	water saturation, percent of pore volume
ϕ	=	porosity, percent bulk volume
ϕ_e	=	effective porosity, percent of bulk volume
ϕ_t	=	total porosity, percent of bulk volume
PV	=	pore volume
BV	=	bulk volume
GV	=	grain volume
V_g	=	gas volume
V_o	=	oil volume
V_w	=	water volume
ρg	=	grain density
ρb	=	bulk density

*The SI Metric System of Units and SPE Metric Standard, SPE, Dallas, 1984.

API Related Publications Order Form

Date: _____ *(Month, Day, Year)*

❑ API Member *(Check if Yes)*

Invoice To – ❑ Check here if same as "Ship To"

Company:

Name/Dept.:

Address:

City: State/Province:

Zip: Country:

Customer Daytime Telephone No.:

Fax No.:

Ship To – (UPS will not deliver to a P.O. Box)

Company:

Name/Dept.:

Address:

City: State/Province:

Zip: Country:

Customer Daytime Telephone No.:

Fax No.:

(Essential for Foreign Orders)

❑ Payment Enclosed $ _____

❑ Payment By Charge Account:

❑ MasterCard ❑ Visa ❑ American Express

Account No.:

Name (As It Appears on Card):

Expiration Date:

Signature:

❑ Please Bill Me

P.O. No.:

Customer Account No.:

State Sales Tax – The American Petroleum Institute is required to collect sales tax on publications mailed to the following states: AL, AR, CT, DC, FL, GA, IL, IN, IA, KS, KY, ME, MD, MA, MI, MN, MO, NE, NJ, NY, NC, ND, OH, PA, RI, SC, TN, TX, VT, VA, WV, and WI. Prepayment of orders shipped to these states should include applicable sales tax unless a purchaser is exempt. If exempt, please print your state exemption number and enclose a copy of the current exemption certificate.

Exemption Number: State:

Quantity	Order Number	Title	SO*	Unit Price	Total
		RP 39, *Recommended Practices on Measuring the Viscous Properties of a Crosslinked Water-Based Fracturing Fluid (Coming Summer, 1998)*			
		RP 45, *Analysis of Oilfield Waters (Coming Fall, 1998)*			

Shipping and Handling – All orders are shipped via UPS or First Class Mail in the U.S. and Canada. Orders to all other countries will be sent by Airmail. U.S. and Canada, $5 per order handling fee, plus actual shipping costs. All other countries, for Airmail (standard service) add 25% of order value. All other countries, for UPS Next Day, add an additional 10% of order value.

Rush Shipping Charge – FedEx, $10 in addition to customer providing FedEx account number: _____. UPS Next Day, $10 plus the actual shipping costs (1-9 items). UPS Second Day, add $10 plus the actual shipping costs (1-9 items).

Rush Bulk Orders – 1-9 items, $10. Over 9 items, add $1 each for every additional item.

NOTE: Shipping on foreign orders cannot be rushed without FedEx account number.

Subtotal	
State Sales Tax *(see above)*	
Rush Shipping Charge *(see left)*	
Shipping and Handling *(see left)*	
Total *(in U.S. Dollars)*	

**To be placed on Standing Order for future editions of this publication, place a check mark in the space provided. Pricing and availability subject to change without notice.*

Mail Orders: American Petroleum Institute, Order Desk, 1220 L Street, N.W., Washington, DC 20005-4070

Fax Orders: (202) 962-4776 Phone Orders: (202) 682-8375

To better serve you, please refer to this code when ordering: G M 4 3 2 6 0 2 9 8

The American Petroleum Institute provides additional resources and programs to industry which are based on API Standards. For more information, contact:

• Training and Seminars	Ph: 202-682-8490 Fax: 202-682-8222
• Inspector Certification Programs	Ph: 202-682-8161 Fax: 202-962-4739
• American Petroleum Institute Quality Registrar	Ph: 202-962-4791 Fax: 202-682-8070
• Monogram Licensing Program	Ph: 202-962-4791 Fax: 202-682-8070
• Engine Oil Licensing and Certification System	Ph: 202-682-8233 Fax: 202-962-4739
• Petroleum Test Laboratory Accreditation Program	Ph: 202-682-8064 Fax: 202-962-4739

In addition, petroleum industry technical, patent, and business information is available online through API EnCompass™. Call 212-366-4040 or fax 212-366-4298 to discover more.

To obtain a free copy of the API Publications, Programs, and Services Catalog, call 202-682-8375 or fax your request to 202-962-4776. Or see the online interactive version of the catalog on our World Wide Web site – http://www.api.org.

American Petroleum Institute

Helping You Get The Job Done Right.

Additional copies available from API Publications and Distribution: (202) 682-8375

Information about API Publications, Programs and Services is available on the World Wide Web at: http://www.api.org

American Petroleum Institute

1220 L Street, Northwest
Washington, D.C. 20005-4070
202-682-8000

Order No. G40002

Recommended Core Handling/Preservation Field Procedures

Introduction

Following are recommended field procedures for handling, preserving and shipping unconsolidated cores obtained from shallow borings. Coring methods are beyond the scope of these procedures, as every site will have it's own unique problems and those are best addressed by the consultant and coring companies involved.

The goal of any coring operation is to obtain representative, *undisturbed* samples. If the core is being submitted to a laboratory for petrophysical analysis, it is recommended that the laboratory be contacted in advance to discuss the test program and required core size. Often, a compromise must be met between what the laboratory requires and what is practical in the field. Generally a 2" diameter by 6" long sleeve is suitable for most basic tests; horizontal and vertical permeability, TOC, density, grain size, porosity and pore fluid saturations. If larger diameter/continuous coring equipment is available, then a more detailed lithological and petrophysical data profile can be generated.

Procedure

1. Remove sample from core sampler as soon as possible.
   a. If core is in sleeves, fill any void space with saran wrap to minimize core movement, then seal with Teflon film and tape on plastic end caps.
   b. If core is not in sleeves, slide gently from sampler on to split PVC core supports - contact PTS for details. Wrap with Saran and secure with clear box tape.
2. Label each core section with top and bottom depths. Fractions of a foot should be recorded in tenths. Additionally, label multiple sleeves sequentially with A, B, C... etc starting with A on the top (shallowest) sleeve.
3. Immediately place cores in a cooler containing dry ice and freeze to minimize migration of core fluids (alternative: pack core into an ice chest with frozen “Blue Ice” and foam packing/cushioning material).
4. Ship cores at the end of each day by overnight courier (FedEx or AirBorne Express, etc.) to PTS Laboratories. Contact PTS for labeling requirements.

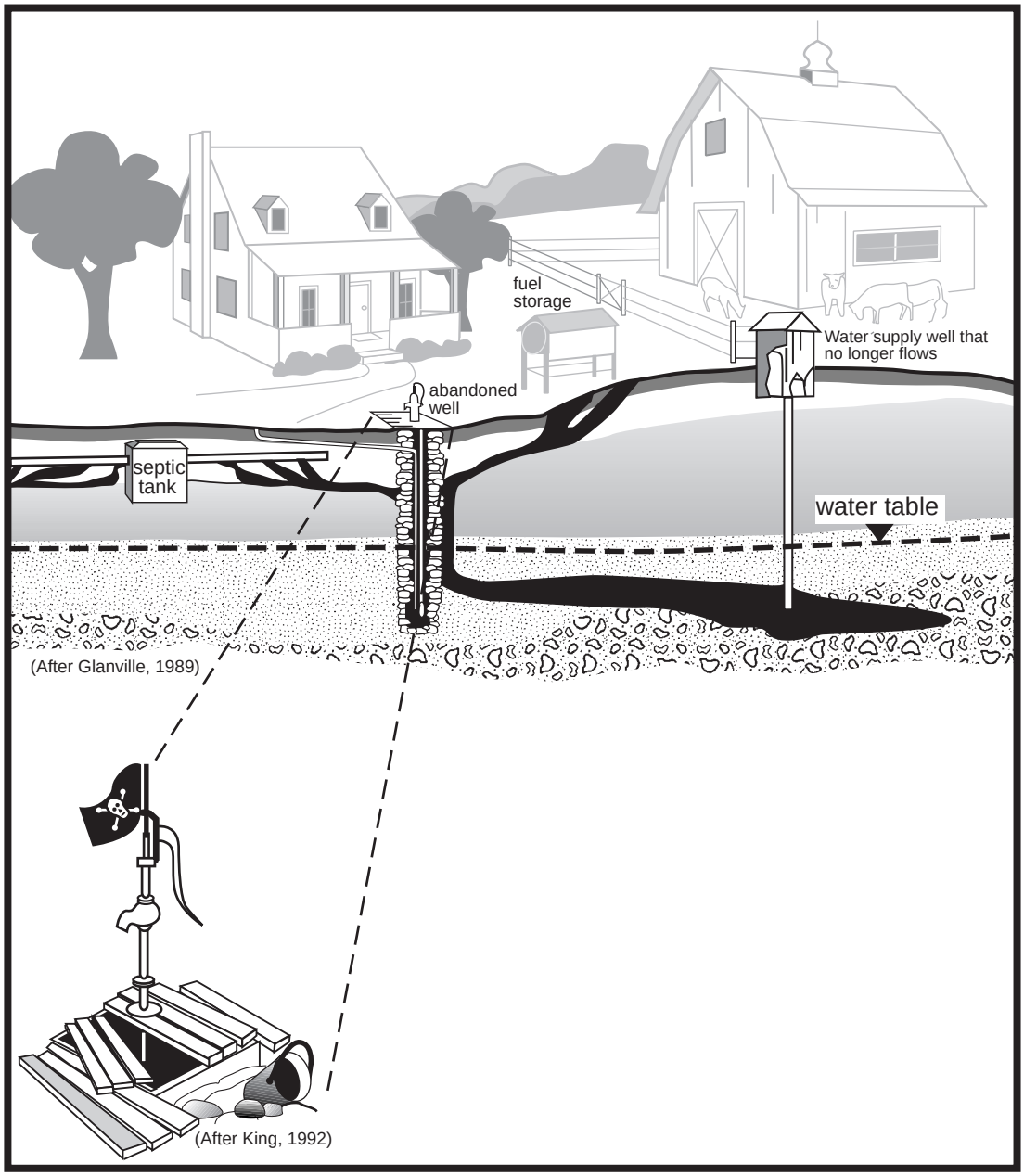
Applicable ASTM Standards

D3550-84: Ring Lined Barrel Sampling of Soils; split spoon or one-piece sampling barrel.
D1587-83: Thin-Walled Tube Sampling of Soils; Pitcher/Shelby tubes.
D4220-89: Preserving and Transporting Soil Samples; basic, does not address contaminated samples.

8100 Secura Way – Santa Fe Springs, CA 90670
Phone 562.347.2500 Fax 562.907.3610
www.ptslabs.com

ATTACHMENT B - ODNR WELL ABANDONMENT GUIDANCE

State of Ohio
Technical Guidance
for
Sealing Unused Wells



by the

State Coordinating Committee on Ground Water
1996

TECHNICAL GUIDANCE FOR SEALING UNUSED WELLS

BY THE

STATE COORDINATING COMMITTEE ON GROUND WATER

1996

Table of Contents

Table of Contents ii

List of Figures iii

List of Tables iii

Table of Contents continued iii

Preface iv

Acknowledgements v

Introduction 1

Overview of Regulations 1

Reasons to Properly Seal an Unused Well 3
- Eliminate Physical Hazard 3
- Prevent Ground Water Contamination 3
- Prevent Further Loss of Confining Pressure 3
  - Who Should Perform Well Sealing? 5

Types of Wells Defined by Method of Construction 5
- Dug Wells 5
- Driven Wells 5
- Drilled Wells 6
  - Bored 6
  - Cable Tool 7
  - Rotary 7
  - Vibratory 9

Types of Wells Defined by Aquifer Characteristics 9

Preparation for Sealing 12
- Well Information 12
- Casing Issues 12

Sealing Materials 14
- Cement–Based Grouts 14
  - Cement Properties 14
  - Cement Types 16
  - Neat Cement Grout 16
  - Concrete Grout 17
  - Other Cement Additives 17
- Bentonite—Based Grouts 17
  - Clay Properties 17
  - Properties of Bentonite/Water Slurries 17
  - High-Solids Bentonite Grout 18
  - Powdered Bentonite/Clay Grout 19
  - Granular Bentonite Slurries 19
  - Coarse Grade Bentonite 19
  - Pelletized Bentonite 20
  - Availability Of Bentonite Grout Materials 20
- Fill Materials 20

Procedures for Sealing the Well 20
- General Sealing Procedures 20
- Specific Well Sealing Procedures 23
  - Sealing Dug and Bucket-Drilled Wells 23
  - Sealing Driven Wells 24
  - Sealing Wells Drilled Through a Single Unconsolidated Aquifer or an Unconfined, Unconsolidated Aquifer 24
  - Sealing Wells Drilled Through Multiple Unconsolidated Aquifers 25
  - Sealing Wells Drilled Through Confined, Unconsolidated Aquifers 26
  - Sealing Wells Drilled Through Single Consolidated Aquifers 28
  - Sealing Wells Drilled Through Multiple Consolidated Aquifers 28
  - Sealing Wells Drilled Through Fractured or Cavernous Formations 29
  - Sealing Flowing Wells Drilled Through Single or Multiple Consolidated Aquifers 30
  - Sealing Wells of Unknown Construction 30

Table of Contents *continued*

Conclusions 30
References 32
Glossary 34
Appendix 1 37
Contact Agencies 37
Appendix 2 39
Cost Considerations 39
Appendix 3 41
List of Acronyms 41
Appendix 4 42
Sealing Monitoring Wells and Boreholes 42
Sealing Materials 42
Procedures 42
Planning 42
Field Procedure 43

List of Figures

Figure A. Example well sealing report 2
Figure B. Reasons to properly seal an unused well 4
Figure D. Driven well 6
Figure C. Typical dug well design 6
Figure E. Well construction using hollow stem augers 8
Figure F. Cable tool-drilled wells 8
Figure G. Rotary-drilled well with screen, developed in a sand and gravel aquifer 9
Figure H. Generalized map of ground water resources in Ohio 10
Figure I. Confined and unconfined aquifers 11
Figure J. Example well log and drilling report 13
Figure K. Grouting bedrock well using tremie pipe method 22
Figure L. Sealing wells with coarse grade bentonite products using pouring method 23
Figure M. Sealing procedures for dug or other shallow, large diameter wells 24
Figure N. Methods for sealing wells penetrating multiple aquifers 25
Figure O(1). Reducing or stopping flow of well by casing extension 26
Figure O(2). Using inflatable packer to restrict flow 27
Figure O(3). Pouring disinfected gravel into well to reduce flow 28
Figure P. Sealing procedures for wells penetrating fractured or cavernous formations 29

List of Tables

Table 1 Grout Properties 15
Table 2 Permeability of Various Sealing Materials 15
Table 3 Cement Curing Time Required 15
Table 4 Grout Slurry Densities 18
Table 5 Summary of Recommended Well Sealing Procedures 31
Table 6 Table Comparing Volumes of Different Well Sealing Materials Required to Seal a 100 Foot Well 40

Preface

In early 1992, Ohio's State Coordinating Committee on Ground Water identified a list of major issues and problems that they determined should be addressed in some form by the Committee. The issue of the lack of consistent standards and regulations regarding the sealing of abandoned wells and test borings was identified as a major issue of concern by the Committee. The Natural Resource Conservation Service (NRCS, formerly the Soil Conservation Service) approached the Committee in the spring of 1994 regarding a new USDA cost sharing program for sealing abandoned wells that could be implemented by the states. The program required that the states develop technical standards that would be adopted by the state technical committee at the NRCS. Once these technical standards were adopted, counties participating in the cost share program could choose to offer cost share funds to farmers for sealing abandoned wells on agricultural properties. Based on this new opportunity from the NRCS, and due to increasing concerns by many of the participating state agencies and the well drilling industry, the Committee decided to form a workgroup in June, 1994 to develop consistent technical standards for sealing abandoned wells and test borings. Both the Ohio Environmental Protection Agency and the Ohio Department of Health have committed to revising their rules regarding well sealing to be consistent with the resulting new technical guidance document. The Well Sealing Workgroup began meeting in July, 1994; what follows is the product of eighteen months of meetings, research, edits, and revisions.

Throughout this document are references to proprietary materials or products. These references should in no way be interpreted as endorsements for any particular brand name or manufacturer, and are used only for illustrative or comparative purposes.

This guidance does not apply to wells constructed for the purpose of injecting fluids into the subsurface (except as it may augment, not supersede, rule requirements). The authority over injection wells depends on the well classification. For more information contact the Ohio Environmental Protection Agency, Division of Drinking and Ground Waters, Underground Injection Control Unit.

Acknowledgements

The preparation of the Technical Guidance for Sealing Unused Wells involved the contribution and hard work of a number of individuals on the Well Sealing Workgroup of the State Coordinating Committee on Ground Water. The development of the Technical Guidance was supported by the State Coordinating Committee on Ground Water and its member agencies. Special appreciation and acknowledgement is given to Katherine Sprowls for her extensive work in organizing, editing and authoring these guidelines. The workgroup also thanks the many industry professionals who graciously took the time to review and provide comments on the guidelines. In addition, the following are deserving of special thanks for the time and effort they devoted to generating the figures found in the guidelines: Janet Welday of the U. S. Geological Survey, Anne Mischo of the Ohio State University Extension, and Wayne Jones and David Orr of the Ohio Department of Natural Resources. Grateful acknowledgement is given to the following workgroup members for their technical research and text authorship, report editing and preparation:

Well Sealing Workgroup Members

Katherine Sprowls (*Editor*)	Division of Water, Ohio Department of Natural Resources
Rebecca Petty (*Workgroup Chair*)	Division of Water, Ohio Department of Natural Resources
Robert Van Horn	Division of Geological Survey, Ohio Department of Natural Resources
Lisa Koenig	Division of Drinking and Ground Waters, Ohio Environmental Protection Agency
Scott Golden	Bureau of Local Services, Ohio Department of Health
Russell Smith	Bureau of Local Services, Ohio Department of Health
Kevin Hodnett	State Fire Marshal, Ohio Department of Commerce
Larry Berger	Division of Plant Industry, Ohio Department of Agriculture
Denise Dumouchelle	United States Geological Survey
Ken Wolfe	Natural Resource Conservation Service
Hewitt Fredebaugh, Jr.	Fredebaugh Well Drilling
Alan Belasco	Belasco Drilling

State Coordinating Committee on Ground Water Member Agencies

Ohio Environmental Protection Agency

Ohio Department of Natural Resources

Ohio Department of Health

Ohio Department of Agriculture

Ohio Public Utilities Commission

Ohio Department of Development

Ohio Department of Commerce - State Fire Marshal

Ohio Department of Transportation

United States Geological Survey

Natural Resources Conservation Service

—Rebecca Petty

Introduction

An unused or abandoned water **well**[1] is a well that is no longer in service or is in such a state of disrepair that continued use for the purposes of accessing **ground water** is unsafe or impracticable. Abandoned wells can be found almost anywhere: on farms, industrial sites, and in urban areas. Those marked by windmill towers and old hand pumps are easy to spot. Many lie hidden beneath weeds and brush. These wells are open traps waiting for unsuspecting children, hunters, and animals (Gordon, 1988). In addition, wells are often abandoned when their **yield** has diminished, or the quality of the water they supply has degraded. Each year, many wells are abandoned when homes are connected to community water supplies. No accurate accounting of abandoned wells exists for the State of Ohio. However, it has been estimated that there could be more than 200,000 unused wells in the state (Golden, pers. comm., 1995).

The number of potential contaminants that may enter these wells is unlimited. Fuel, fertilizer, solvents, sewage, animal waste, pesticides and numerous other contaminants have been introduced into ground water through improperly sealed abandoned wells. If a substance can be dissolved, carried, or mixed in water, it has the potential for entering ground water through an improperly sealed abandoned water well (King, 1992).

This guidance outlines the materials and methodologies that should be used to properly seal a well. The intent of this guidance is to provide a comprehensive discussion of all elements involved in the well sealing process, including basic ground water principles and an introduction to well drilling and construction methods. Readers familiar with these topics can move directly to the sections dealing with well sealing procedures, which start on page 20.

In addition, this guidance covers the procedures for sealing **monitoring wells** and **boreholes**. Readers interested in these procedures are probably already conversant with most of the information presented throughout this document. Therefore, the procedures for sealing monitoring wells and boreholes are addressed separately in Appendix 4.

Any well to be abandoned should be sealed to prevent vertical movement of water. The sealing method chosen should be dependent on both well construction and site **geologic/hydrogeologic conditions**. Whenever there is doubt about either the construction of the well or the site hydrogeology, the choices of sealing material and procedure should be those affording the greatest probability of providing a permanent seal.

Overview of Regulations

Current regulations for private (Ohio Administrative Code (OAC) 3701-28-07) and public water wells (OAC 3745-9-10) require that boreholes not converted into wells, and wells not being used to obtain water or provide information on quality, quantity, and water level, be sealed. However, the regulations specify only that the well/**test boring** be completely filled with **grout** in order to seal the **aquifer** and protect the ground water.

The authority for enforcement for public water supplies is the Ohio Environmental Protection Agency (EPA)/Division of Drinking and Ground Waters (DDAGW) Drinking Water Program and the authority for enforcement for private wells is the Ohio Department of Health (ODH) and local health departments.

Proper sealing of all abandoned wells must be documented per the Ohio Revised Code (ORC) Section 1521.05 (B). A well sealing report (Figure A) must be submitted to the Ohio Department of Natural Resources (ODNR), Division of Water. An appropriate form can be requested from the ODNR (614-265-6739). Also, ODH's Private Water Systems Rules require that the local health department be notified when an abandoned well has been sealed. In most counties, sending the local health department a copy of ODNR's well sealing report is an acceptable form of notification.

[1] All terms in bold print can be found in the glossary.

WATER WELL SEALING REPORT
(For Abandoned or Unused Wells)
OHIO DEPARTMENT OF NATURAL RESOURCES
Division of Water, Water Resources Section
1939 Fountain Square Drive
Columbus, Ohio 43224-1360

LOCATION

County DELAWARE Township GENOA Section 4
Property Owner E. J. FUDD
Address of Property 12345 SMOTHERS RD., WESTERVILLE, OH, 43081
Location: 1/2 miles EAST (n, e, s, w) of SUNBURY RD. (nearest intersection)

on the NORTH (n, e, s, w) side of SMOTHERS ROAD (road)
name

ORIGINAL WELL

ODNR Well Log Number N/A Copy attached? Yes or (No)
(circle one)

MEASURED CONSTRUCTION DETAILS Date of measurements 8/31/92

Depth of Well 101.5 Static Water Level 14.5
Size of Casing 8 INCH Length of casing ?
Well Condition ABANDONED

SEALING PROCEDURE

Method of Placement PRESSURE GROUT - 1" TREMIE TUBE

Placement:	From	To	Sealing Material	Volume
	101.5	SURFACE	BENSEAL/E-Z MUD	385 GAL

Was Casing Removed? Yes or (No)
(circle one)
Condition of Casing GOOD
Perforations: From To
From To
Date Sealing Performed 8/31/92
Reason(s) for Sealing WELL ABANDONED - NO LONGER NEEDED AND IN THE WAY OF CONSTRUCTION

CONTRACTOR

Name ACME DRILLING COMPANY ODH Registration # 3456
Address 1234 MAIN ST.
City/State/Zip SOCKERDOWNE, OH, 56789 Signature W. E. Coyote

DNR 7810.93 **SUBMIT COMPLETED FORM TO ODNR-DIVISION OF WATER**

Figure A. Example well sealing report

Reasons to Properly Seal an Unused Well

There are many reasons for properly sealing unused wells. The most important of these include: eliminating physical hazards, preventing ground water contamination, and preventing further loss of confining pressure in **confined aquifers**.

Eliminate Physical Hazard

One of the most obvious reasons to properly seal a well is the physical hazard (Figure B). A good example of this danger was seen a few years ago as the nation witnessed the rescue of a small child from an unsealed abandoned water well that was less than 10 inches in diameter. It is also quite common to find animal remains in unsealed abandoned wells. Other than being the reason for the unfortunate creature's demise, an additional hazard is the possible bacterial contamination of the aquifer caused by the decay of the animal. There also have been cases cited where improperly sealed **geotechnical borings**, used to obtain **stratigraphic** information during highway construction, have caused potholes to occur in newly-constructed highways (Smith, 1994). Geotechnical borings are often drilled on farmland that is in the process of being sold for commercial or industrial use. Imagine the consequences if the farmer's prize-winning Guernsey (or other livestock, for that matter) steps into an open borehole and breaks a leg. These are just a few of the hazards that could result from the existence of unsealed abandoned wells of any type.

Prevent Ground Water Contamination

Another reason to properly seal a well is to prevent ground water contamination (Figure B). There are four ways that an unsealed abandoned well could contaminate the ground water: by intermixing of waters between aquifers, by surface water entering the aquifer, by direct disposal of contaminants down the well, and by bacterial contamination from decomposition of animal bodies and waste products.

Poorly constructed wells or wells that are screened across multiple aquifers can cause intermixing of water between the aquifers. Depending upon the hydrogeologic conditions, poor quality water can move upward or downward into a pristine aquifer. Ground water zones penetrated by a well may have physical or chemical qualities that are incompatible. Chemical reactions may occur that result in undesirable products such as iron sulfides and calcium sulfate (Smith, 1994).

Contaminated surface water can enter a well if the well cap has been broken or removed, or if there are holes in the well **casing** due to damage or deterioration with age. In addition, contaminated surface water can seep down along the space (called the **annular space**) between the casing and the borehole wall of an improperly grouted well. This is an important consideration because most older water wells do not meet today's construction standards.

Illegal direct disposal of contaminants down unused wells is a common occurrence. Open wells offer tempting disposal receptacles for liquid and solid waste. People seem naturally compelled to throw or pour unwanted material down an open hole (Smith, 1994).

Abandoned wells are often preferred havens for a host of arthropods (spiders, earwigs, and centipedes) that prefer dark, moist, calm places. Also these can become subsurface dwellings for rodents and reptiles. The bodies and waste products of these colonists add nutrients and undesirable bacteria to the ground water (Smith, 1994).

Prevent Further Loss of Confining Pressure

It is important to seal a well penetrating a confined aquifer(s) to preserve the confined (or "pressurized") conditions (Figure B). These confining conditions allow the water to reach a certain level in a well (called the **static level**). The static level will be higher than the depth at which the water is encountered in the aquifer; in some cases, the well will flow because the static level is higher than the ground surface. A reduction in the confining pressure may cause water levels in neighboring wells to drop because the **hydraulic head** is no longer high enough to allow the water in these wells to maintain their original static levels. Reduced confining pressures may result from water in a deeper aquifer moving upward into **formations** containing no water, or into **saturated zones** of lower hydraulic head. In the case of flowing wells, the pressure can be reduced simply because of the constant flow of

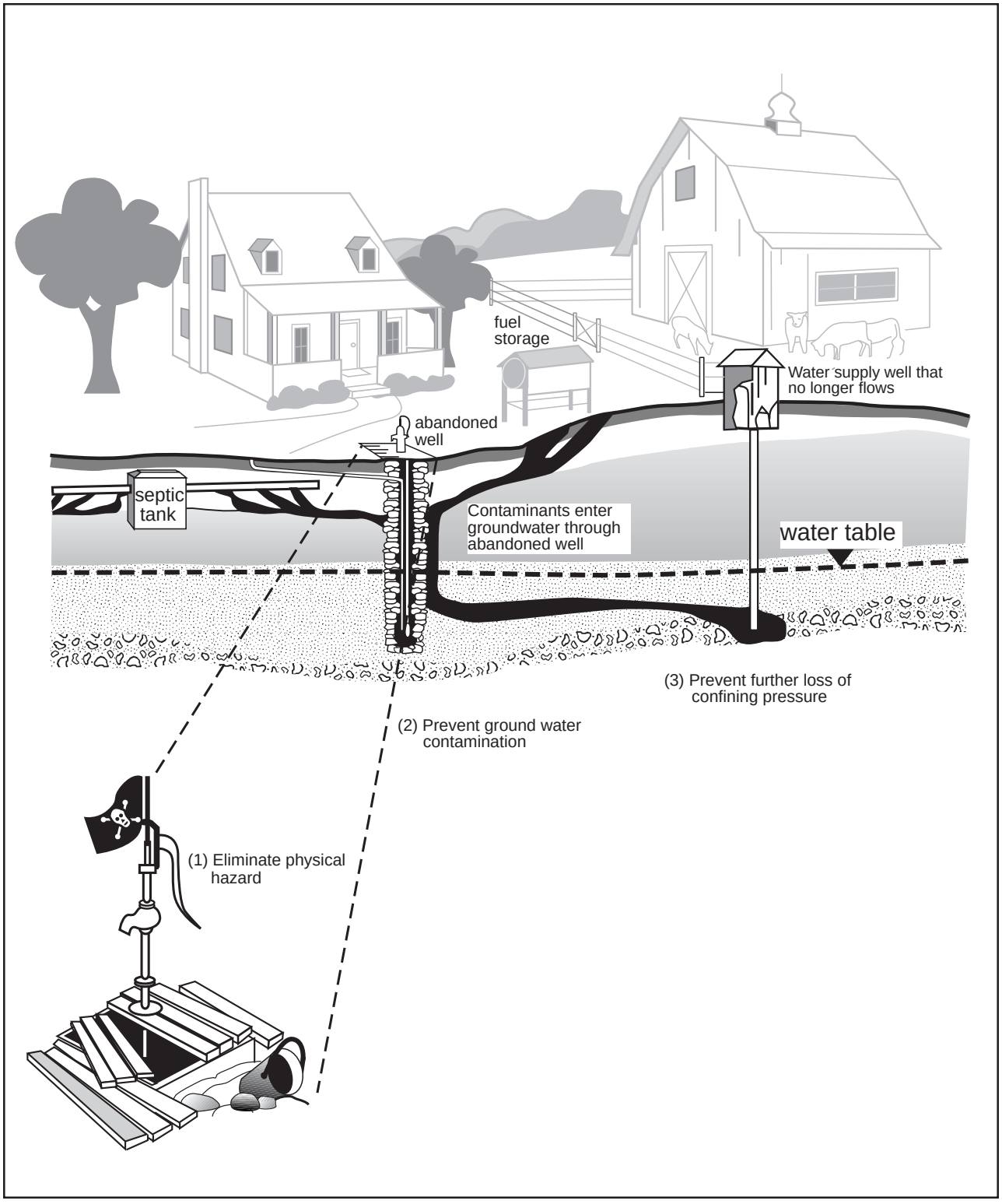


Figure B. Reasons to properly seal an unused well
(After Glanville, 1989, and King, 1992)

water from the aquifer onto the ground surface. Therefore, it is especially important to ensure that abandoned wells penetrating confined aquifers are properly sealed.

Who Should Perform Well Sealing?

It may seem that anyone who can rip open a bag of grout and pour it down the hole could seal a well. However, effective well sealing requires skill and cannot be done carelessly. A sealed-in leaking shaft or hole will most likely result if too little grout is emplaced, or it is mixed improperly, or seals are not set at the right depths, or annulus openings are left outside the casing. The consequence is continued threats to other wells and direct pathways to the ground water (Smith, 1994).

There are no qualifications specified in regulation for persons who can perform well sealing. Based on the difficulty in sealing many wells, and the equipment and knowledge involved, it is strongly recommended that well sealing be completed by an experienced registered drilling contractor. Some work may also need to be supervised by a qualified hydrogeologist or qualified engineer. It should be noted that all professional contractors do not have the same experience. An experienced contractor should be able to provide a description of work to be performed and a list of references proving his/her qualifications. Personnel should be trained and equipped for sites with potential for exposure to contamination or other hazards (Smith, 1994).

In some instances, shallow large diameter **dug wells** can be successfully sealed by a non-professional with minimal amount of special equipment. Be aware, however, that once a well has been sealed improperly, it is costly to correct because the defective seal has to be drilled out. An experienced registered drilling contractor should be consulted, at a minimum, in all sealing situations. It is recommended that wells with one or more of the following characteristics be sealed by an experienced registered drilling contractor only:

- drilled wells,
- flowing wells,
- wells greater than 100 feet in depth,
- wells where water is seeping from around the casing,
- wells where pumping equipment is difficult to remove,
- wells which produce gas, and
- monitoring wells (Zahniser and Gaber, 1993).

Types of Wells Defined by Method of Construction

Wells can be described in different ways, either by their method of construction, or by the type of aquifer in which they are developed. To describe wells by their method of construction, it is necessary to understand what it takes to "make a well." There are three commonly used methods of well construction: digging (by hand or backhoe), driving, and drilling.

Dug Wells

Dug wells can be defined as any wells not installed by drilling rigs. They are usually large diameter (greater than 24 inches) and fairly shallow (25 feet or less, although dug wells deeper than 25 feet are not uncommon), and are constructed by digging with a backhoe or by hand (Figure C). Casing installed in dug wells can vary from concrete pipe and vitrified tile to cobbles and bricks. In some cases, dug wells are improperly used as **cisterns** for roof runoff or hauled water.

Driven Wells

Driven wells, for the purposes of this document, will refer to well points exclusively (Figure D). Well points are installed only in **unconsolidated** formations. Well points are typically small diameter, shallow wells used to supply water for a single household. Many of these wells are installed by the homeowners themselves. Well points consist of a **well screen** with a hardened point which is hammered into place (by hand or machine) using a large weight. Sections of pipe are added to the screen in order to advance the screen to the desired depth.

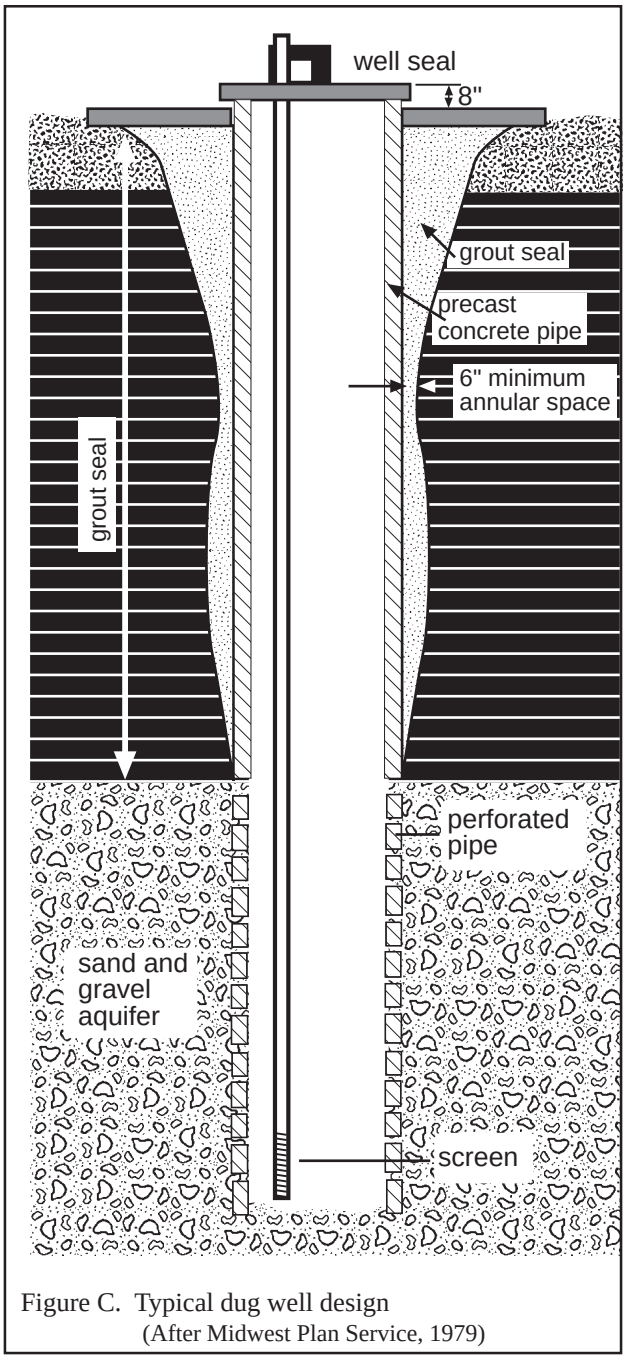


Figure C. Typical dug well design
(After Midwest Plan Service, 1979)

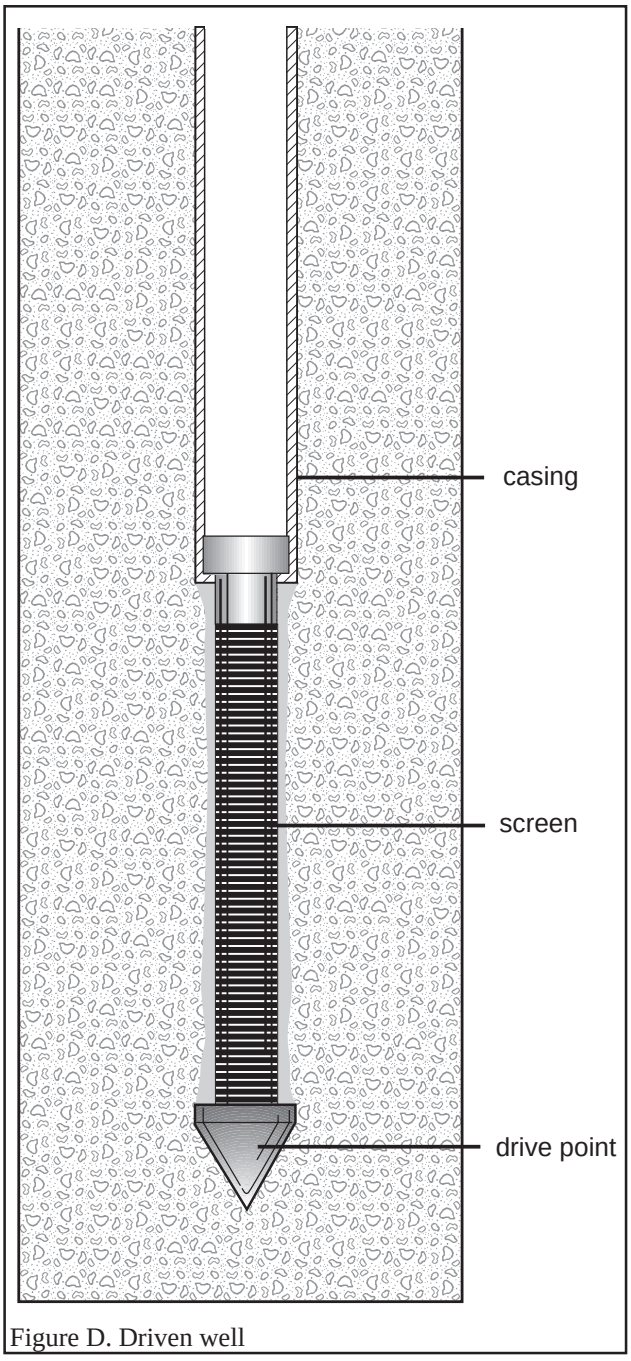


Figure D. Driven well

Drilled Wells

The third major category of well construction methods is that of drilled wells. Drilled wells are those that are constructed using machines designed specifically for the task of well installation. There are several drilling methods commonly used today: boring, cable tool, rotary, and vibratory drilling.

Bored

Bored wells are also known as augered wells and are used to construct wells in unconsolidated formations. There are three principal types of augers used for well drilling: bucket augers, solid-stem augers, and hollow-stem augers. The bucket auger has the largest diameter of the three types of augers, and is the most frequently used augering technique for water supply wells in Ohio. The bucket is cylindrical with hardened teeth on the bottom and has a diameter of 18" to 48". The bucket can remove 24" to 48" of material at a time. Wells drilled with a bucket auger normally range in depth from 50 to 150 feet, but in some areas they can reach 250 feet in depth (Driscoll, 1986). In Ohio, a

bucket-augered well generally is cased with concrete pipe or vitrified tile, and in many respects will resemble a dug well.

Solid-stem augers consist of spiral flanges welded to a pipe. One length of pipe (or auger section) is called a flight; multiple auger sections are often referred to as continuous flighting. The leading auger flight has a special bit or cutter head attached that cuts a hole for the flights to follow. Flights are added as the hole is drilled deeper. **Cuttings** from the drilling process are brought to the surface by the action of the augers (Driscoll, 1986). Boreholes constructed with solid stem augers are typically used for geotechnical, or, less commonly, environmental purposes, rather than water supply wells.

Hollow-stem augers are similar to solid stem augers in design, except that **drill rods** can pass through the auger sections. The leading drill rod has a pilot assembly attached to drill slightly ahead of the lead auger flight (Figure E). The outside diameter of these augers can range from 4 1/4" to 18", with corresponding inside diameters of 2 1/4" to 12 1/4". Because the flights are hollow, they can be used as temporary casing to hold the hole open while the permanent casing is installed. As the well is being installed, the augers are removed. Wells drilled with hollow stem augers have been used to construct water supply wells, but they are more often used to construct monitoring wells.

Cable Tool

Cable tool (sometimes called "spudder") rigs operate by repeatedly lifting and dropping a string of **drill tools** into the hole (Figure F). The **drill bit** at the bottom of the drill tools breaks or crushes the formation and when mixed with water forms a slurry. After drilling a certain number of feet, the bit and tools are pulled from the hole and the slurry is removed by **bailing**. In unconsolidated formations, casing is driven into the hole behind the drill bit so that the hole will stay open. When the desired depth has been reached, the casing can be pulled back to expose a screen, if one is to be installed. In some cases, drilling a well deep into unconsolidated sediments requires that two strings of casing be used; that is, the driller will start with a particular diameter casing, then when this size casing can no longer be driven, a smaller diameter casing is set inside the larger casing and the drilling and driving process continues until the well is completed. This technique works because the smaller diameter casing has less friction working against it. However, when drilling a well into the bedrock with a cable tool rig, usually a single casing string is set a few feet into the bedrock, and drilling continues open hole until the desired depth is reached. Cable tool drilling is still the most commonly used method of drilling water wells in Ohio. About 79 percent of the drilling contractors operating in the state of Ohio own cable tool rigs.

Rotary

Rotary rigs use one of two methods to rotate the drill bit: a table drive or top head drive. The rotation of the table or top head is transferred to the drill rods, which in turn rotate the bit. Mud rotary rigs use a roller cone bit at the end of the drill rods. The drill cuttings are circulated out of the hole with water or **drilling mud**. When the appropriate depth has been reached, the drill rods are withdrawn from the hole. The casing and screen (if needed) can then be set in the open borehole. Since it is necessary to drill an oversized borehole with this type of drilling method, the outside diameter of the well casing will be at least 2" smaller than the diameter of the borehole. Therefore, it is important that the space between the casing and borehole wall (annular space) be sealed to prevent contamination from the surface, and to hold the casing in place in the borehole.

Air rotary drilling rigs operate in basically the same way as mud rotary. However, instead of using drilling mud to clean the cuttings out of the borehole, a combination of compressed air and water is used. Air rotary rigs also run roller cone bits, but, in addition, they have the capability to run a down-the-hole hammer. The down-the-hole hammer is used for **consolidated** formations only. Compressed air is forced down the drill rods to operate the piston-like action of the hammer (bit). The hammer pulverizes the material being drilled. The air, in combination with water or foam, lifts the cuttings out of the hole. Hole sizes can range from 4 1/8" to 30" (Ingersoll-Rand Co., 1988). Usually a well will be drilled with mud through unconsolidated formations to the bedrock formation, if that is the aquifer. After the casing is set and grouted into place, the well can continue to be drilled with a combination of air and water until the desired depth is reached. Both methods of rotary drilling are frequently used in Ohio to construct water supply wells. Figure G shows typical rotary-drilled well construction.

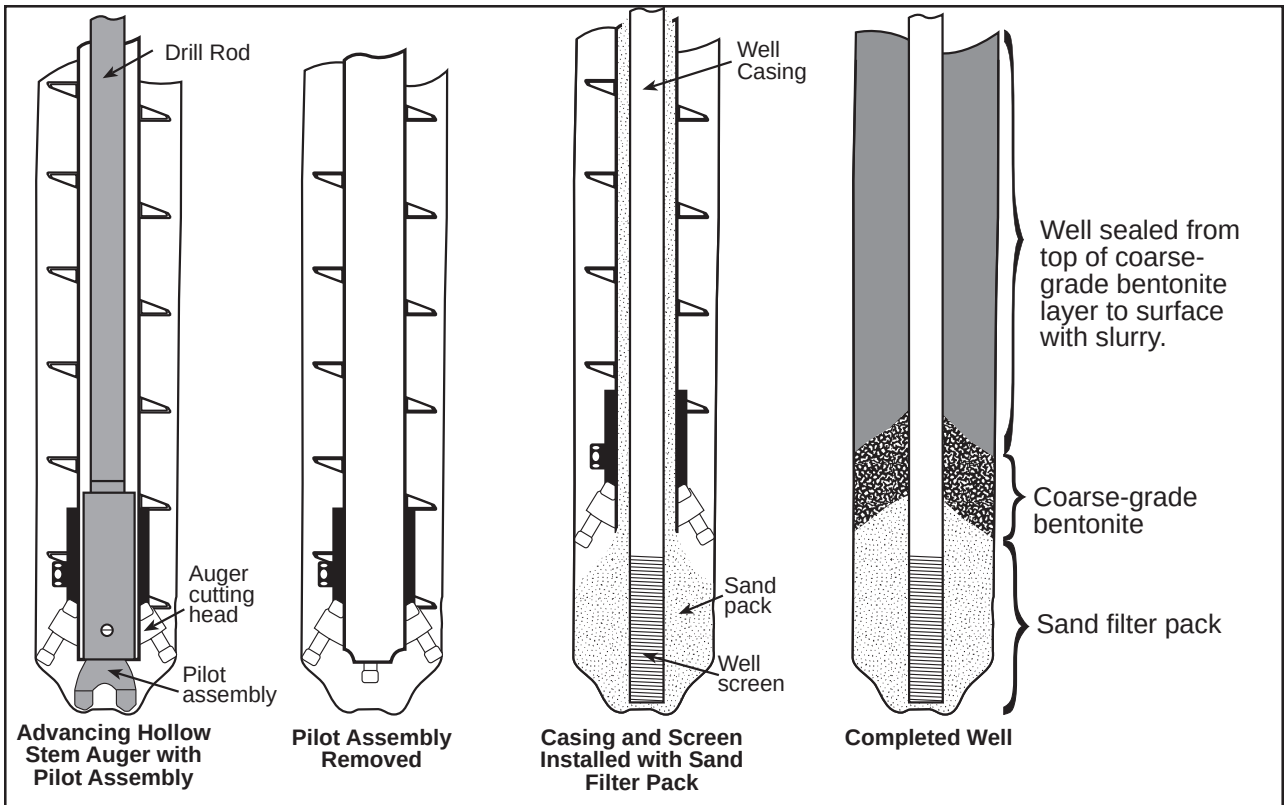


Figure E. Well construction using hollow stem augers
(Modified from Hackett, 1987 and 1988)

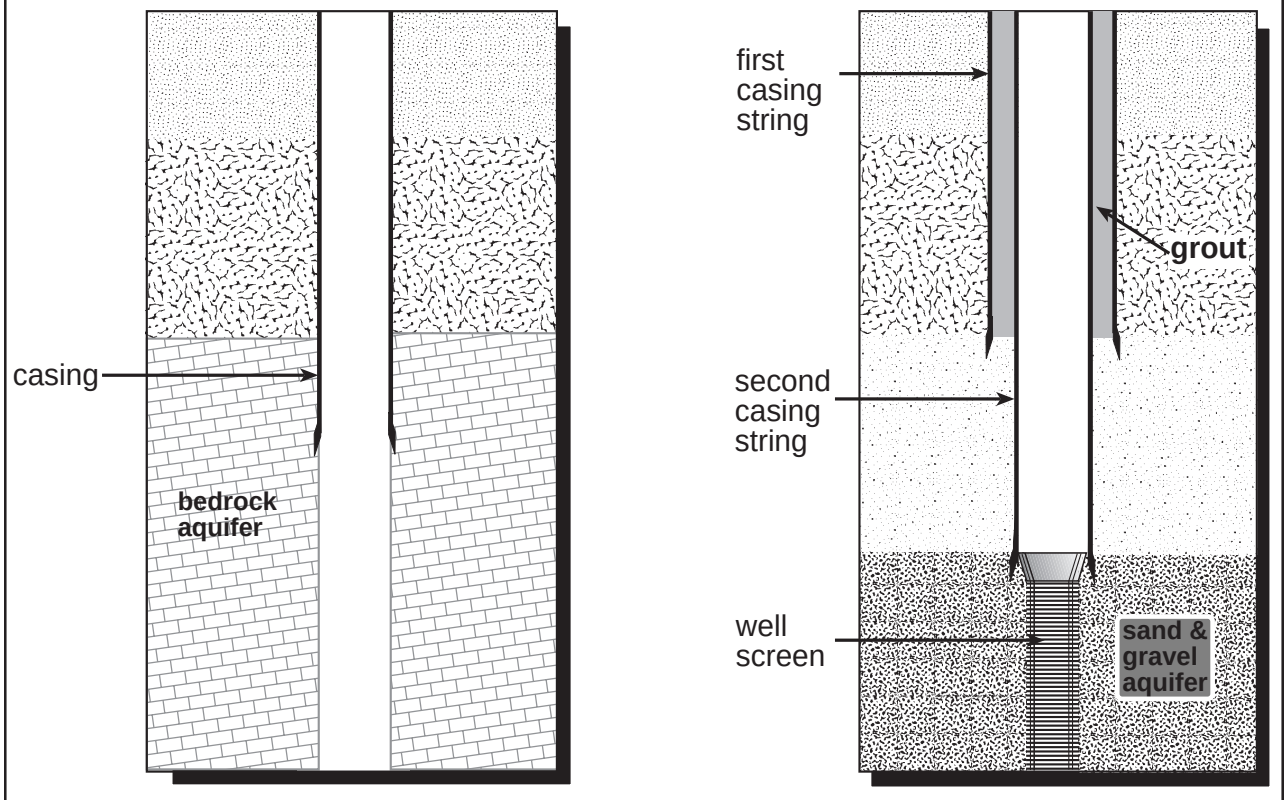


Figure F. Cable tool-drilled wells

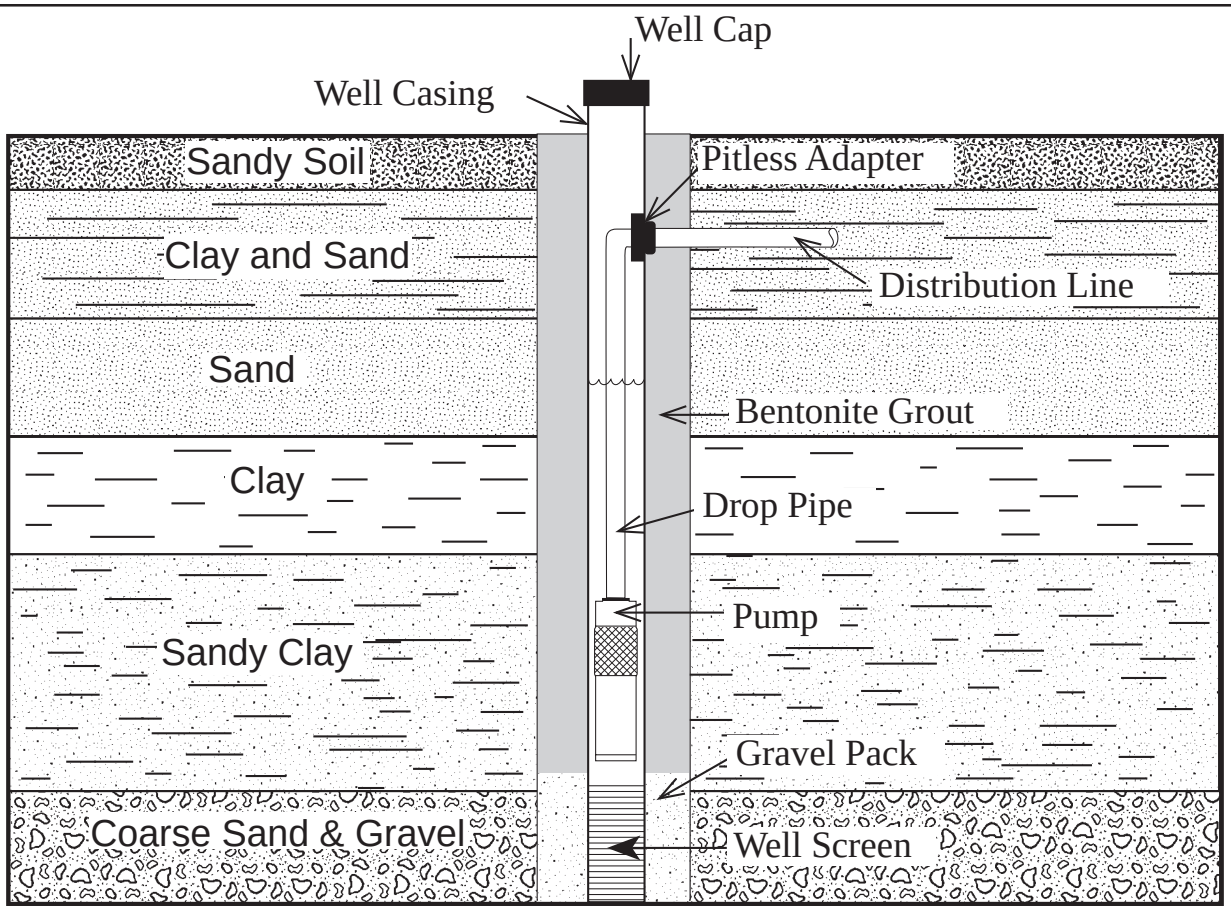


Figure G. Rotary-drilled well with screen, developed in a sand and gravel aquifer

Another method of rotary drilling is reverse rotary. Reverse rotary drilling is most often used to construct large diameter (24 inches or greater) water supply wells. Reverse rotary rigs are similar to air or mud rotaries in design, but are larger in size. The bit is rotated by table drive exclusively, as the top head drive does not develop enough torque to turn the size of the bit required to drill large diameter wells. The major difference between the reverse rotary and the other rotary methods described here is the pattern of fluid circulation. With reverse rotary, the drilling fluid is added to the borehole through the annular space, then the fluid and cuttings are removed from the hole by suction through the drill rods. The fluid and cuttings are deposited into a mud pit, where the cuttings settle out and the fluid is recirculated. The resulting large-diameter borehole allows easy installation of **filter pack** and well screens, which are necessary to properly develop high capacity wells in unconsolidated formations. Reverse rotary drilling can also be used in most consolidated formations (Driscoll, 1986).

Vibratory

Vibratory drilling involves the use of a resonance source through the drill rods to drill a hole to the desired depth. The resonance through the casing (rods in this case) pushes the cuttings into the side wall of the hole and into the center of the pipe. This method produces a minimal amount of cuttings, uses no drilling mud, and produces a continuous core. This drilling method is used mostly for geotechnical and environmental sampling purposes. Monitoring wells can be set through the casing if desired.

Types of Wells Defined by Aquifer Characteristics

Wells can be described by the types of aquifers in which they are developed. An aquifer is a geologic formation, group of formations, or part of a formation that is capable of yielding water to a well or spring. Figure H shows the statewide distribution of expected well yields from aquifers (this map is very general and should not be used to determine well yields on a site-specific basis). These

yields, are directly related to the types of geologic formations that comprise the aquifers. For example, the white areas on the map signify aquifers that typically yield less than five gallons per minute to a well. The aquifers in these areas generally consist of shales, or interbedded limestone and shale, or, in some cases, interbedded shale, limestone, and sandstone.

Geologic formations in Ohio will be either consolidated or unconsolidated. Consolidated formations are those that are lithified, that is, hardened into rock. A borehole penetrating a consolidated formation would be able to stay open indefinitely without the benefit of casing. Consolidated aquifers in Ohio generally consist of sandstone, limestone, or shale. The most productive consolidated aquifer is cavernous limestone. Sealing wells that penetrate fractured and cavernous rock may have special problems due to loss of grouting material.

Unconsolidated formations are usually soft and loose (there are some exceptions). Wells penetrating unconsolidated formations must be cased, otherwise the borehole walls will collapse. Unconsolidated aquifers in Ohio consist of silt, sand, gravel, or any combination of the three. Many of the state's most prolific wells are developed in sand and gravel aquifers.

Consolidated and unconsolidated aquifers can also be confined or unconfined. Unconfined aquifers are aquifers in which there are no **confining beds** between the zone of saturation and the surface (see Figure I for example). They are often referred to as water table aquifers. The upper surface of

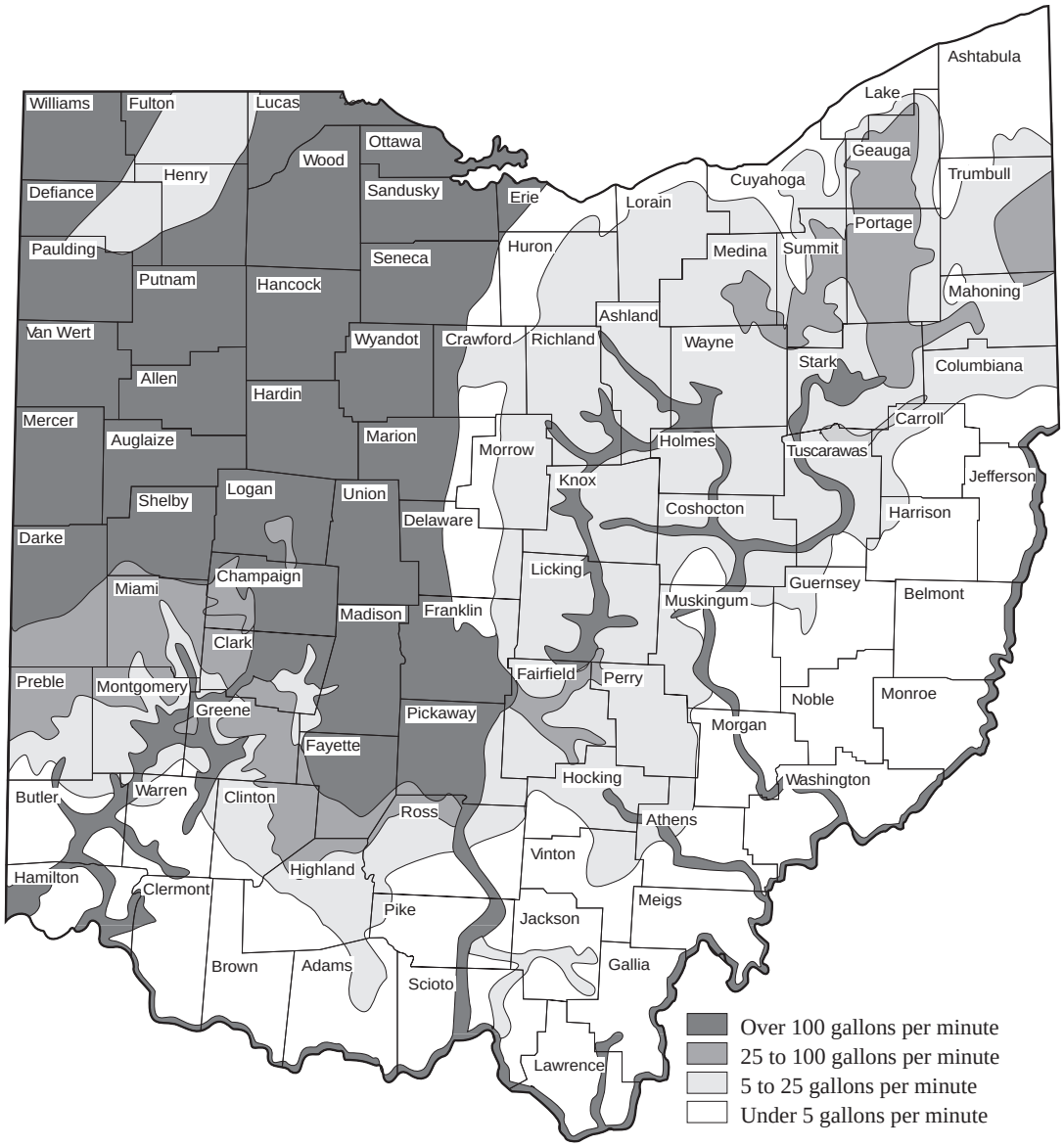


Figure H. Generalized map of ground water resources in Ohio

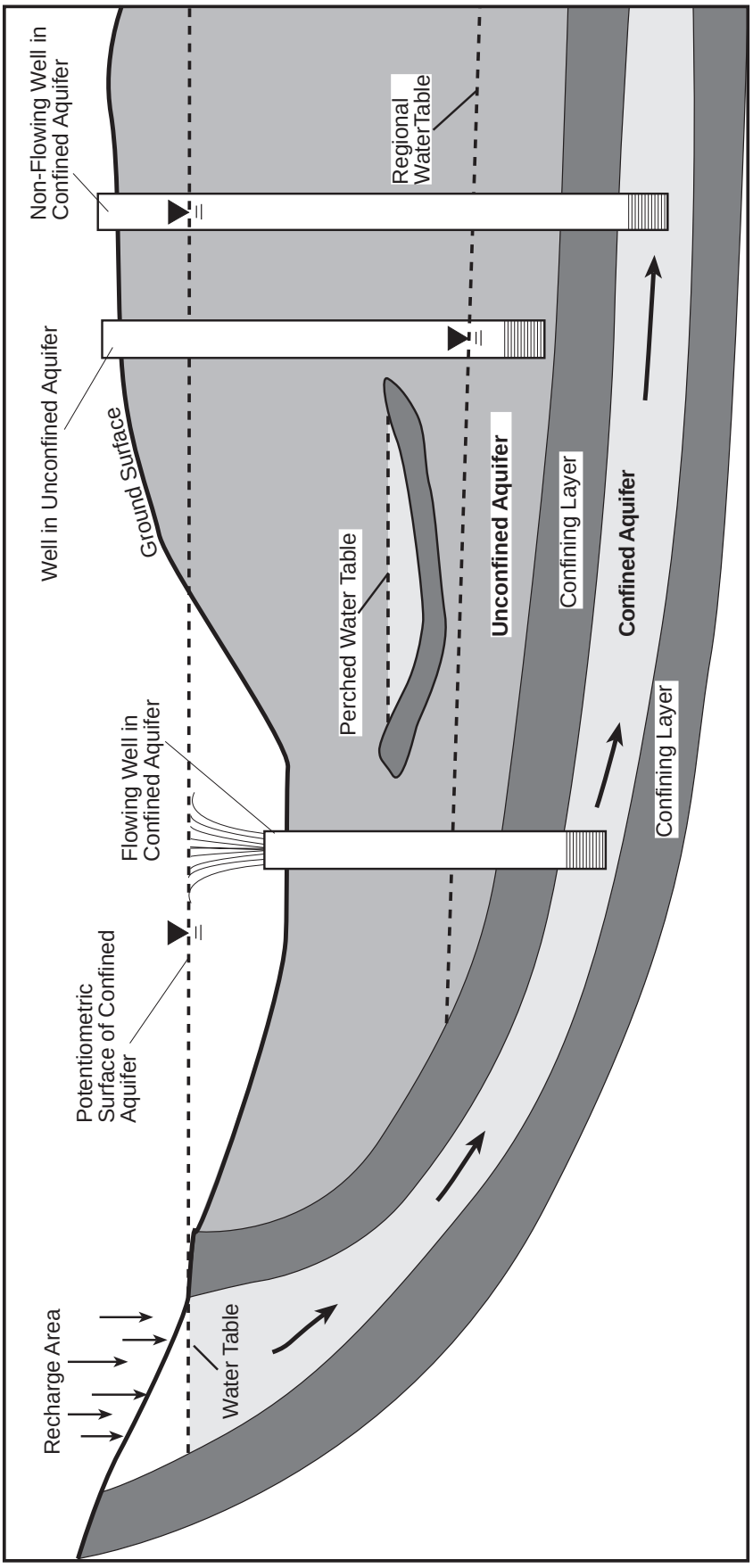


Figure I. Confined and unconfined aquifers
(After U. S. Department of the Interior, 1977)

an unconfined aquifer is in direct contact with the atmosphere through open **pores** of the material above and is everywhere in balance with the hydraulic head in the **recharge** area. Therefore, the static level in a well penetrating an unconfined aquifer will be the same as the level of the water table. Confined aquifers are aquifers that are overlain by a confining bed (see Figure I also). The confining bed has a significantly lower **permeability** than the aquifer. When a confined aquifer is penetrated by a well, the water will rise above the confining unit to an elevation that is equivalent to the hydraulic head of the confined aquifer. If this elevation is greater than the top of the well, the water will flow from the well (commonly called an artesian well). The term artesian well, however, includes any well developed in a confined aquifer where the water level rises above the top of the aquifer, not just those that are flowing.

Wells developed in confined aquifers can present a special problem when it comes time to seal them. Also, wells screened across several aquifers will require more care in sealing. These wells must be sealed in such a manner so that there is no intermixing of water between the aquifers. In addition, careful consideration must be given to choosing an appropriate sealing method for wells screened in a single aquifer but penetrating several aquifers. These scenarios and others will be addressed in the section on **Procedures For Sealing The Well**.

Preparation for Sealing

Well Information

Information concerning the geology and physical condition of the well, such as total depth, formations encountered, and diameter are important in determining the sealing method. Geologic conditions vary throughout the state and different methods of sealing are needed to meet these varying conditions. Well construction details are needed to determine the type and amount of materials needed to seal the well.

The best source of information is the "Well Log and Drilling Report" that is completed by the driller at the time of construction. These logs contain well construction information and a record of formations encountered during well installation. Figure J is an example of a well log. An accurate well log will enable a drilling contractor to select the most appropriate sealing method for that well. Copies of these reports are filed with the Ohio Department of Natural Resources, Division of Water, and, within the last 10 to 15 years, each county health department. To obtain a copy of a well log and drilling report, it is necessary to know the county in which the well was drilled, the township within that county, the street address, the name of the property owner at the time the well was drilled, the names of the nearest cross roads, and the approximate year in which the well was drilled. It may not be possible to discover all of this information, but the Division of Water will do a file search with the information that is available. Occasionally, a well log and drilling report may not be on file, either because the well was drilled before the filing law went into effect (1947) or because, for some reason, the log was not sent to the Division. Call the Division of Water, Water Resources Section at 614-265-6740 to have the files searched for a specific well log and drilling report.

Logs for nearby wells should be reviewed if a well log and drilling report cannot be located. Often, wells on adjacent properties will be of similar depths and construction. If a well is free of obstructions (including old pumps), then the depth may be easily determined with a weighted measuring tape or rope. Local drilling contractors will also be familiar with the general geologic conditions in the area.

Other methods for examining the condition of a well include casing-depth indicators, borehole video cameras, and geophysical logging equipment such as calipers and gamma-ray probes. These tools are commonly used in the maintenance of public supply wells and in scientific investigations. These methods are likely to be expensive, but most wells will not need such detailed investigations. Local drilling contractors should be able to locate firms possessing this equipment, if necessary.

Casing Issues

Well casing is usually constructed of plastic (PVC), steel (in drilled wells), or concrete pipe, vitrified tile, brick, or cobbles (in dug wells). The well screen is a section of wire-wrapped or machine-slotted casing through which water enters the well. In many older wells, it was common

DNR 7802.94
TYPE OR USE PEN
SELF TRANSCRIBING
PRESS HARD

WELL LOG AND DRILLING REPORT

Ohio Department of Natural Resources
Divison of Water, 1939 Fountain Square Drive
Columbus, Ohio 43224 Phone (614) 265-6739

Permit Number WI9500009

COUNTY FRANKLIN TOWNSHIP WASHINGTON SECTION/LOT No. 3 (Circle One)

OWNER/BUILDER (Circle One or Both) R. D. RUNNER (First Last) PROPERTY ADDRESS (Address of well location) 12345 RINGS ROAD (Number Street) DUBLIN (City)

LOCATION OF PROPERTY 1/8 MILE EAST OF COSGRAY, NORTH SIDE OF RINGS ROAD 43002 (Zip Code + 4)

CONSTRUCTION DETAILS

CASING *(Length below grade) Borehole Diameter 9 in.

[1] Diameter 5 in. Length* 157 ft. Wall Thickness SDR21 in.

[2] Diameter ____ in. Length* ____ ft. Wall Thickness ____ in.

Type: [1] Steel [1] Galv. [X] PVC [1] [2] Other ____

Joints: [1] Threaded [1] Welded [X] Solvent [1] [2] Other ____

Liner: Length ____ Type ____ Wall Thickness ____ in.

SCREEN

Type (wire wrapped, louvered, etc.) MACHINE SLOTTED Material PVC

Length 5 ft. Diameter 5 in.

Set between 162 ft. and 157 ft. Slot 0.050

GROUT

Material BENSEAL/E-Z MUD Volume used 280 GALLONS

Method of installation 1" TREMIE TUBE

Depth: placed from 152 ft. to SURFACE ft.

GRAVEL PACK (Filter Pack)

Material #4 PARRY SAND Volume used 500 LBS

Method of installation GRAVITY

Depth: placed from 165 ft. to 152 ft.

Pitless Device [X] Adapter [ ] Preassembled unit

Use of Well RESIDENTIAL

[X] Rotary [ ] Cable [ ] Augered [ ] Driven [ ] Dug [ ] Other ____

Date of Completion 2/17/95

WELL LOG*

INDICATE DEPTH(S) AT WHICH WATER IS ENCOUNTERED.

Show color, texture, hardness, and formation: sandstone, shale, limestone, gravel, clay, sand, etc.	From	To
YELLOW BROWN CLAY	0	11
GRAY CLAY & GRAVEL	11	63.5
SAND & GRAVEL	63.5	65.5
GRAY SILTY CLAY	65.5	89
SAND WITH GRAVEL	89	92
GRAY CLAY & GRAVEL	92	144
SAND	144	150
SAND & GRAVEL	150	165

WELL TEST

[ ] Bailing [ ] Pumping* [X] Other AIR LIFT

Test rate 25 gpm Duration of test 3/4 hrs.

Drawdown 20 ft.

Measured from: [ ] top of casing [X] ground level [ ] Other ____

Static Level (depth to water) 30 ft. Date: 2/17/95

Quality (clear, cloudy, taste, odor) CLEAR

*(Attach a copy of the pumping test record, per section 1521.05, ORC)

PUMP

Type of pump SUBMERSIBLE Capacity 20 gpm

Pump set at 100 ft.

Pump installed by OTHERS

WELL LOCATION

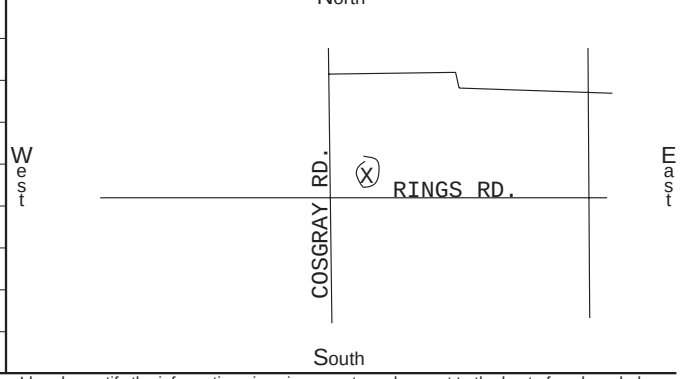
Location of well in State Plane coordinates, if available:

Zone ____ x ____ y ____

Elevation of well ____ ft./m. Datum plain: [ ] NAD27 [ ] NAD83

Source of coordinates: [ ] GPS [ ] Survey [ ] Other ____

Sketch a map showing distance well lies from numbered state highways, street intersections, county roads, buildings or other notable landmarks.



*(If additional space is needed to complete well log, use next consecutively numbered form.)

I hereby certify the information given is accurate and correct to the best of my knowledge.

Drilling Firm ACME DRILLING COMPANY Signed W. E. Coyote

Address 1234 MAIN ST. Date 2/27/95

City, State, Zip SOCKERDOWNE, OH 56789 ODH Registration Number 3456

Completion of this form is required by section 1521.05, Ohio Revised Code - file within 30 days after completion of drilling.

ORIGINAL COPY TO - ODNR, DIVISION OF WATER, 1939 FOUNTAIN SQ. DRIVE, COLS., OHIO 43224

Blue - Customer's copy Pink - Driller's copy Green - Local Health Dept. copy

Figure J. Example well log and drilling report

practice to cut slots with a torch in the bottom two or three feet of casing to produce a home-made screen. These types of "screens", however, are highly inefficient and susceptible to corrosion and plugging. The casing and screen prevent the surrounding formations from caving into the well. Some formations are sufficiently consolidated that the well will remain open and no well screen is required.

Water can migrate along the space between the casing and the borehole wall; therefore, the best way to seal a well is to remove the casing and screen (if any), and grout the open hole. If there is no well log or other information about the construction of the well, or if the well casing is in poor condition, then it is probably best not to remove the casing (unless the well is an environmental well that can be easily overdrilled). Consideration should be given to having a registered drilling contractor rip or perforate the casing in such cases.

Sealing Materials

The data in this section relies heavily on information found in the Michigan Water Well Grouting Manual (Gaber and Fisher, 1988).

Materials used for sealing abandoned water wells must have certain properties to make them desirable for use. The ideal grout should 1) be of low permeability in order to resist flow through them, 2) be capable of bonding to both the well casing (if necessary) and borehole wall to provide a tight seal, 3) be chemically inert or nonreactive with formation materials or constituents of the ground water with which the grout may come in contact, 4) be easily mixed, 5) be of a consistency that will allow the grout to be pumped and remain in a pumpable state for an adequate period of time, 6) be capable of placement into the well through a 1-inch diameter pipe, 7) be self-leveling in the well, 8) have minimal penetration into permeable zones, 9) be capable of being easily cleaned from mixing and pumping equipment, 10) be readily available at a reasonable cost, and 11) be safe to handle.

Grouting materials currently used in water wells are comprised of either cement or bentonite. Although there are advantages and disadvantages with each material and none of the grout materials available today exhibit all of the desirable characteristics listed above, field experience has shown each to be suitable under most geological conditions. Table 1 lists advantages and disadvantages of cement and bentonite grouts.

Final permeability of the grout is recommended to be 1×10^{-7} centimeters per second to retard fluid movement. Table 2 shows approximate permeability values for various sealing materials.

Cement–Based Grouts

Cement Properties

Portland cement is the main ingredient in cement-based grouts such as neat cement or concrete. Cement is a mixture of lime, iron, silica, alumina, and magnesia components. The raw materials are combined and heated to produce cement clinker. The clinker is ground up and mixed with a small amount of gypsum or anhydride to control setting time.

When Portland cement is mixed with water (producing neat cement), several chemical reactions occur. Heat is generated as the mixture cures and changes from a slurry to a solid. This is referred to as the heat of hydration and results in a temperature increase in the formation material at the cement/borehole interface and the well casing, if any remains in the hole (Troxell, et.al., 1968; Portland Cement Association, 1979). The amount of heat given off is dependent upon several factors such as cement composition, use of additives, and surrounding temperatures. Excessive heat of hydration may adversely affect the structural properties of any PVC plastic well casing left in the borehole (Molz and Kurt, 1979; Johnson et.al., 1980).

The setting of cement is controlled by temperature, pressure, water loss, water quality, and other factors (Smith, 1976). Warm water used for slurry preparation and warmer air temperature will cause faster setting than cold water and cooler air temperature. Cement in the borehole will tend to set faster at the bottom since the weight of the cement column will increase hydrostatic pressure on the cement at the bottom. Water expelled from the cement into permeable zones will also result in an increased rate of setting. Standard Portland cement will reach its initial set in about 4 hours at a 50°F curing temperature. Table 3 shows total curing times for various cement grouts.

Table 1
Grout Properties

	Advantages	Disadvantages
CEMENT-BASED GROUTS	Suitable Permeability	Shrinkage & Settling
	Easily Mixed & Pumped	Long Curing Time
	Hard-Positive Seal	High Density Results in Loss To Formations
	Supports Casing	Heat of Hydration
	Suitable For Most Formations	Affects Water Quality
	Proven Effective Over Decades Of Field Use	Equipment Clean-Up Essential
	Properties Can Be Altered With Additives	Casing Cannot Be Moved After Grouting
BENTONITE-BASED GROUTS	Suitable Permeability With High Solids Grouts	Premature Swelling And High Viscosity Result in Difficult Pumping
	Non-Shrinking & Self-Healing	Difficult Mixing
	No Heat of Hydration	Subject to Wash Out in Fractured Bedrock
	Low Density	Subject to Failure From Contaminated Water
	No Curing Time Required	Equipment Clean-Up Difficult
	Casing Movable After Grouting	Limited Field Experience
		Usage Instructions Vary For Each Product
		Limited Availability

Table 2
Permeability of Various Sealing Materials

Sealing Material	Permeability (K) in cm/sec
Neat Cement (6 gal water/94 lb sack)	10^{-7}
Bentonite Grout (20% Bentonite)	10^{-8}
Bentonite Pellets	10^{-8}
Granular Bentonite	10^{-7}
Granular Bentonite/Polymer Slurry (15% Bentonite)	10^{-8}
Coarse Grade Bentonite	10^{-8}
(From American Colloid Co, and N.L. Baroid/N.L. Industries, 1989)	

Table 3
Cement Curing Time Required

Grout Type	Curing Time
Neat Cement - Type I	48 Hours
Concrete Grout - Type I	48 Hours
Neat Cement w/2% $CaCl_2$	24 Hours
Hi-Early Cement - Type III	12 Hours
Concrete Grout - Type III	12 Hours

Cement Types

Several types of cement are manufactured to accommodate various chemical and physical conditions which may be encountered. The American Society for Testing and Materials (ASTM) Specifications C150 (ASTM, 1992) is the standard used by cement manufacturers.

Portland cement Types I and IA are readily available throughout Ohio. Type II cement is available at some of the larger building supply outlets. Other cements are available by special order through cement suppliers. The different types of cement and their appropriate usage are described as follows:

Type I — General purpose cement suitable where special properties are not required.

Type II — Moderate sulfate resistance. Lower heat of hydration than Type I. Recommended for use where sulfate levels in ground water are between 150 and 1500 ppm.

Type III — High-early-strength. Ground to finer particle size which increases surface area and provides faster curing rate (approximately $^1/_4$ of the time it takes for Type I to cure). When Type III cement is used, the water to cement ratio must be increased to 6.3 to 7 gallons of water per sack.

Type IV — Low heat of hydration cement designed for applications where the rate and amount of heat generated by the cement must be kept to a minimum. Develops strength at a slower rate than Type I.

Type V — Sulfate-resistant cement for use where ground water has a high sulfate content. Recommended for use where sulfate levels in ground water exceed 1500 ppm.

Expansive-type cements are also available in Ohio. This type of cement will expand upon curing by use of additives in the mix, such as gypsum or aluminum powder.

Neat Cement Grout

Neat cement slurry is comprised of Portland cement and fresh water, with no aggregate present. It was first used as a grouting material in Texas and Oklahoma oil fields in the early 1900's (Smith 1976). Neat cement has since been used extensively in both the oil & gas and water well industries. Field experience has shown it to be effective for sealing off formations when properly applied. It can be mixed using a wide variety of methods. Generally, lower pressures are developed while pumping neat cement grouts. The main disadvantages of using neat cement are shrinkage upon curing, possible formation of a **microannulus** around the casing, and, in some cases, mixing according to manufacturer's specifications, which can result in a thick mixture that is difficult to pump.

In some states, neat cement is considered superior to bentonite-based grouts in situations where bedrock is encountered within 25 feet of ground surface. This is because it will form a hard, rock-like seal consistent with the bedrock and will not wash out or dilute from higher ground water flow rates encountered in some highly fractured formations.

The amount of shrinkage or settling, and compressive strength, of neat cement is dependent upon the proportion of water to cement in the slurry (Coleman and Corrigan, 1941; Halliburton Services, 1981). As the water to cement ratio increases, the compressive strength of the neat cement will decrease and shrinkage will increase. Laboratory studies and field experience have demonstrated that settling of cement particles will occur, resulting in a drop in the grout level (Coleman and Corrigan, 1941, Kurt, 1983). The top of the hardened neat cement grout mass will generally be a few feet below the slurry level due to this settling. Field observations show that the amount of settling will usually be 5 to 10 percent of the total grouted depth if the neat cement is mixed at 5 to 6 gallons of water per sack.

The American Petroleum Institute (API) recommends a water to cement ratio of 0.46 by weight or 5.2 gallons of water per 94-lb sack of cement. This is the amount of water needed to hydrate the cement. More than 5.2 gallons/sack ratio will thin the grout and make it easier to pump, but will adversely affect the grout's sealing properties. This guideline recommends that the maximum amount of water mixed per sack of cement be 6 gallons. The neat cement slurry at 6 gallons of water per sack of cement should weigh a minimum of 15 lbs/gal before pumping. At weights greater than 16 lbs/gal, pumping of the slurry becomes difficult due to higher **viscosity** and pumping pressure. **Density** measurements of the slurry using a **mud balance** are recommended to assure proper water-to-cement ratios.

Under certain conditions it may be necessary for the consulting engineer or regulatory agency to specify an increase in the water to cement ratio. Factors such as the cement type, addition of additives, and quality of ground water will affect the grout performance and should be considered when planning the grouting operation.

Concrete Grout

Concrete grout consists of Portland cement, sand, and water. The addition of sand to a neat cement slurry results in less shrinkage and tighter bonding to the casing and borehole. Also, the sand in the slurry will aid in bridging pores in permeable formations. Concrete grout should be used only under specific sealing circumstances, such as for sealing flowing wells, sealing water wells with natural gas or methane present, and sealing wells with cavernous zones. Concrete should be handled only by experienced registered drilling contractors due to the exacting requirements for its successful installation. Concrete grout must be pumped down a tremie pipe, or, if the borehole is free of water, poured down. Placing concrete grout through a column of water will cause the separation of sand from the slurry and result in placement problems. If concrete grout is used on a routine basis, it should be pumped through a metallic grout pipe because it is highly abrasive on plastic pipe. Concrete grout can also cause excessive pump wear.

Other Cement Additives

Accelerators may be added to cement to decrease its setting time when attempting to cement off flows in and around casings. This will allow the cement to set before it is washed out of the hole. Calcium chloride is the most common and readily available accelerator. It is generally used at between 2 and 4 percent by weight of cement. Accelerators should be used with caution since miscalculations or equipment breakdown can result in a cemented grout pump or hose. Other additives such as retarders, weight-reducing agents, weighting agents, lost circulation control agents, and water reducing agents are available for cements but are not routinely used for water well sealing.

Bentonite—Based Grouts

Clay Properties

Clays are the principal ingredient of all bentonite-based grouts and drilling muds. They may be characterized as naturally occurring substances which exhibit colloidal-like properties (remain in suspension in water for a long period of time) and varying degrees of **plasticity** when wet (Bates, 1969). The term clay is frequently applied to a variety of fine-grained materials including clays, shales, and clayey soils. They are all composed of small crystalline particles which are known as the clay minerals.

The common characteristic associated with clays is the very small particle size that has a very high surface area to mass ratio. Negative electrical charges on the particle surface result in the interaction of clays with other particles and water. This, coupled with the ability of certain clays to swell many times their original volume when **hydrated**, accounts for many of the properties and uses for clays.

The variety of bentonite commonly used in grouting materials and drilling muds is one in which the clay mineral is predominantly sodium-rich montmorillonite. Mined at relatively few locations, the majority of the high-grade sodium bentonite is obtained in Wyoming, Montana, and South Dakota (Gray and Darley, 1981). These clays are characterized by their ability to absorb large quantities of water and swell 10 to 12 times in volume. Bentonite particles tend to remain in suspension an indefinite period of time when placed in water. The resulting slurry is of low density and high viscosity. Bentonites that have calcium as the predominant exchangeable ion are less desirable as sealing materials because they have significantly lower swelling ability (Gaber and Fisher, 1988). That is why mixing cement and bentonite is ineffective for preventing shrinkage of cement as it cures. Calcium ions in the cement replace sodium ions in the bentonite by a process called ion exchange. The resulting calcium bentonite has little or no swelling capability, and is therefore unable to prevent shrinkage of the cement (Smith, 1994).

Properties of Bentonite/Water Slurries

Three important physical properties of a water/bentonite slurry are: 1) density, 2) viscosity, and 3) gel strength. A review of these properties will aid in understanding what makes a good bentonite grout.

Density is defined as the weight per unit volume of a fluid and is commonly expressed in pounds per gallon. The terms weight and density, although technically distinct, are frequently used interchangeably in the drilling industry. The density of grout determines how much pressure is exerted on the formation when the fluid is at rest and is a direct indicator of the amount of clay solids present. The higher the density, the more solids are suspended in solution. Density is measured using a mud balance. A mud balance measures a specific volume of grout slurry in pounds per gallon. The densities of various sealing materials can be seen in Table 4.

Measurements should be taken after each grout batch is mixed and a grout sample should also be collected after the grout appears at the surface. The grout discharged from the well should have a density equal to that of the grout before it was pumped. The grout must be pumped into the well until dilution is minimal.

Viscosity is a measure of a fluid's resistance to flow. The higher the viscosity of a fluid, the more difficult it becomes to pump. The viscosity of bentonite-based grouts is dependent upon a number of factors including: 1) the density, 2) the size and shape of the clay particles, and 3) the charge interaction between the particles (Driscoll, 1986). Viscosity can be measured with a Marsh funnel viscometer, which determines the time it takes to dispense one quart of fluid through the funnel. Water has a Marsh funnel viscosity of approximately 26 seconds; bentonite-based grouts should have a 70 second viscosity. Grout should be periodically checked for adequate viscosity. A low viscosity grout will make a less effective seal than a grout with the proper viscosity.

Gel strength is a measure of internal structural strength. It is an indication of a fluid's ability to support suspended particles when the fluid is at rest. Gel strength is caused by the physical alignment of positive and negative charges on the surface of the clay particles in solution. Gel strength is responsible for the quasi-solid (plastic) form of a clay/water mixture.

Table 4
Grout Slurry Densities

Product	Water Ratio	Minimum Density Lbs/Gal	Volume ft^3/sack
Neat Cement	6.0 gal./sack of cement	15.0	1.28*
	5.2 gal. recommended/sack of cement	15.6	1.18*
Neat Cement & CaC1 (accelerator)	6.0 gal./sack of cement CaC1- 2 to 4 lbs. sack of cement	15.0	1.28
Concrete Grout	1 sack of cement and an equal volume of sand per 6 gallon maximum water	17.5	2.0
Bentonite			
Benseal/EZ-Mud	Benseal - 1.5 pounds/gallon of water EZ-Mud - 1 quart/100 gallons of water	9.25	4.75
Volclay	2.1 pounds/gallon of water	9.4	3.6

(*From Halliburton Services, 1981)

The gel strength is affected by how well the clay particles are dispersed in solution and the amount of water the particles have absorbed. Gel strength is not typically measured in the field. However, it is related to the fluid density and is dependent largely on the quality of the bentonite.

High-Solids Bentonite Grout

Within the last decade, bentonite products developed specifically for well grouting have appeared on the market. Some use chemical additives when mixing to control the development of viscosity and gel strength. By design, these products are meant to be easy to pump, place, and clean up. Premature swelling and/or high viscosities may make them difficult to pump when they are not mixed properly. Generally, bentonite grouts require higher pumping pressures than neat cement grouts (Gaber and Fisher, 1988). It also is important to know the environment into which the bentonite will be placed.

For example, high concentrations of chlorides in the water will supress the hydration of bentonite unless it has been mixed with an agent that counteracts the effect of the chlorides (Smith, 1994).

The bentonite-based grouts currently available can be broadly grouped into four classifications. The classifications reflect the degree of processing and the particle size of the bentonite constituent. The four classes of materials are: 1) powdered bentonite, 2) granular bentonite, 3) coarse grade bentonite, and 4) pelletized bentonite. Each class of bentonite requires a different handling and placement method. Manufacturers recommend that mixing and placement methods should be assessed with regard to the depth to the water table, the required depth of grouting, and other pertinent geological information.

Powdered Bentonite/Clay Grout

Powdered bentonite/clay products available are similar in texture, appearance, and packaging to the high yield drilling mud grade bentonite. They are a mixture of bentonite clays (sodium and calcium) and other clays and do not possess the expansion characteristics of grouts containing predominantly sodium bentonite. They are marked as high solids clay grout with a resulting slurry of 15 to 20 percent clay solids by weight of water and are designed to have extended workability. When properly applied, they result in a flexible seal of low permeability. Adequate mixing of this product requires the use of a venturi-type mixer and a mud rotary type mud pump and recirculation system or a paddle mixer.

Some products utilize an inorganic chemical additive (magnesium oxide) referred to as an initiator, to aid in the development of gel strength. Exclusion of the initiator can result in decreased set strength, affecting the quality of the seal. Failure to meet manufacturer's density requirements or placement of the grout on top of a lower density material (e.g., drilling mud or water) can result in a disappearance of the grout material from the well. This is due to a lack of gel strength development, resulting in settling of bentonite material in the well or loss to surrounding formations. For this reason, the use of these products requires placement of the material the entire length of the borehole. A bentonite pellet or neat cement cap a few feet thick is also recommended near the surface.

Granular Bentonite Slurries

Granular bentonites are generally manufactured from high-yield, non-drilling grade bentonite. The bentonite is processed to provide coarse granular particles (predominantly 8 to 20 **mesh**) which possess considerably lower surface area-to-mass ratios than the finely ground, powdered bentonite. This results in slower water absorption and delayed hydration and expansion when compared to a finely ground bentonite.

One advantage of the granular bentonite slurry is that the delay in swelling of the bentonite particles for a short period of time (15 minutes or less) allows preparation of a slurry possessing a lower viscosity. If mixing and pumping are done efficiently, the granular bentonite slurries allow placement of a high density grout in a low viscosity state. Expansion of the bentonite then occurs downhole. Granular bentonite may be prepared with 15 to 20 percent bentonite content by weight. This results in a set grout which exhibits excellent permeability and gel strength characteristics.

These products rely on the addition of a synthetic organic polyacrylamide polymer to suppress the hydration and delay swelling of the bentonite particles. The use of such products requires particular attention to the manufacturer's mixing recommendations. One recommended mixing procedure requires addition of the polymer to water at a rate of 1 quart of polymer per 100 gallons of water prior to adding the granular bentonite at 1-$^1/_2$ to 2 lbs. per gallon (Smith and Mason, 1985). Mixing requires the use of blade or paddle-type mixers or grout mixers with recirculation. Centrifugal pumps are not recommended for mixing or pumping granular bentonite slurries. Upon addition of the bentonite, pumping of the grout material must be accomplished before swelling of the bentonite occurs. If expansion occurs prematurely the slurry cannot be pumped and the batch is wasted.

Coarse Grade Bentonite

Coarse grade bentonite, also referred to as crushed or chip bentonite, is processed by the manufacturer to provide a large particle size and density. The bentonite particles are sized from $^3/_8$ to $^3/_4$ inch

and are intended to fall without bridging through a column of water in a borehole. When placed properly, the coarse grade bentonite provides a high density, flexible down-hole seal of low permeability.

Due to the size of the coarse grade bentonites, care should be taken in their use. Since the material cannot be pumped, placement of the material requires pouring from the surface. Placement may be accompanied by tamping to insure that bridging has not occurred. The bentonite must be poured slowly, and the pouring rate should not exceed the manufacturer's specifications.

Prior to using this material, it should be sieved through 1/4-inch mesh screen to remove fines which have accumulated in the bag during shipment. These fines, if not removed, will clump if they hit water and increase chances of bridging. Water should be poured on top of any coarse grade bentonite above the water table to induce swelling.

Pelletized Bentonite

The pelletized bentonite consists of 1/4 to 1/2-inch size, compressed bentonite pellets. As with coarse grade bentonite, pelletized bentonite provides a dense and flexible seal. Pelletized bentonite can be poured directly into the well through standing water. Precautions similar to those for the use of coarse grade bentonite are required to avoid bridging.

Availability Of Bentonite Grout Materials

Bentonite products are not as widely available as Portland cement and must be obtained from water well equipment suppliers. New bentonite grouting products with higher solids and improved workability will most likely be developed by the bentonite industry in the future.

Fill Materials

Complete filling with a bentonite material may not be economically feasible in wells that have exceedingly large volumes. Most wells in this category are of large diameter and, therefore, present a physical hazard. This type of well must be filled with load-bearing materials (Gordon, 1988). For example, large diameter, shallow dug wells can be filled with clean clay, such as that sold in 100 pound bags as fireclay (Peck, 1987). This clay, however, should not be used as a sealing material or fill material in other types of wells because it does not seal as effectively as commercially prepared bentonite products (Carlton, 1975) and can bridge in deeper, smaller diameter wells. There are other well types - such as wells completed in fractured limestones or extremely coarse gravel - that can contribute to the excessive loss of sealing materials. Fill materials can be used in such instances to bridge fractures and large voids in the water-producing zone before sealants are emplaced (Gordon, 1988).

There are a variety of low-cost materials which can be used to fill space where an impermeable seal is not required in a certain section of the borehole, as specified in the next section of these guidelines. Depending upon the application, these materials may be clean, disinfected sand, gravel, crushed stone, or clay. In all cases, the material should be clean — free of sticks, leaves, or other foreign matter. Additionally, the material should be free of any toxic chemical residue. All fill materials considered for well sealing should be sized to the well being sealed, meaning that the material should have particle-size diameters small enough not to cause bridging (Gordon,1988).

Procedures for Sealing the Well

General Sealing Procedures

The first step in the well sealing process is to remove all obstructions from the well. These obstructions can include pumps and related equipment, such as drop pipes, **pitless adapters**, and suction lines. Pumps that are stuck and cannot be pulled should be pushed to the bottom of the hole, if possible. Other obstructions could consist of trash, animal remains, and debris such as large rocks or pieces of wood. If there is a possibility that the well has been contaminated, which could be evidenced by the presence of items such as empty pesticide containers, fertilizer bags, or a strong odor, the well owner should inform the appropriate agency (see Appendix 1) before sealing begins.

After the obstructions have been removed from the well, the next step is to decide what to do with the casing. As discussed earlier, the casing can be removed (by pulling, overdrilling, drilling out, or

jacking out), ripped or perforated, or left intact if the annular seal is judged to be adequate. The treatment of the casing will depend on the condition and type of casing, and the type of well being sealed. Another factor to consider when attempting casing removal is the type of formation in which the well is developed. Wells developed in unconsolidated formations should be sealed by simultaneously removing the casing and adding the sealing and /or fill material(s) to the well. This is necessary because once the casing is removed from a well developed in an unconsolidated formation, the borehole walls may collapse, preventing complete sealing of the borehole. It is highly recommended that this and all other steps in the sealing process be performed by an experienced registered drilling contractor.

Ripping or perforating a casing should be done by experienced registered drilling contractors that have the specialized tools needed for this work. Well casings should be ripped or perforated when the casing cannot be removed and:

- voids are known to exist between the casing and the formation, or
- there is a gravel pack (type of filter pack) between the two casings of a double-cased well, or
- the well is located in an area of known ground water contamination, or
- any combination of the above.

Overdrilling is not a commonly used method of casing removal in the water well industry; rather, it is used more often to remove casings from environmental wells. Overdrilling a well requires a drilling contractor to drill a larger diameter borehole over the existing well. The depth of the overdrilled borehole will depend on the construction of the well and local hydrogeologic conditions. After overdrilling, the casing can then be pulled. The borehole where the well was located is then grouted. Well casings should be overdrilled when the casing cannot be pulled, the condition of the annular seal is questionable, and:

- there is water flowing from around the outside of the well casing (this condition can occur in flowing wells), or
- the well is located in an area of known contamination, or
- there is gravel packing connecting two or more hydraulic zones.

Once the casing issue has been resolved, the next step is to disinfect the well. Chlorine is the most commonly used disinfection product, and is sold in many forms: bleach, tablets, pellets, powder, etc.. At concentrations of 150 to 200 parts per million (or milligrams per liter), the chlorine will kill any bacterial organisms that may exist in the water. As a result, water forced back into the formation by the sealing process will not cause bacterial contamination of any wells that are downgradient of the sealed well and developed in the same aquifer. The well may be disinfected before casing removal if it is suspected that the borehole may collapse after removal of the casing.

Installation of the sealing material follows the disinfection process. The method of installation and the sealing materials used must be matched to the type of well being sealed. In most situations (except for shallow wells), the method that provides the easiest installation and best seal is pressure grouting with a neat cement or bentonite slurry. Pressure grouting involves pumping a mixture of neat cement and water, or bentonite, polymer or initiator, and water down a small diameter plastic or steel pipe (called a tremie pipe) which has been lowered to the bottom of the borehole or well (Figure K). As the slurry is pumped, the pipe is pulled back at a rate that keeps the end of the pipe submerged in the slurry. The slurry will displace any water in the well and force it to the surface. The well has been filled when the slurry reaches the surface. Time should be allowed for any settlement of the slurry to occur before the final steps in the well sealing procedure are followed.

Precautions must be taken to ensure that the sealing material does not bridge when sealing wells with coarse grade bentonite products. Chipped bentonite products can be used in wells 200 feet deep or less. Pelletized bentonite products can be used in wells 100 feet deep or less. All coarse grade products used in wells less than 24" in diameter should be poured over a wire mesh screen to eliminate the fine bentonite powder that could cause bridging. These products should be poured at a rate no faster than 3 minutes per 50 pound bag (Figure L). The pouring process should be halted occasionally in order to lower a weighted measuring tape into the well until it reaches the top of the sealing products to confirm that bridging has not occurred. A tamping device should be used where possible to break any bridges that form. The total volume of products used to fill the well should be very close to

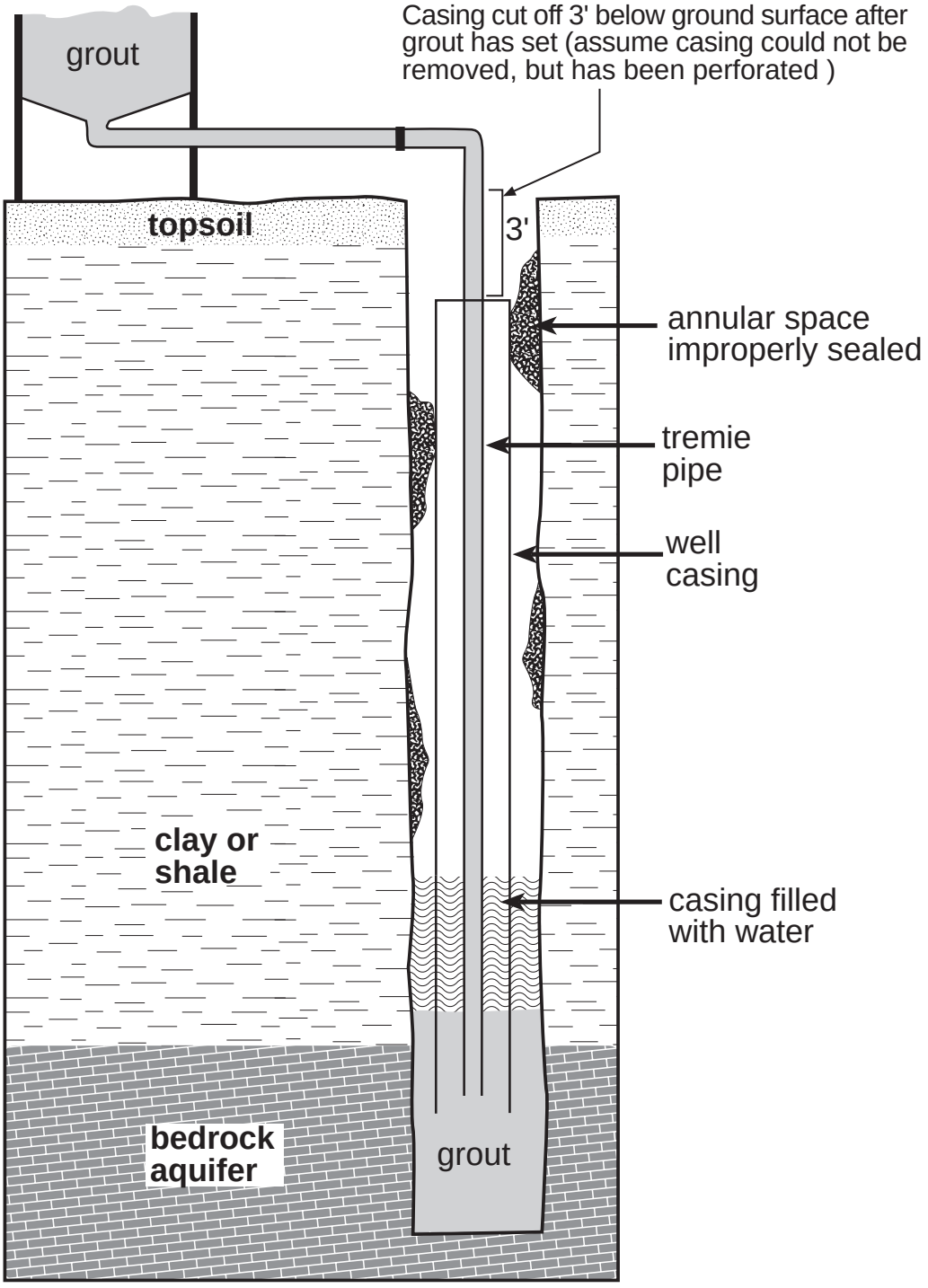


Figure K. Grouting bedrock well using tremie pipe method

the estimate of the amount needed for sealing. After the well has been sealed, it should be left unfinished overnight to see if any settling has occurred.

When the installation of sealing material has been completed, any remaining casing must be cut off to a depth of three to four feet below ground level. Then the remaining hole should be filled with clean soil and mounded at ground surface to ensure that surface water will drain away from the well. This procedure may be subject to change, depending upon the use of the land where the well is located.

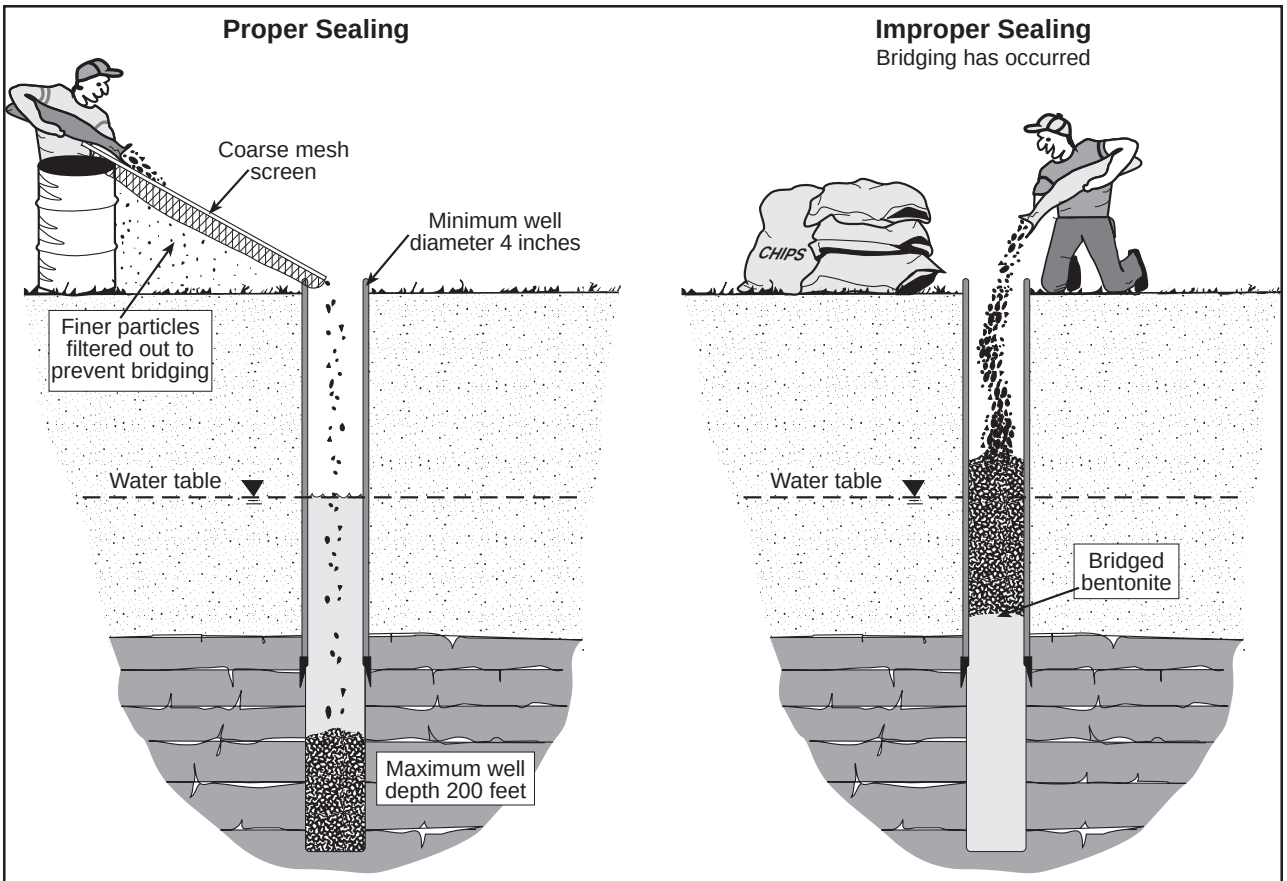


Figure L. Sealing wells with coarse grade bentonite products using pouring method
(Modified from Wisconsin Department of Natural Resources, 1993)

The final step in the well sealing process is the submission of a well sealing report to the Ohio Department of Natural Resources, Division of Water (Figure A). This form is readily available from the Division of Water upon request, and most drilling contractors will have a supply on hand.

Specific Well Sealing Procedures

Sealing Dug and Bucket-Drilled Wells

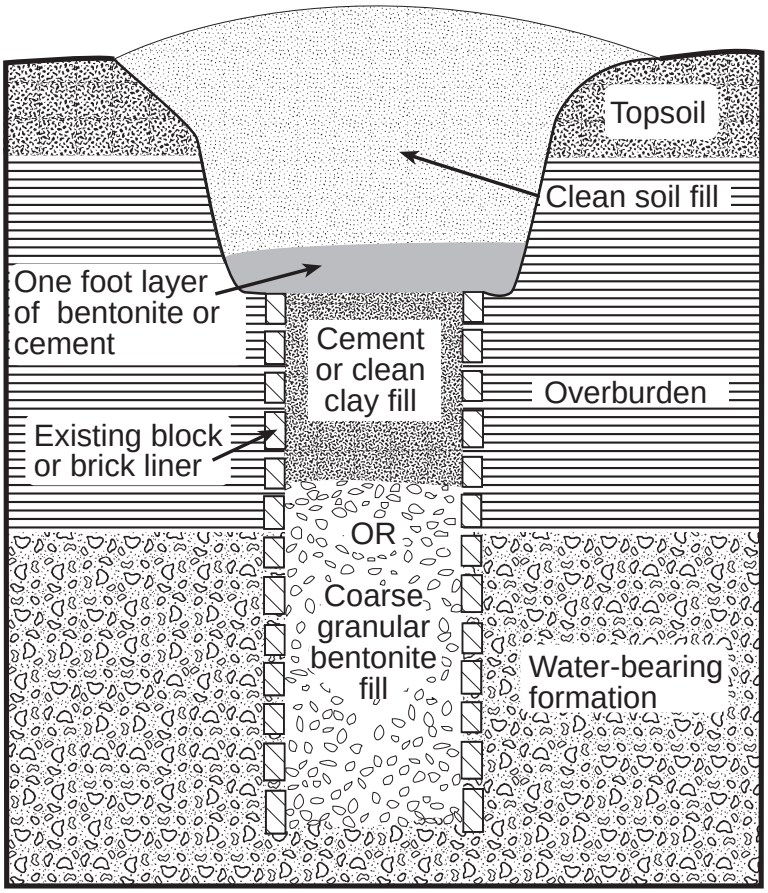
The procedure for sealing dug and bucket-drilled wells differs somewhat from the general procedures described earlier in this guidance (Figure M). Once any obstructions are removed, the liner (casing equivalent) should be left intact except for the upper 3 to 5 feet (this number is dependent upon the measured static level in the well). The static water level in the well should be measured, and the well pumped dry if it contains water. The well should be disinfected, wet or dry.

Backfill with clean clay or cement to the measured water level if the static water level is less than five feet below ground surface, and the well is greater than twenty-four inches in diameter and twenty-five feet or less in depth. Remove the liner to the depth of the measured water level and excavate 6 inches beyond the original outside diameter of the well. Add a one foot thick layer of impermeable sealing material at the depth of the measured water level. This layer could consist of coarse grade bentonite or cement. If the impermeable sealing material used is coarse grade bentonite, it should be hydrated before completion if the well has been pumped dry. Five gallons of potable water should be added per fifty pound bag of bentonite. Fill the remainder of the well with clean soil and mound at ground surface to ensure drainage of surface water away from the well.

Backfill with clean clay or cement to the measured water level if the static water level is greater than five feet below the ground surface, and the well is greater than twenty-four inches in diameter and twenty-five feet or less in depth. Add a one foot thick layer of impermeable sealing material and

hydrate. Then add more clean clay or cement to three feet below the ground surface. Remove the top three feet of liner and excavate six inches beyond the original outside diameter of the well. Add another one foot of sealing material and hydrate it if necessary. Fill the remainder of the well with clean soil and mound at ground surface to facilitate drainage of surface water.

Wells less than or equal to twenty-four inches in diameter with water levels greater than five feet below ground surface, or, wells greater than twenty-four inches in diameter and greater than twenty five-feet in depth, should be treated as drilled wells and sealed in a manner appropriate for drilled wells.



(Well is 25 feet deep or less and greater than 24 inches in diameter, and has a static level of less than five feet.)

Figure M. Sealing procedures for dug or shallow, large diameter wells

Sealing Driven Wells

After inspection and removal of any obstructions, the well should be disinfected. Casing removal should then be attempted. The well should then be filled with coarse grade or pelletized bentonite, bentonite slurry, or cement slurry to ground surface. The casing should be cut off three feet below the ground surface if it was not successfully removed. The area excavated around the well to access the casing should be filled with clean soil and mounded at ground surface to ensure drainage of surface water away from the well.

Sealing Wells Drilled Through a Single Unconsolidated Aquifer or an Unconfined, Unconsolidated Aquifer

After any existing obstructions have been removed from the well, the casing should be removed, ripped or perforated, or left intact. The type and condition of the casing will determine which procedure should be attempted. Before installing the sealing materials, the well should be disinfected. Then, clean sand and/or gravel can be placed from the bottom of the well to the top of the aquifer, or to twenty-five feet below ground surface, whichever is encountered first. Next, the well should be pressure grouted with bentonite or cement slurry, or coarse grade bentonite products can be slowly poured, from twenty-five feet to ground surface. Hydrate any coarse grade or pelletized bentonite products used, if necessary. Periodically tamp the coarse grade bentonite products to prevent bridging. These filling and sealing procedures should be done as the casing is being removed from the hole, if removal is to be attempted. If the well is not of great depth or large diameter, an alternative sealing method would be to pressure grout the entire well bore from bottom to ground surface with bentonite or cement slurry as the casing is being removed from the hole.

The casing should be cut off three feet below ground surface if it was not removed while sealing. The remaining excavation should be filled with clean soil and mounded at ground surface.

Sealing Wells Drilled Through Multiple Unconsolidated Aquifers

Remove any obstructions present in the well. After the well has been disinfected, simultaneously install the sealing materials and remove the casing, if it is to be removed. The well should be pressure grouted from the bottom of the well to ground surface with bentonite or cement slurry if the well penetrates multiple aquifers but is open only to one, or penetrates multiple aquifers and is screened across multiple zones. Or, if there is detailed information available on the depth and thickness of each aquifer penetrated, it may be possible to place clean sand and/or gravel within each aquifer zone, and place an impermeable sealing material, such as neat cement or bentonite products, between each aquifer corresponding to the confining unit present (Figure N). Then the well should be sealed from the top of the uppermost aquifer to the surface with neat cement or bentonite products. Coarse grade bentonite products (chips) can be slowly poured into the well until they appear at ground surface if the well is 200 feet deep or less, and is 4 inches or more in diameter (Figure N). Pelletized bentonite products can be used if the well is 100 feet deep or less, and is 4 inches or more in diameter. These bentonite products, when used, should be periodically hydrated, if necessary, and periodically tamped to prevent bridging.

Any casing not removed should be cut off three feet below ground level, and the remainder of the well can be filled with clean soil and mounded at ground surface.

Sealing Wells Drilled Through Confined, Unconsolidated Aquifers

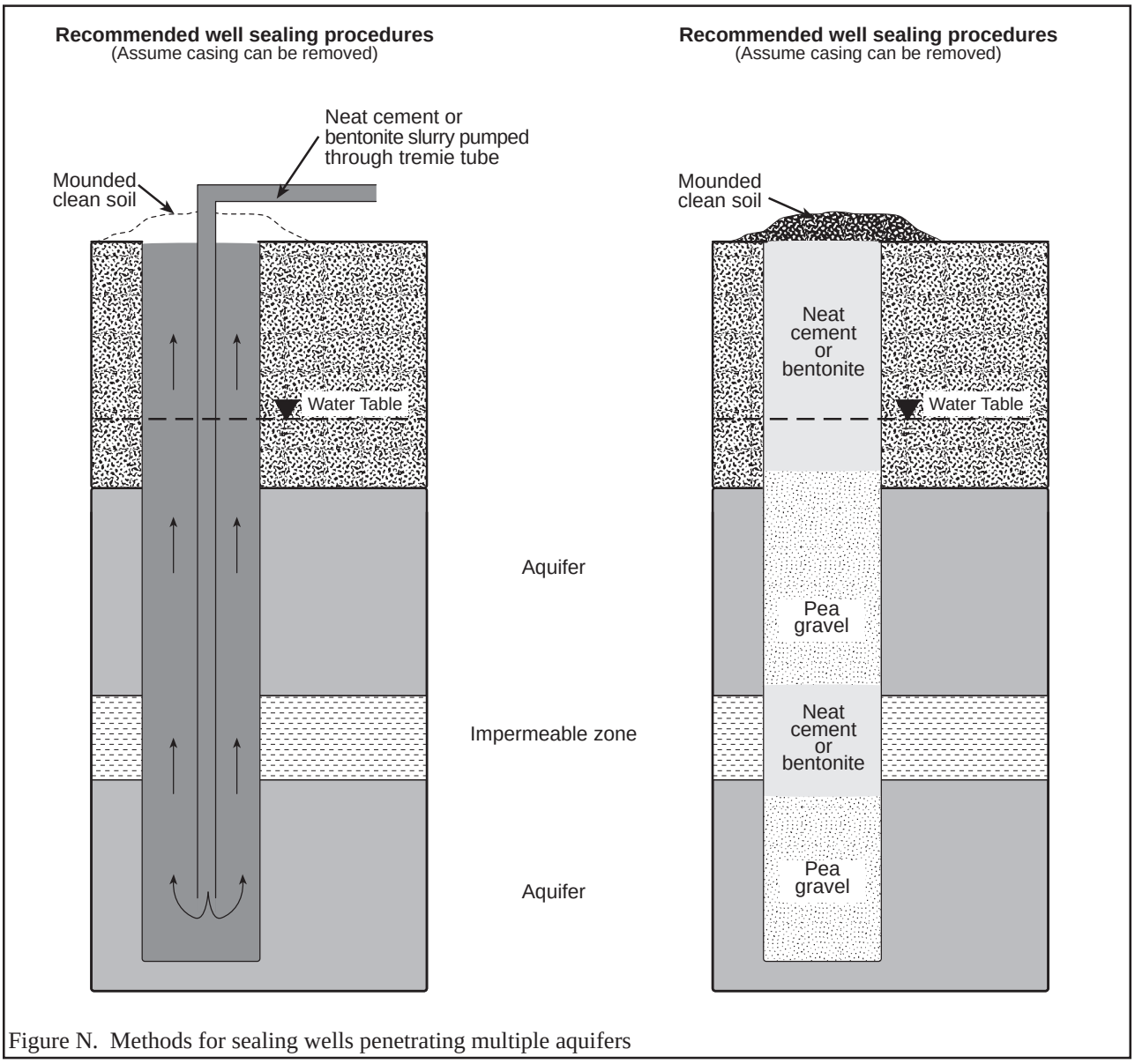


Figure N. Methods for sealing wells penetrating multiple aquifers

Wells drilled through confined aquifers can be difficult to seal because they can flow. This guidance strongly recommends that an experienced registered drilling contractor be consulted in all sealing situations, but it is especially important when dealing with these types of wells.

Once any obstructions are cleared, the casing must be removed, ripped or perforated, or left intact, depending upon the condition of the casing and the proposed sealing method. The well should then be disinfected, if possible, before the sealing materials are installed. If the well is not flowing, clean sand and/or gravel may be placed from the bottom of the well to the top of the producing zone or to twenty-five feet below the ground surface, whichever is encountered first. This should be followed by pressure grouting with cement or bentonite slurry, or by slowly pouring in coarse grade bentonite products or pelletized bentonite, from twenty-five feet to ground surface. Any coarse grade bentonite products used should be periodically hydrated, if necessary, and tamped to prevent bridging.

If the well is flowing from within the casing only, an attempt should be made to determine the hydraulic head. Extend the casing high enough to keep the well from flowing if the hydraulic head is low enough to permit casing extension (Figure O(1)). Pressure grout with cement from the bottom of the well to the ground surface. Coarse grade bentonite products (100 feet or less for pelletized bentonite, 200 feet or less for chipped) may be poured from the bottom of the well to ground surface if the

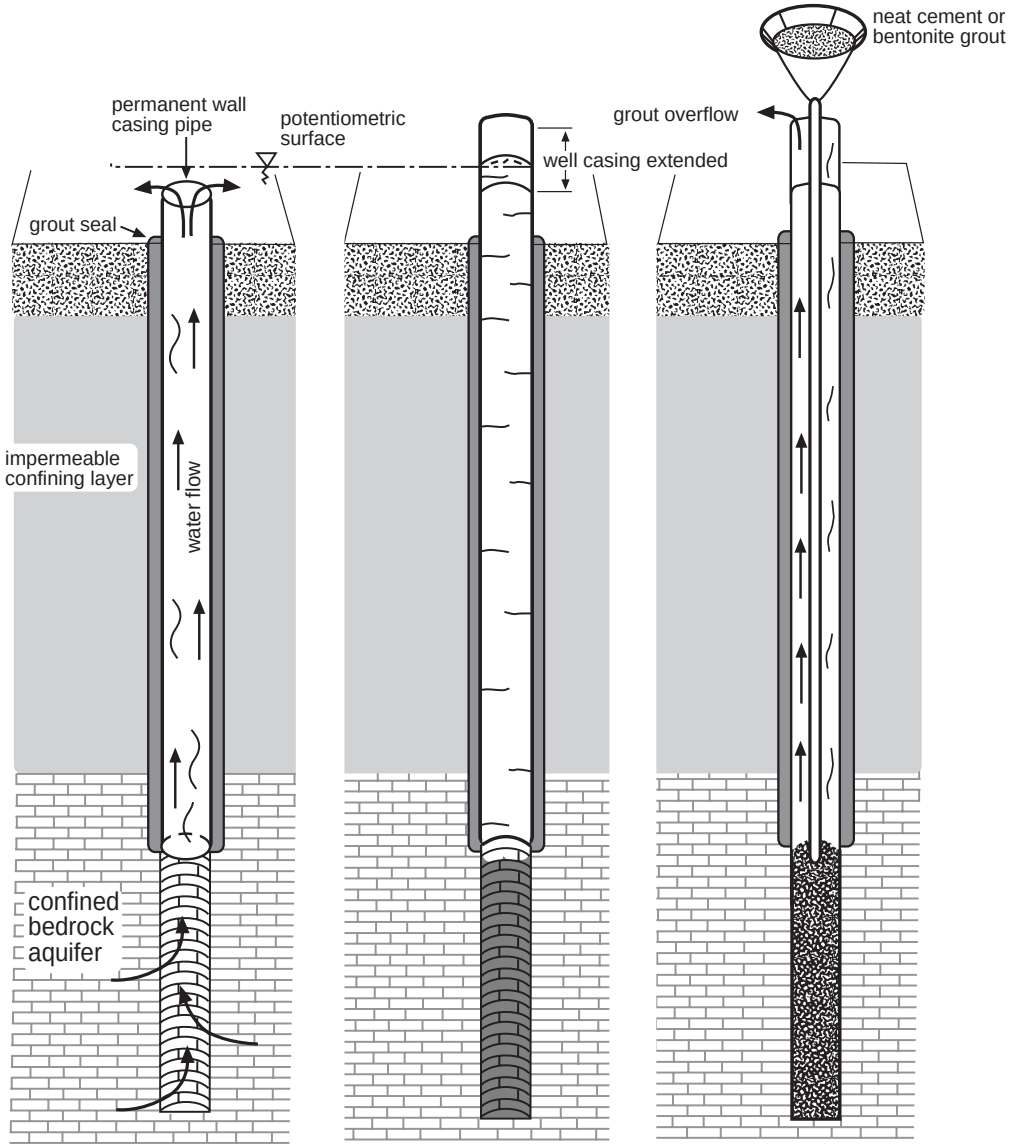


Figure O(1). Reducing or stopping flow of well by casing extension
(After Wisconsin regulations, 1994)

flow has been completely stopped by casing extension. The casing can then be cut off three to four feet below ground surface, and the remaining excavation filled with clean soil and mounded.

The well can be sealed by attempting to stop the flow, or by other methods that will allow sealing while the well is flowing, if the hydraulic head is too high to permit casing extension. An inflatable packer should be placed at the top of the producing formation to attempt to stop or restrict the flow (Figure O(2)). Then the well can be pressure grouted with cement or bentonite slurry through the packer from the bottom of the hole to the bottom of the packer. The packer can then be deflated and grouting continued to ground surface. It may also be possible to stop the flow by placing a shut-in device on top of the well. A tremie tube can be inserted through the shut-in device and the well pressure grouted with cement or bentonite slurry from the bottom of the well to the ground surface.

Another alternative for slowing the flow is to pour disinfected gravel into the well in an attempt to reduce the flow to a point where it becomes possible to use pressure grouting equipment to seal the well (Figure O(3)). Once the grout has set, any remaining casing can be cut off three to four feet below the ground surface. The resulting hole can then be filled with clean soil and mounded at ground surface.

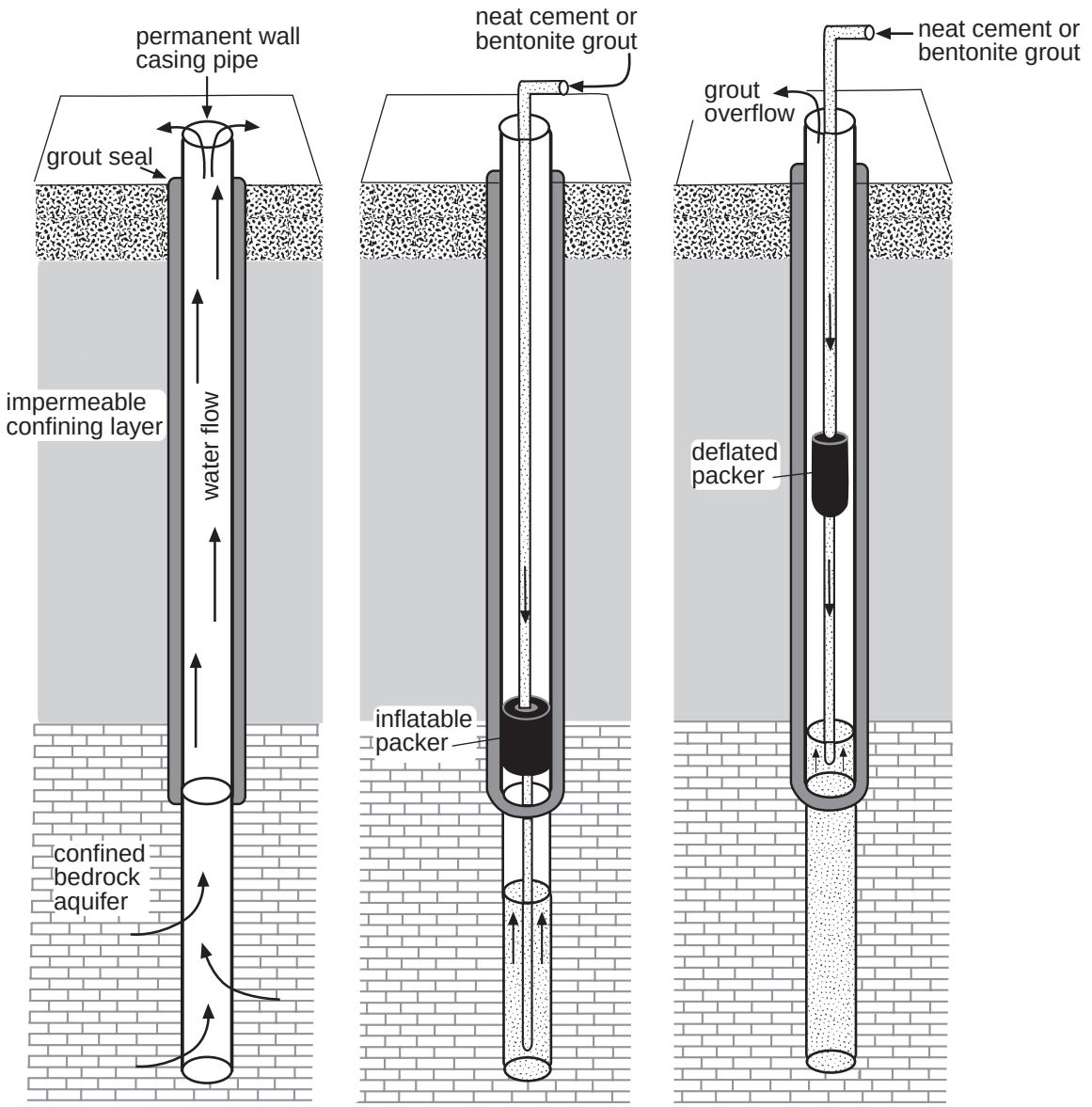


Figure O(2). Using inflatable packer to restrict flow
(After Wisconsin regulations, 1994)

The well should be pressure grouted continuously from bottom to top with a fast setting cement if the well is flowing from within the casing and around the outside of the casing, and if the casing can be removed by overdrilling. If the casing cannot be removed, tremie lines should be run along the outside of the casing if there is an annular space, and down through the middle of the casing, so that the well bore and the annular space can be continuously and simultaneously pressure grouted from the bottom of the well to the ground surface.

Sealing Wells Drilled Through Single Consolidated Aquifers

These types of wells should be sealed in the same manner as wells drilled through single unconsolidated aquifers, except that the casing removal and sealing need not occur simultaneously. Please refer to that section for sealing procedures.

Sealing Wells Drilled Through Multiple Consolidated Aquifers

Any obstructions should be removed and the casing should be removed, perforated, ripped, or left intact. The well then should be disinfected prior to the installation of the sealing materials. The well should be pressure grouted with cement or bentonite slurry from the bottom of the well to the ground surface. Alternatively, if there is enough information available about the construction of the well and

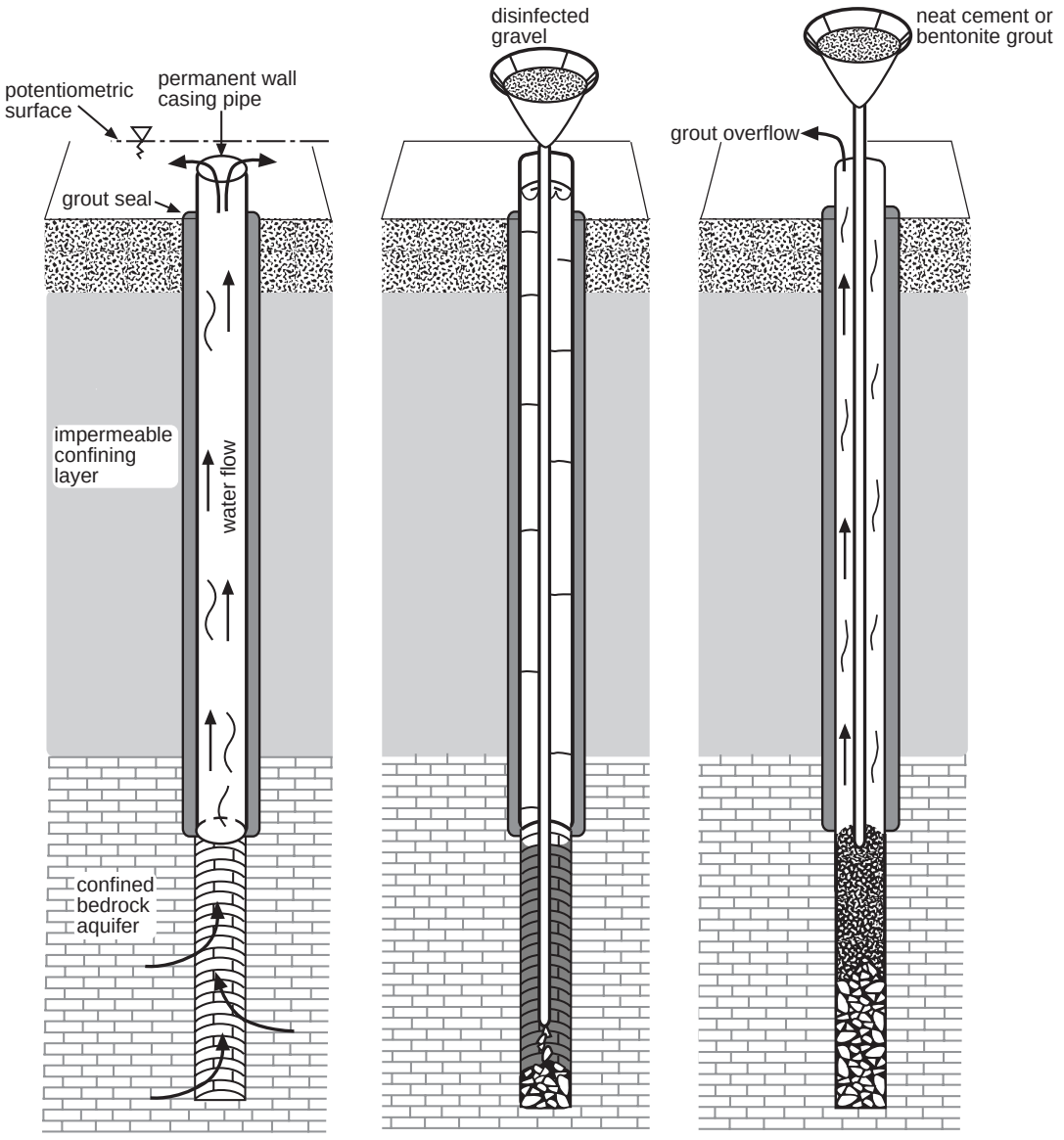


Figure O(3). Pouring disinfected gravel into well to reduce flow
(After Wisconsin regulations, 1994)

formations penetrated, gravel fill can be used in the water-bearing zones, while an impermeable sealing material (bentonite or cement) can be used to seal the confining layers. An impermeable material also can be used as a seal from the top of the uppermost aquifer to the surface. Any remaining casing should then be cut off three to four feet below the ground surface, and the resulting excavation should be filled with clean soil and mounded. Refer to Figure N for details on these sealing methods.

Sealing Wells Drilled Through Fractured or Cavernous Formations

Once initial issues of obstructions, casing, and disinfection have been taken care of, the next step is to try to determine the depth(s) at which the fractured or cavernous zones occur. During sealing, a plug may be placed above the cavernous interval, and then the well may be pressure grouted with cement or bentonite slurry from the top of the plug to the ground surface. A well with a highly fractured zone of known depth could be sealed in the same manner (Figure P). The well could be filled with coarse gravel to the top of the fractured or cavernous zone, and then pressure grouted from the top of that zone to the ground surface (no plug would be needed above the gravel fill) if the fractured or cavernous zone is not extensive. The casing should be cut off three to four feet below the ground surface, and the remaining hole filled with clean soil and mounded, to complete the sealing process.

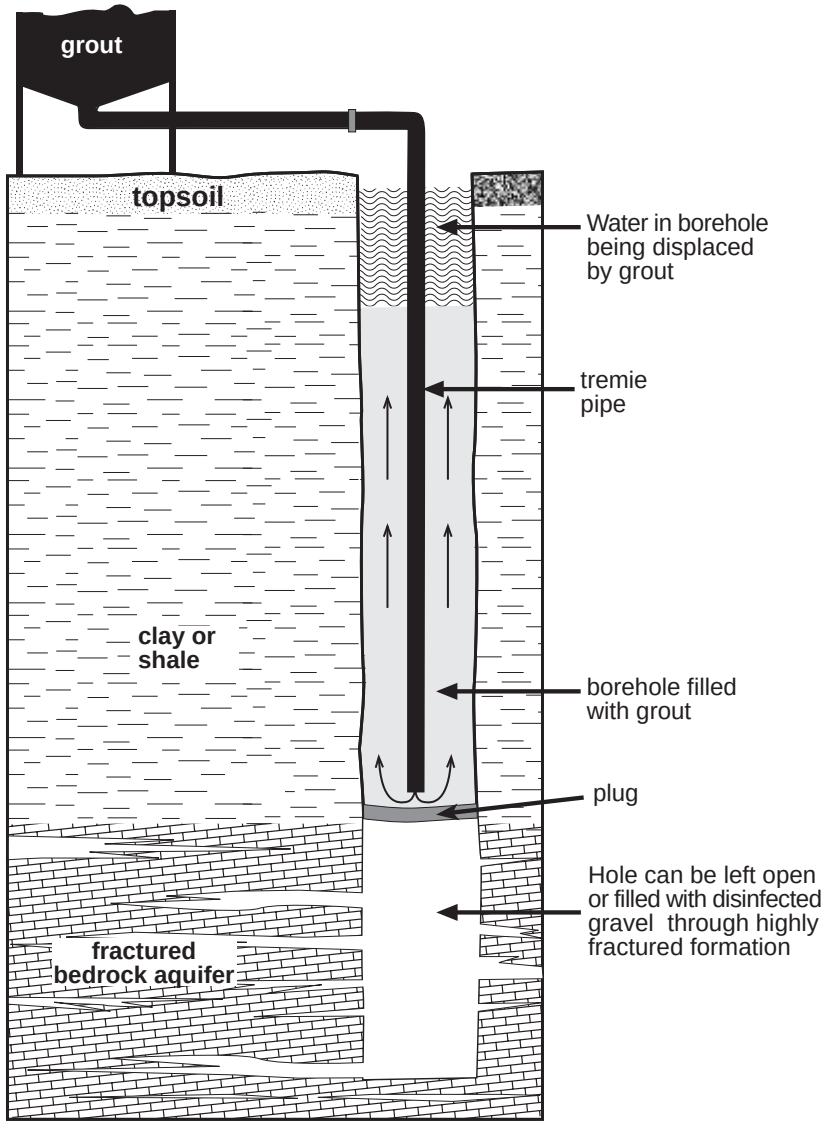


Figure P. Sealing procedures for wells penetrating fractured or cavernous formations

Sealing Flowing Wells Drilled Through Single or Multiple Consolidated Aquifers

Flowing wells drilled through consolidated aquifers can be sealed in the same manner as wells drilled through confined, unconsolidated aquifers. Please refer to the procedures in that section for well sealing information.

Sealing Wells of Unknown Construction

After any obstructions have been removed, the casing should be left in place due to the lack of information on the construction details of the well. Disinfect the well, then attempt to determine the depth of the well. The well should be pressure grouted from bottom to top with bentonite or cement slurry if the depth of the well is 300 feet or less, and the diameter is twenty four inches or less. Wells greater than 300 feet in depth should be pressure grouted as well, but specialized equipment may be required to pump grout to these depths.

The casing should be cut off three to four feet below ground surface. The remainder of the well should be filled with clean soil and mounded at ground surface.

The final step in all of these well sealing procedures is to file a well sealing report with the Ohio Department of Natural Resources, Division of Water. A summary of all the well sealing procedures discussed in this section can be found in Table 5.

Conclusions

Unsealed or improperly sealed abandoned wells present a very real threat to the quality of ground water in Ohio. With potentially as many as 200,000 existing abandoned wells scattered across the state, it is apparent that steps must be taken now to guarantee the future quality of the state's ground water resources.

The guidelines outlined in this document are the result of a genuine need for information on how to seal abandoned wells properly. While these guidelines are not intended to cover every possible scenario, they can certainly serve as a reference for basic methodologies to be followed in commonly encountered situations. It is strongly recommended that a well owner consult an experienced registered drilling contractor when preparing to seal a well. This is the best way to ensure that the well will be sealed in a manner appropriate for that type of well under those specific geologic conditions.

Table 5 Summary of Recommended Well Sealing Procedures

WELL TYPE		MATERIALS							METHOD OF INSTALLATION
		Clean fill		Concrete	Neat cement slurry	Coarse grade bentonite		Bentonite slurry w/70 second viscosity	
		Gravel and/or sand	Clay			Chips	Pellets		
UNCONSOLIDATED FORMATION(S)	Dug & bucket-drilled wells >24" in diam. and ≤ 25' in depth	No	Yes-to static level[1]	No	Yes	Yes	Yes	No	Material may be poured into well
	Driven wells	No	No	No	Yes	Yes[5]	Yes[5]	Yes	Slurry must be tremied, coarse grade bentonite must be slow-poured[2]
	Wells drilled through single aquifer ≤ 24" in diam., >25' deep	Yes- to top of aquifer or 25' below ground surface[3]	No	No	Yes	Yes[2]	Yes[2]	Yes	Slurry must be tremied, gravel may be poured, coarse grade bentonite must be slow-poured
	Wells drilled through multiple aquifers ≤ 24" in diam., >25' deep	Yes[4]	No	No	Yes	Yes[2]	Yes[2]	Yes	Slurry must be tremied, coarse grade bentonite must be slow-poured
	Flowing wells drilled through single or multiple aquifers	No[7]	No	Yes	Yes	Yes[6]	Yes[6]	Yes	Slurry must be tremied
CONSOLIDATED FORMATION(S)	Wells drilled through single aquifer ≤ 24" in diam., >25' deep	Yes-to top of aquifer or 25' below ground surface[3]	No	No	Yes	Yes[2]	Yes[2]	Yes	Slurry must be tremied, gravel poured, coarse grade bentonite must be slow-poured
	Wells drilled through multiple aquifers ≤ 24" in diam., >25' deep	Yes[4]	No	No	Yes	Yes[2]	Yes[2]	Yes	Slurry must be tremied, gravel poured, coarse grade bentonite slow-poured
	Wells drilled through fractured or cavernous formations	Yes-through fractured or cavernous zones only	No	Yes	Yes-from top of plug or cavern fill to surface	Yes-from top of plug or cavern fill to surface	Yes-from top of plug or cavern fill to surface	Yes-from top of plug or cavern fill to surface	Slurry must be tremied, gravel only (no sand should be used) poured, coarse grade bentonite slow-poured
	Flowing wells drilled through single or multiple aquifers	No[7]	No	Yes	Yes	Yes[6]	Yes[6]	Yes	Slurry must be tremied
Drilled wells of unknown construction		No	No	No	Yes	No	No	Yes	Slurry must be tremied
Any wells >24" in diam. and >25' deep		Yes[4]	No	No	Yes	Yes[2]	Yes[2]	Yes	Slurry must be tremied, chips and pellets must be slow-poured and tamped periodically

[1] The clay fill must be topped with a 1ft. thick layer of bentonite or cement extending 6" beyond the original outside diameter of the well.
[2] The bentonite chips or pellets must be poured slowly over a wire mesh screen and into the well at a rate no faster than 3 minutes per 50 pound bag. Pellets should be used in wells no deeper than 100 feet, chips in wells no deeper than 200 feet. These products should be tamped periodically and hydrated if they are placed above the water table, or if the well is dry. The diameter of the well must be 4" or greater.
[3] The well should then be filled to the surface with cement or bentonite products.
[4] Gravel and/or sand may be used in aquifer zones provided there is an impermeable plug of bentonite or cement placed between these zones, and from the top of the uppermost zone to the surface.
[5] Coarse grade bentonite may be used in wells 25 feet deep or less as long as it is tamped periodically. Wells deeper than 25 feet must be sealed with a tremied slurry.
[6] Coarse grade bentonite may be used if flow has been stopped by casing extension and products are able to fall to the bottom of the well. Well should be 200 feet deep or less, and at least 4 inches in diameter.
[7] Gravel may be used in flowing wells only to slow flow enough to allow grouting to proceed.

References

Aller, L., T.W. Bennett, G. Hackett, R.J. Petty, J.H. Lehr, H. Sedoris, D.M. Nielsen and J.E. Denne, 1991. Handbook of suggested practices for the design and installation of ground water monitoring wells. Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, EPA/600/4-89/034.

American Colloid Company, no year. Volclay grout product information. American Colloid Company, Arlington Heights, Illinois.

American Society for Testing and Materials, 1992. Standard specification for portland cement, method C150-92. Annual Book of American Society for Testing Material Standards, Philadelphia, Pennsylvania, Volume 4.01, pp. 121-125.

_____, 1993. Decommissioning of ground water wells, vadose zone monitoring devices, boreholes, and other devices for environmental activities, method D5299-92. Annual Book of American Society for Testing Material Standards, Philadelphia, Pennsylvania, Volume 4.08, pp. 270-285.

American Water Works Association, 1984. Appendix I: abandonment of test holes, partially completed wells, and completed wells. American Water Works Association Standard for Water Wells, American Water Works Association, Denver, Colorado, pp. 45-47.

Bates, R.L., 1969. Geology of the industrial rocks and minerals. Dover Publications, Inc., New York, New York.

Carlton, R.W., 1975. Potential use of Ohio clays in the well-plugging industry. Ohio Department of Natural Resources, Division of Geological Survey, Report of Investigations No. 97, 9 pp.

Coleman, J.R. and G.I. Corrigan, 1941. Fineness and water-cement ratio in relation to volume and permeability of cement. Petroleum Technology, Technical Publication No. 1226, pp. 1-11.

Driscoll, F.G., 1986. Groundwater and wells. Johnson Filtration Systems, St. Paul, Minnesota, 1089 pp.

Freeze, R.A. and J.A. Cherry, 1979. Groundwater. Prentice-Hall, Inc., Englewood Cliffs, New Jersey, 604 pp.

Gaber, M.S. and B.O. Fisher, 1988. Michigan water well grouting manual. Michigan Department of Public Health, Ground Water Quality Control Section, 83 pp.

Glanville, T., 1989. Plugging abandoned wells. Iowa State University Extension, Ames, Iowa, 6 pp.

Golden, S., personal communication, 1995. Ohio Department of Health, Bureau of Local Services.

Gordon, D.L., 1988. Guidelines for plugging abandoned water wells. Iowa Department of Natural Resources, Technical Information Series 15, 46 pp.

Gray, G.R. and H.C.H. Darley, 1981. Composition and properties of oil well drilling fluids. Gulf Publishing Co., Houston, Texas.

Hackett, G., 1987. Drilling and constructing monitoring wells with hollow-stem augers part 1: drilling considerations. Ground Water Monitoring Review, Fall 1987, pp. 51-62.

_____, 1988. Drilling and constructing monitoring wells with hollow-stem augers part 2: monitoring well installation. Ground Water Monitoring Review, Winter 1988, pp. 60-68.

Halliburton Services, 1981. Halliburton cementing tables. Duncan, Oklahoma.

Ingersoll-Rand Company, 1988. The hole story. Ingersoll-Rand Company, Roanoke, Virginia, 11 pp.

Johnson, R.C. Jr., C.E. Kurt and G.F. Dunham, Jr., 1980. Well grouting and casing temperature increases. Ground Water, Volume 18, No. 1, pp. 7-13.

King, D.P., 1992. Sealing abandoned water wells: a campaign primer. Illinois Department of Energy and Natural Resources, 51 pp.

Kraemer, C.A, J.A. Shultz and J.W. Ashley, 1991. Monitoring well post installation considerations in: D.M. Nielsen (editor), Practical Handbook of Ground-Water Monitoring. Lewis Publishers, Inc., Chelsea, Michigan, pp. 333-363.

Kurt, C.E., 1983. Cement-based seals for thermoplastic water well casings. Water Well Journal, Volume 37, No. 1, pp. 38-40.

Midwest Plan Service, 1979. Private water systems handbook. Midwest Plan Service MWPS-14, Iowa State University, Ames, Iowa, 72 pp.

Molz, F.J. and C.E. Kurt, 1979. Grout-induced temperature rise surrounding wells. Ground Water, Volume 17, No. 3, pp. 264-269.

N.L. Baroid, 1989. Baroid drilling fluids products. N. L. Baroid/N. L. Industries, Inc., Houston, Texas.

Ohio Environmental Protection Agency, 1995. Technical guidance manual for hydrogeologic investigations and ground water monitoring, chapter 9. Columbus, Ohio.

Peck, C., 1987. "Grouting as seen by a cable tool driller" in Drill Bits, Fall 1987. Ohio Department of Natural Resources, Division of Water, 6 pp.

Portland Cement Association, 1979. Design and control of concrete mixtures. Twelfth Edition, Skokie, Illinois.

Renz, M.E., 1989. In-situ decommissioning of ground water monitoring wells. Water Well Journal, Volume 43, No. 5, pp. 58-60.

Smith, D.K., 1976. Cementing. Society of Petroleum Engineers of AIME, New York, New York.

Smith, S.A., 1994. Well & borehole sealing: importance, materials, and recommendations for decommissioning. Ground Water Publishing Co., 69 pp.

Smith, S. and C. Mason, 1985. Bentonite grouting. Water Well Journal, Volume 39, No. 2, pp. 35-37.

Troxell, G.E., H.E. Davis and J.W. Kelly, 1968. Composition and properties of concrete. McGraw-Hill, New York, New York.

U. S. Department of the Interior, 1977. Ground water manual. U. S. Department of the Interior, Bureau of Reclamation, U. S. Government Printing Office, Washington, D. C., p. 8.

Wisconsin Administrative Code, 1994. Well and drillhole abandonment. Wisconsin Administrative Code, Section NR 812.26.

Wisconsin Department of Natural Resources, 1993. Well abandonment. Wisconsin Department of Natural Resources, Madison, Wisconsin, 6 pp.

Zahniser, T.E. and M. Gaber, 1993. Plugging abandoned wells. Michigan State University Extension, Bulletin WQ 40, 6 pp.

Glossary

Annular space	the space between the well casing and the borehole wall.
Aquifer	a geological formation, part of a formation, or group of formations that is capable of yielding a significant amount of water to a well or spring.
Bailing	the use of a bucket, or rigid tube or pipe with a valve to remove fluid volumes or debris and cuttings from a well.
Borehole	a hole in the earth made by a drill; the uncased drill hole from the surface to the bottom of the well.
Casing	an impervious, durable pipe placed in a well to prevent the walls from caving and to seal off surface drainage or undesirable water, gas, or other fluids, and prevent their entering the well.
Cistern	a large receptacle used for storing water, especially an underground tank in which rainwater is collected.
Confining bed	a body of impermeable or distinctly less permeable material stratigraphically adjacent to one or more aquifers.
Confined aquifer	an aquifer bounded above and below by beds of distinctly lower permeability than that of the aquifer itself and which contains groundwater under pressure greater than that of the atmosphere. This term is synonymous with the term “artesian aquifer.”
Consolidated	lithified geologic materials. In Ohio, these materials constitute formations such as sandstone, limestone, and shale.
Cuttings	chips removed from the borehole by a bit in the process of well drilling.
Density	the mass or quantity of a substance per unit volume, usually expressed in grams per cubic centimeter.
Drill rod	the extension rods used to attach the bit to the drilling rig to enable the penetration into the earth.
Drill bit	a device used on the end of a drilling stem or rod for the purpose of penetrating earth formations. Drilling bits are usually made of a hardened material so as to last an extended period of time.
Drilling mud	a special mixture of clay, water, and chemical additives pumped down hole through the drill pipe and drill bit. The mud is used to lubricate and cool the bit and to float cuttings to the surface for removal.
Drill tools	general term associated with all equipment used in the drilling process. Tools, bits, rods, stems, etc.
Dug well	a well excavated into a generally shallow, unconsolidated aquifer in which the side walls may be supported by material other than standard weight steel casing.
Filter pack	siliceous, well-rounded, clean, and uniform sand or gravel that is placed between the borehole wall and the well screen to prevent formation material from entering through the screen.
Formation	a body of consolidated or unconsolidated rock characterized by a degree of lithologic homogeneity which is prevailingly, but not necessarily, tabular and is mappable on the earth’s surface or traceable in the subsurface.
Geologic conditions	the distribution, types, and structural features of earth materials present in any given area.

Geotechnical boring	borings installed to determine the geological and engineering properties of subsurface soils.
Ground water	any water below the surface of the earth in a zone of saturation.
Grout	As used in these guidelines, grout is a fluid mixture of water and cement or water and bentonite that is of a consistency to be pumped through a small-diameter pipe.
Hydrated	the incorporation of water into the chemical composition of mineral.
Hydraulic head	the height of the free surface of a body of water above a given subsurface point; a reflection of the ground water level plus the pressure head.
Hydrogeologic conditions	the occurrence, distribution, and quality of subsurface water within consolidated and/or unconsolidated earth materials in a given area.
Mesh	one of the openings in a screen or sieve. The value of the mesh is usually given as the number of openings per linear inch.
Microannulus	for the purpose of this guidance document the term means the space between the sealing material and the casing and/or the formation. This is caused by the shrinkage of the sealing material.
Monitoring well	any excavation that is drilled, cored, bored, washed, driven, dug, jetted, or otherwise constructed for the purpose of extracting groundwater for physical, chemical, or biological testing, or for the purpose of determining the quantity or static level of ground water on a continuing basis.
Mud balance	a scale that measures a specific volume of grout slurry (density) and is expressed in pounds per gallon.
Permeability	the capacity of a porous rock, sediment, or soil for transmitting fluid; a measure of the relative ease of fluid flow across a pressure gradient.
Pitless adapter	a device or an assembly of parts which permits water to pass through the casing or extension thereof; provides access to the well and to the parts of the water system within the well; and provides for the transportation of the water and the protection of the well and water therein from surface or near surface contaminants.
Plasticity	the capability of being deformed permanently without rupture.
Pore	a tiny opening, usually microscopic, in consolidated or unconsolidated materials.
Recharge	the processes by which water is absorbed and is added to the saturation zone, either directly into a formation, or indirectly by way of another formation.
Saturated zone	the portion of consolidated or unconsolidated materials in which all of the pore space is occupied by water.
Static level	the distance measured from the established ground surface to the water surface in a well being neither pumped nor under the influence of pumping nor flowing under artesian pressure.
Stratigraphic	the arrangement of consolidated and unconsolidated strata.
Test boring	a boring designed to obtain information on ground water quality and/or geological and hydrogeological conditions.
Unconsolidated	not lithified but loose, soft geologic materials. Alluvium, soil, gravel, clay, and overburden are some of the terms used to describe a formation consisting of unconsolidated materials.
Viscosity	the property of a fluid or semi-liquid to offer internal resistance to flow.
Well	any excavation, regardless of design or method of construction, created for

	any of the following purposes: (1) removing ground water from or recharging water into an aquifer; (2) determining the quantity, quality, level, or movement of ground water in or the stratigraphy of an aquifer; and (3) removing or exchanging heat from ground water.
Well screen	a machine-slotted or wire-wrapped portion of casing used to stabilize the sides of the borehole, prevent the movement of fine-grained material into the well, and allow the maximum amount of water to enter the well with a minimum of resistance.
Yield	the quantity of water which may flow or be pumped from the well per unit of time.

Appendix 1

Contact Agencies

Ohio Department of Agriculture, Pesticide Regulation Section

The Ohio Department of Agriculture (ODA) does not currently provide routine well analysis for pesticides; however, the Pesticide Regulation Section of ODA will sample any well where it is suspected that the use of a pesticide may have contaminated the well. Samples must be collected by an ODA inspector to protect sample integrity, and then analyzed at the ODA laboratory in Reynoldsburg. The Pesticide Regulation Section will investigate to determine how the well was contaminated if a water sample is positive for a pesticide. The ODA will advise the well owner on how to clean up the well, and, if necessary, take appropriate enforcement action under Ohio Pesticide Law. The Ohio Department of Agriculture can be contacted at 614-728-6200.

Ohio Department of Commerce, Division of State Fire Marshal, Bureau of Underground Storage Tank Regulations (BUSTR)

In the event that any potable or non-potable water well is suspected of being contaminated with petroleum from a leaking petroleum underground storage tank (such as those used at gas stations), contact BUSTR at 1-800-686-2878 before sealing the well.

Ohio Department of Health, Division of Quality Assurance

For information on specific regulatory requirements for sealing private wells, or for questions about possible contamination with substances other than pesticides or petroleum products, contact the local health department or the Ohio Department of Health Private, Water System Program (PWSP) at 614-466-1390.

To determine the registration status of a particular private water system contractor (i.e., drilling contractor or pump installer), contact the local health department or the ODH-PWSP Public Inquiries Assistant at 614-466-0148.

Ohio Department of Natural Resources, Division of Mines & Reclamation

The Division of Mines and Reclamation regulates the abandonment of test borings for coal and industrial minerals exploration through the permitting process under the Ohio Revised Code Chapter 1513 and 1514. Most exploratory borings are mined through the removal of the coal or industrial mineral. Those borings that are not removed by mining are required to be properly sealed using procedures approved by the Division. The Division also recommends that the coal operator properly seal any original private water supply wells that are replaced by a new well drilled as a result of a water supply replacement order by the Chief. The Division investigates any ground water contamination complaints related to coal and industrial minerals mining activities.

Ohio Department of Natural Resources, Division of Oil & Gas

Personnel in the Groundwater Protection Section of the Division investigate ground water contamination cases when oil and gas operations are the suspected cause. If there is reason to believe that an unsealed, unused well on a property is an oil or gas well, the Division also has an Idle and Orphan Well Program that addresses the need to seal abandoned oil and gas wells. For more information on these two programs, contact the Division's Central Office at 614-265-6926.

Ohio Department of Natural Resources, Division of Water

The Ohio Revised Code, Section 1521.05, requires that a well sealing report be filed with the Division of Water for all wells sealed in the State of Ohio. Copies of the well sealing report can be obtained from the Division by calling 614-265-6739. The Division also collects well log and drilling reports required to be filed by drilling contractors for wells drilled across the state. This authority also comes from Section 1521.05 of the Ohio Revised Code. Requests for copies of well log and drilling reports on file can be made by calling 614-265-6740.

Ohio Environmental Protection Agency

The Ohio Revised Code 6111.42 gives the Ohio EPA authority to prescribe regulations for the drilling, operation, maintenance, and sealing of abandoned wells as deemed necessary by the director to prevent the contamination of underground waters in the state, except that such regulations do not

apply to non-public potable wells. Currently, the Ohio EPA, Division of Drinking and Ground Waters, has regulations for the sealing of public water supply wells (OAC 3745-9-10) and for wells used for the purpose of injecting fluids into the ground (OAC 3745-34-07, 60, and 36). Sealing of monitoring wells is generally handled by the Division that has regulatory authority over the site/facility. For information on specific regulatory requirements for public drinking water wells or for injection wells, the Division of Drinking and Ground Waters should be contacted at 614-644-2752.

The Ohio EPA has no regulations/requirements for a person to report contamination in their private well. Reporting of ground water contamination is only required if an entity is monitoring ground water in accordance with hazardous or solid waste rules. In general, the Ohio EPA will not respond to a request to evaluate a contaminated private well unless the local or state health department requests assistance in investigating the source of the problem. However, this will not affect how the well should be sealed, but may affect when it is sealed if additional investigation is initiated.

An exception to this occurs if the well was used to inject fluid waste. If it was used as an injection well, the owner/operator must contact the Division of Drinking and Ground Waters, Underground Injection Control Unit (U.I.C.) of the Ohio EPA at 614-644-2905. Specific requirements must be followed for the sealing of injection wells.

Appendix 2

Cost Considerations

When obtaining estimates for well sealing from local drilling contractors, it is necessary to understand upon what the contractor's price will be based. As seen in the guidelines, there are many steps in sealing a well properly. The preliminary steps, which involve researching the history of the well by obtaining a well log and drilling report or talking to neighbors, can be done by the well owner. However, except perhaps in the case of shallow dug wells, the remaining steps of the sealing process should be handled by a registered drilling contractor with experience in sealing abandoned wells.

The contractor must consider many factors in order to provide the best estimate possible. Besides the cost of the materials used to seal the well, the contractor must also consider the cost of the labor and equipment used to install them. For example (brand names used in this section are for illustrative purposes only and do not imply an endorsement of any particular product), most contractors can currently buy a bag of Benseal (a granular bentonite product installed in slurry form) from their supplier for approximately $7 per bag. The contractor must then add the cost of labor for installing the Benseal, the cost of the pumping equipment, and the cost of the polymer mixed with the Benseal. This will raise the price per bag of Benseal to approximately $75 per bag, installed. Multiply this by the 5 bags it takes to seal a 100 foot deep, 6 inch diameter well (see Table 6, and note that the number of bags required has been rounded up to the nearest whole bag), and the result is an estimate of $375. Or, suppose the well is to be sealed with a neat cement slurry. The installed price of cement slurry per bag is about $30. To seal the same well cited above, it will cost almost $480 because it will take nearly 16 bags of cement to fill the well (Table 6). These prices will probably not reflect the removal of any obstructions from the well, such as pumping equipment, the disinfection of the well, or removing, perforating, or cutting off the casing. These are all factors that will affect the contractor's estimate.

Another commonly used well sealing material is coarse grade bentonite, which consists of chips of bentonite ranging from $^3/_8$" to $^3/_4$" in size. Coarse grade bentonite is poured into the well, not pumped like a slurry. Therefore, the equipment and labor costs are lower. Assume that Holeplug is to be used to seal a 100 foot deep, 6 inch diameter well. Most contractors could probably purchase Holeplug from their suppliers for about $7 per bag. Add labor to install the Holeplug, and the price is raised to approximately $15 per bag. However, it will take almost 29 bags of Holeplug (Table 6) to fill the well. The result is a cost estimate of $435. Then, any of the factors mentioned in the previous discussion could add to this cost.

In some cases, more than one type of material may be used to seal the well. If the well to be sealed penetrates a highly fractured bedrock formation, such as limestone, the contractor may wish to fill the fractured portion of the well with gravel or aggregate, then finish the sealing process with a cement or bentonite slurry or a coarse grade bentonite product. Table 6 shows volumes of a standard size aggregate required to fill wells of varying depths and diameters. One cubic yard (27 cubic feet) of #8 aggregate (roughly $^3/_8$" in size) averages about $16. With labor included the cost averages $40. Keep in mind that the well should not be filled entirely with this aggregate. There must be impermeable material between the top of the aggregate layer and the ground surface. This type of sealing procedure should be used only under certain geologic conditions.

The most costly factor in sealing a water well is removing or perforating the casing. Depending upon the equipment used to remove or perforate the casing and the hours of labor involved in the process, the cost of the well sealing operation could be increased anywhere from $200 to $500. Some of the other factors, such as removing existing pumping equipment and cutting off the casing below ground surface, also may be labor intensive if any difficulties are encountered. This too will add to the total sealing price.

Obviously, there are many factors to be considered when a contractor provides an estimate for sealing a well. By being aware of these factors, the well owner can ask the contractor intelligent questions and be involved in the estimation process. This also will give the well owner a basis for evaluating different estimates to determine which contractor to hire to do the job.

Table 6 Table Comparing Volumes of Different Well Sealing Materials Required to Seal a 100 Foot Well

Hole Diameter Inches	Gallons Per Foot	Gallons to be Plugged in 100'well	Bags Required to Plug a 100 ft Well*			Hole Volume Cu Ft/Ft depth	Feet Filled by One Bag of Holeplug	Bags of Holeplug to Fill 100' Well*	Cu Ft of #8 Aggregate to Fill a 100' Well
			Benseal	Enviroplug	Neat Cement				
2	0.17	17	1	1	2	0.022	31.30	4	2.2
3	0.38	38	2	2	4	0.049	14.30	7	4.9
4	0.67	67	3	3	7	0.087	7.90	13	8.7
5	1.00	100	4	5	11	0.136	5.10	20	13.6
6	1.51	151	5	7	16	0.196	3.50	29	19.6
7	2.05	205	7	10	22	0.267	2.60	39	26.7
8	2.70	270	9	13	28	0.349	2.00	51	34.9
9	3.40	340	11	16	35	0.442	1.60	64	44.2
10	4.20	420	13	19	44	0.545	1.30	79	54.5
11	5.00	500	16	23	52	0.660	1.10	95	66.0
12	6.00	600	19	27	62	0.785	0.89	113	78.5
15	9.50	950	30	43	98	1.227	0.57	177	122.7
18	13.60	1360	42	61	140	1.767	0.39	255	176.7
20	16.80	1680	52	75	173	2.181	0.32	315	218.1
25	26.00	2600	80	117	267	3.409	0.20	491	340.9
30	38.00	3800	117	170	390	4.909	0.14	707	490.9
60	152.00	15200	468	679	1559	20.322	0.04	2500	2032.2

* Number of bags has been rounded up to the next whole bag.

Yield Calculations:

Neat Cement: One 94 lb bag plus 6 gallons of water equals 9.75 gallons of grout
Benseal: One 50 lb bag plus 10 oz. of E-Z Mud plus 30 gallons water equals 32.5 gallons of grout
Enviroplug: One 50 lb bag plus 2.5 lb of activator plus 20 gallons of water equals 22.4 gallons of grout

Holeplug is a granular bentonite product, $^{3}/_{8}$"– $^{3}/_{4}$" in size that is poured, not pumped, into a well.

Table based on product information published by NL Baroid, Wyo-Ben. Inc., and Chemgrout Inc.

Appendix 3

List of Acronyms

ASTM	-	American Society for Testing and Materials
API	-	American Petroleum Institute
BUSTR	-	Bureau of Underground Storage Tank Regulations
DDAGW	-	Division of Drinking and Ground Waters
EPA	-	Evironmental Protection Agency
OAC	-	Ohio Administrative Code
ODA	-	Ohio Department of Agriculture
ODH	-	Ohio Department of Health
ODNR	-	Ohio Department of Natural Resources
ORC	-	Ohio Revised Code
PWSP	-	Private Water System Program
UIC	-	Underground Injection Control

Appendix 4

Sealing Monitoring Wells and Boreholes[1]

Boreholes that are not completed as monitoring wells and monitoring wells that are no longer being sampled or used for ground water level measurements must be sealed properly to: 1) prevent poor quality water from one saturated zone entering another, 2) prevent contamination of the ground water by surface contaminants, 3) restore an aquifer to as close to its original condition as possible, 4) eliminate physical hazards, and 5) reduce potential for future liability. A suitable program should be designed and implemented to meet these objectives.

The sealing material and the method of sealing depend on: 1) the design and construction of the well/borehole, 2) hydrogeologic conditions, 3) chemical environment, 4) safety hazards and 5) disposal of contaminated materials removed. In general, well sealing should consist either of a method for well removal and simultaneous grouting of the borehole with bentonite, neat cement, or a method for grouting in-place that ensures complete sealing.

Sealing Materials

The chosen sealing material should:

- Not react with contaminants, ground water, or geologic materials.
- Have a hydraulic conductivity comparable to or lower than that of the in-situ material.
- Form a tight bond with the borehole wall and the casing.
- Be resistant to cracking and/or shrinking.
- Be of sufficient structural strength to withstand subsurface pressures.
- Be capable of being placed at the appropriate depth.

No single material will exhibit all of the characteristics mentioned above. Therefore, every situation must be evaluated carefully to determine the appropriate choice. Generally, materials used are comprised of concrete, neat cement, or sodium bentonite.

Most wells completed in unconsolidated formations or non-creviced rock may be satisfactorily sealed with neat cement or bentonite. Wells that penetrate limestone or other creviced or channeled rock formations should be filled with concrete grout or neat cement to ensure seal permanence. The use of fine-grained materials to seal creviced rock may not be desirable because the materials might be displaced by flow of water through crevices (American Water Works Association, 1984). Neat cement or sodium bentonite should be used for sealing an abandoned well/borehole below the water table (Gordon, 1988). Pure sodium bentonite placed above the water table should be hydrated if it is not installed in slurry form due to the lack of water for hydration of pellets or chips. At no time should a borehole or well be backfilled with cuttings or with any materials of unknown integrity. However, in some geologic environments, such as coarse gravel, where excessive loss of sealing materials may occur, or when grout may affect the water quality of nearby monitoring wells, clean sand or gravel in conjunction with regular materials can be used (Gordon, 1988; Kraemer et al., 1991).

Procedures

Planning

Careful review should be conducted prior to sealing monitoring wells. This may include:

- Review of records pertaining to well construction and repair or modifications.
- Review of all analytical chemical data for soil and ground water.
- Review of the hydrogeologic/geologic characteristics in the vicinity of the well.
- Current conditions of the well such as total depth, amount of siltation, etc.

---

[1] The information in this section is from Chapter 9 of the Ohio Environmental Protection Agency's "Technical Guidance Manual for Hydrogeologic Investigations and Ground Water Monitoring", 1995.

If a well is to be left in place, borehole geophysical techniques may be helpful in determining its integrity. This may include caliper logs to measure inside diameter; television logs to identify casing breaks, screen size, etc.; gamma logs to verify geologic information; cement bond logs to determine if the casing is firmly attached to the grout; flow logs to determine if vertical flow occurs within the casing; and hydraulic integrity tests to determine if the casing is intact (ASTM, 1993).

Prior to the sealing of monitoring wells, it is recommended that a work plan detailing the procedures/methods be submitted to the appropriate regulatory authority[2]. The information should include:

- Reasons for sealing.
- Identification and location coordinates.
- Casing diameter and material.
- Screen material, length, and depth.
- Total depth.
- Geologic materials opposite well screen.
- Drilling log and construction diagrams.
- Type and concentrations of contaminants present[3], if any.
- Procedure for disposal of any contaminated soil, well construction material, and water.
- Method for sealing.
- Type of sealing material.
- An estimation of the volume of sealing material needed.
- Measures to protect the health and safety of individuals.

Field Procedure

Monitoring wells have often been sealed by pulling the surface casing where possible, followed by pouring cement or bentonite into the hole. This procedure is inappropriate, especially if the construction of the well is unknown or the well intake spans more than one saturated zone. Incomplete seals may form due to bridging. Additionally, the procedure has little effect on the filter pack, which may allow communication between saturated zones.

The following basic procedure is recommended for sealing monitoring wells and boreholes. Steps 1 and 2 are not necessary for sealing exploratory boreholes. It should be understood that no single method and material are suitable for all situations. Site-specific characteristics may merit modifications or procedures not discussed below. Additional information can be found in the references listed.

1. Inspect the well and remove any obstacles (i.e., pumps, pressure lines, other debris, etc.) that may interfere with the placement and performance of the sealing material. If necessary, a camera survey can help to identify the depth and construction of the well if this information is not known. The outer protective casing should be removed.

2. Since the primary purpose of sealing is to eliminate vertical fluid movement, it is strongly recommended that the casing and screen be removed and the boring be overdrilled to remove the annular seal and filter pack, unless it can be determined that the original grout seal is intact. When the well is removed, there is less concern about channeling in the annular space or an inadequate casing/grout seal (Aller et al., 1991). The casing and well screen can be removed by pulling or bumping the casing, overdrilling around the casing using a hollow stem auger, or drilling out the well using a solid stem auger or rotary bit. The method used should depend on the type, length, and diameter of the casing, conditions of the annular seal, and site geology. Aller et al. (1991) and ASTM 5299-92 provided a discussion on various removal techniques. The borehole should be overdrilled using a bit with a diameter at least $1^1/_2$ times greater than the original diameter of the borehole. Drilling should

---

[2] If a regulated entity is conducting a hydrogeologic investigation or a ground water monitoring program, a well sealing work plan should be submitted prior to initiating the program. In this situation, a separate workplan is not necessary.

[3] If contamination was detected or suspected in the original well or boring, appropriate health and safety requirements should be followed.

be slightly deeper than the original depth to assure complete removal. To achieve an effective seal, the borehole should be cleared of any excess mud filtercake.

In some instances, such as when safety problems occur, or when dealing with large diameter wells, casing removal can be difficult. If the well construction is known, the screen and filter pack do not span more than one saturated zone, and circumstances prevent complete removal of casing and screen, then the following procedure can be used (based on Renz, 1989):

a. The well can be filled with clean silica sand to one foot above the screen in the event that the screened area is adjacent to a highly permeable formation.

b. One foot of bentonite pellets can be placed above the screen in a manner that prevents bridging (i.e., through a tremie pipe or by tamping after installation).

c. The pellets should be hydrated.

d. To allow the sealant to permeate and be effective, the casing should be perforated to one foot above the bentonite seal either by splitting it vertically (synthetic casing) or by making horizontal cuts every two feet with retractable blade (steel casing).

3. The borehole should be pressure grouted using a tremie pipe as the drilling stem is removed. The sealant should be applied in one continuous procedure to prevent segregation, dilution, and bridging (Aller et al., 1991). The pipe should be in constant contact with the sealant to prevent air pockets from forming. The borehole should be sealed from the bottom up to the frost line (approximately two to three feet from the surface). The overflowing grout should be regularly evaluated as it reaches the surface. When the observed material is similar to that being pumped in, this stage of the sealing is considered complete. Wells sealed in-situ should be sealed from the bottom up to approximately five feet from the surface.

4. The grout plug should be inspected 24 hours after installation to check for settling and grout should be added if needed. If the well is sealed in place, the casing should be cut off approximately five feet below ground level and a PVC or stainless steel cap should be emplaced. The boring should be grouted to within two to three feet from the surface with appropriate material. Monitoring wells sealed in place should be marked with a piece of metal to allow for location by a metal detector or magnetometer (Aller et al., 1991).

5. The remaining area above the plug should be completed in a manner that is compatible with the site. For example, its top can be covered with one to two feet of soil if vegetative growth is desired. If the area is to be surfaced, then the final seal can be completed with cement or concrete.

6. Proper sealing of monitoring wells should be documented and reported to the implementing regulatory agency managing the site. The information should include, at a minimum:

- Identification (e.g. registration number, location, owner, other features).
- Well construction details.
- Date, time, person responsible, and contractor/consultant performing the work.
- Authority under which the sealing was performed.
- Procedures and materials used.
- Method/procedures for disposal of any contaminated materials.

Additionally, Ohio Revised Code 1521.05(B) requires that a well sealing report be filed with the Ohio Department of Natural Resources (ODNR) on forms supplied by the Department. Figure A is an example of this form. It can be obtained from ODNR, Division of Water (614-265-6739).

ATTACHMENT B

QUALITY ASSURANCE PROJECT PLAN (QAPP)
FOR THE
REMEDIAL DESIGN / REMEDIAL ACTION
CHEMICAL RECOVERY SYSTEMS, INC. SITE

Prepared for

CRS Site RD/RA Performing Parties
Chemical Recovery Systems, Inc. (CRS) Site
Elyria, Ohio

September 2010

Revised April 2012

Prepared by:

Brown and Caldwell
4700 Lakehurst Court, Suite 100
Dublin, Ohio 43016

139452

Publication Numbers:

EPA: EPA-505-B-04-900C
DoD: DTIC ADA 427486

Intergovernmental Data Quality Task Force

Workbook for Uniform Federal Policy for Quality Assurance Project Plans

Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs

Part 2A: UFP-QAPP Workbook

This Workbook supplements Part 1 of the UFP-QAPP, the UFP-QAPP Manual. Proper completion of these worksheets requires knowledge of the QAPP elements explained in the Manual.

Final
Version 1
March 2005

Publication Numbers:

EPA: EPA-505-B-04-900C
DoD: DTIC ADA 427486

TABLE OF CONTENTS

INTRODUCTION		
QAPP Worksheet No. 1.	Title and Approval Page	1
QAPP Worksheet No. 2.	QAPP Identifying Information	2
QAPP Worksheet No. 3.	Distribution List	6
QAPP Worksheet No. 4.	Project Personnel Sign-Off Sheet	7
QAPP Worksheet No. 5.	Project Organizational Chart	8
QAPP Worksheet No. 6.	Communication Pathways	9
QAPP Worksheet No. 7.	Personnel Responsibilities and Qualifications Table	10
QAPP Worksheet No. 8.	Special Personnel Training Requirements Table	11
QAPP Worksheet No. 9.	Project Scoping Session Participants Sheet	12
QAPP Worksheet No. 10.	Problem Definition	13
QAPP Worksheet No. 11.	Project Quality Objectives/Systematic Planning Process Statements	14
QAPP Worksheet No. 12.	Measurement Performance Criteria Table	17
QAPP Worksheet No. 13.	Secondary Data Criteria and Limitations Table	33
QAPP Worksheet No. 14.	Summary of Project Tasks	34
QAPP Worksheet No. 15.	Reference Limits and Evaluation Table	36
QAPP Worksheet No. 16.	Project Schedule/Timeline Table	45
QAPP Worksheet No. 17.	Sampling Design and Rationale	46
QAPP Worksheet No. 18.	Sampling Locations and Methods/SOP Requirements Table	47
QAPP Worksheet No. 19.	Analytical SOP Requirements Table	48
QAPP Worksheet No. 20.	Field Quality Control Sample Summary Table	49
QAPP Worksheet No. 21.	Project Sampling SOP Reference Table	51
QAPP Worksheet No. 22.	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	52
QAPP Worksheet No. 23.	Analytical SOP Reference Table	53
QAPP Worksheet No. 24.	Analytical Instrument Calibration Table	55
QAPP Worksheet No. 25.	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	59
QAPP Worksheet No. 26.	Sample Handling System	61
QAPP Worksheet No. 27.	Sample Custody Requirements	62
QAPP Worksheet No. 28.	QC Samples Table	65
QAPP Worksheet No. 29.	Project Documents and Records Table	71
QAPP Worksheet No. 30.	Analytical Services Table	72
QAPP Worksheet No. 31.	Planned Project Assessment Table	73
QAPP Worksheet No. 32.	Assessment Findings and Response Actions	74
QAPP Worksheet No. 33.	QA Management Reports Table	75
QAPP Worksheet No. 34.	Sampling and Analysis Verification (Step I) Process Table	76
QAPP Worksheet No. 35.	Sampling and Analysis Validation (Steps IIa and IIb) Process Table	77
QAPP Worksheet No. 36.	Sampling and Analysis Validation (Steps IIa and IIb) Summary Table	78
QAPP Worksheet No. 37.	Data Usability Assessment	79

QAPP Worksheet No. 1

Title and Approval Page

Site Name/Project Name: **CRS Site**
Site Location: **CRS Site, Elyria, Lorain County, Ohio**

Title: **QAPP**
Revision Number: **1**
Revision Date: **4/4/12**
Page: **1** of **79**

Quality Assurance Project Plan for the Chemical Recovery Systems, Inc. Site Remedial Design Activities
Document Title

United States Environmental Protection Agency, Region 5
Lead Organization

Michael Watkins, Brown and Caldwell
Preparer's Name and Organizational Affiliation

6055 Rockside Woods Blvd., Suite 350, Independence, OH 44131, 216-606-1309 , mwatkins@brwncald.com

Preparer's Address, Telephone Number, and E-mail Address

20/09/10 Revised 04/04/12
Preparation Date (Day/Month/Year)

Brown and Caldwell Project Manager: _____
Signature

James A. Peeples, P.E., Brown and Caldwell

Brown and Caldwell Project QA Officer: _____
Signature

Michael L. Watkins, P.G., Brown and Caldwell

USEPA – Remedial Project Manager: _____
Signature

Michelle Kerr, USEPA

Other Approval Signatures: _____
Signature

_____
Printed Name/Title/Date

Document Control Number: _____

QAPP Worksheet No. 2

QAPP Identifying Information

Site Name/Project Name: Chemical Recovery Systems, Inc. Site (CRS Site)
Site Location: 142 Locust Street, Elyria, Lorain County, Ohio
Site Number/Code: USEPA facility identification number - OHD 057001810
Operable Unit: N/A
Contractor Name: Brown and Caldwell
Contractor Number: N/A
Contract Title: N/A
Work Assignment Number: N/A

1. ***Identify guidance used to prepare QAPP:***
   Uniform Federal Policy for Implementing Quality Systems (EPA-505-F-03-001, Uniform Federal Policy for Quality Assurance Project Plans, Parts 1, 2, and 3 (EPA-505-B-04-900A, B, and C.

2. ***Identify regulatory program****:* _CERCLA, Remedial Design/Remedial Action ___

3. ***Identify approval entity****:* United States Environmental Protection Agency, Region 5

4. ***Indicate whether the QAPP is a generic or a project-specific QAPP.*** **(circle one)**

5. ***List dates of scoping sessions that were held:*** _8/1/10 through 9/20/10

6. ***List dates and titles of QAPP documents written for previous site work, if applicable:***

   Title:
   Quality Assurance Project Plan, Revision II (Parsons, February 2003) ____

7. ***List organizational partners (stakeholders) and connection with lead organization:***
   - USEPA, Region 5
   - Ohio EPA
   - Chemical Recovery Solvents, Inc. RD/RA Settling Performing Defendants.

8. ***List data users:*** as listed above

9. ***If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below:***

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Cross-reference to Related Documents
Project Management and Objectives		
2.1 Title and Approval Page	- Title and Approval Page	Worksheet No. 1
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	- Table of Contents - QAPP Identifying Information	Page ii in QAPP Worksheet Nos. 1, 2
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	Worksheet No. 3 Worksheet No. 4
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table	Worksheet No. 5 Worksheet No. 6 Worksheet No. 7\ Worksheet No. 8
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History, and Background	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)	Worksheet No. 9 Worksheet No. 9 Worksheet No. 10,
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site-Specific PQOs - Measurement Performance Criteria Table	Worksheet No. 11 and Worksheet No. 12
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	Worksheet No. 13 Worksheet No. 13
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table	Worksheet No. 14 Worksheet No. 15 Worksheet No. 16

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Cross-reference to Related Documents
Measurement/Data Acquisition		
3.1 Sampling Tasks 3.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 3.1.2.1 Sampling Collection Procedures 3.1.2.2 Sample Containers, Volume, and Preservation 3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures 3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 3.1.2.5 Supply Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures	- Sampling Design and Rationale - Sample Location Map - Sampling Locations and Methods/ SOP Requirements Table - Analytical Methods/SOP Requirements Table - Field Quality Control Sample Summary Table - Sampling SOPs - Project Sampling SOP References Table - Field Equipment Calibration, Maintenance, Testing, and Inspection Table	Worksheet No. 17 Figures in the FSP, and RDWP Worksheet No. 18 Worksheet No. 23 Worksheet No. 20 Worksheet No. 18 Worksheet No. 23 Worksheet No. 20
3.2 Analytical Tasks 3.2.1 Analytical SOPs 3.2.2 Analytical Instrument Calibration Procedures 3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	- Analytical SOPs - Analytical SOP References Table - Analytical Instrument Calibration Table - Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Attachment A to QAPP Worksheet No. 23 Worksheet No. 24 Worksheet No. 25
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	- Sample Collection Documentation Handling, Tracking, and Custody SOPs - Sample Container Identification - Sample Handling Flow Diagram - Example Chain-of-Custody Form and Seal	Worksheet No. 26 and Worksheet Nos 26 and 27
3.4 Quality Control Samples 3.4.1 Sampling Quality Control Samples 3.4.2 Analytical Quality Control Samples	- QC Samples Table - Screening/Confirmatory Analysis Decision Tree	Worksheet No. 28 Worksheet No. 28 and

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Cross-reference to Related Documents
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	- Project Documents and Records Table - Analytical Services Table - Data Management SOPs	Worksheet No. 29 Worksheet No. 20
Assessment/Oversight		
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	- Assessments and Response Actions - Planned Project Assessments Table - Audit Checklists - Assessment Findings and Corrective Action Responses Table	 Worksheet No. 31 Worksheet No. 31 Worksheet No. 32
4.2 QA Management Reports	- QA Management Reports Table	Worksheet No. 33
4.3 Final Project Report		Worksheet No. 33
Data Review		
5.1 Overview		
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table - Usability Assessment	Worksheet No. 34 Worksheet No. 35 Worksheet No. 36 Worksheet No. 37
5.3 Streamlining Data Review 5.3.1 Data Review Steps To Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining		

Distribution List

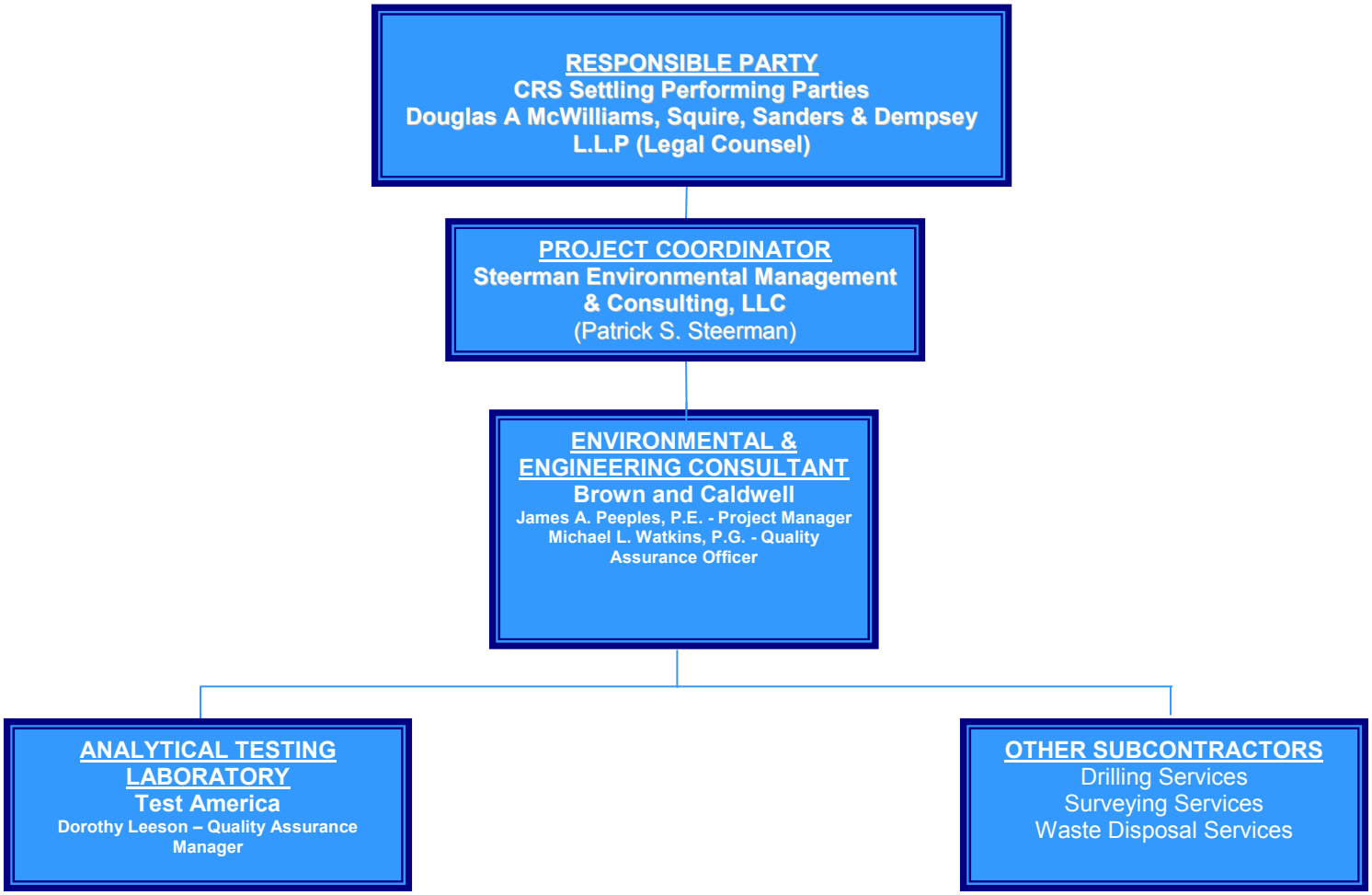
QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Michelle Kerr	Project Manager/ Coordinator	USEPA	312-886-8961	312-697-2658	Kerr.Michelle@epamail.epa.gov	
Lawrence J. Antonelli	Project Coordinator	OEPA	330-963-1127	330-487-0769	larry.antonelli@epa.state.oh.us	
James A. Peeples, P.E.	Project Manager	Brown and Caldwell	614-410-6144	614-410-3088	jpeeples@brwncald.com	
Michael L. Watkins, P.G.	QA Officer	Brown and Caldwell	216-606-1309	2166061350	mwatkins@brwncald.com	
Brown And Caldwell Field Team	Various	Brown and Caldwell	Various	Various	Various	
CRS Performing Parties Group	Various	Various	Various	Various	Various	
Patrick S. Steerman	PRP Group's Project Coordinator	Steerman Environmental Management & Consulting, LLC	404-329-9006	404-329-9006	psteerman@charter.net	
Dorothy Leeson	Quality Assurance Manger	TestAmerica Laboratories, Inc.	330-497-9396	330-497-0772	dorothy.leeson@testamericainc.com	

Project Personnel Sign-Off Sheet

Organization:_____

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read

Project Organizational Chart



Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Contact USEPA	Steerman Environmental Management & Consulting, LLC.	Patrick Steerman	404-329-9006	All written and most verbal from the consultants and contractors for the Group will be made through Mr. Steerman, unless otherwise directed by Mr. Steerman.
Point of Contact at USEPA	USEPA	Michelle Kerr	312-886-8961	Materials to be submitted to USEPA will be provided to the point of contact at USEPA.
Point of Contact at OEPA	OEPA	Lawrence J. Antonelli	330-963-1127	Materials to be submitted to OEPA will be provided to the point of contact at Ohio EPA.
Manage Project Phases	Brown and Caldwell	James Peeples	614-410-6144	Changes in scope and requests by USEPA or others will be provided by Patrick Steerman. Mr. Peeples will coordinate BC resources accordingly.
QAPP changes in the field	Brown And Caldwell Field Sampling Manager	Michael Watkins	216-606-1309	Field driven adjustments, or non-conformance, to the QAPP will be addressed through Mr. Watkins, and reported to Mr. Peeples.
Reporting Analytical Lab Quality Issues	TestAmerica	Alesia Danford	330-966-9783	Deviations in compliance with QA/QC will be reported to Mr. Peeples by Ms. Danford .
QAPP Amendments	Brown and Caldwell	Michael Watkins	216-606-1309	QAPP amendments, as necessary, will be initiated by Mr. Watkins, after authorized by Mr. Peeples and Mr. Steerman.

Personnel Responsibilities and Qualifications Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Michelle Kerr	Project Manager/ Coordinator	USEPA	USEPA Project Coordinator	
Lawrence J. Antonelli	Project Coordinator	OEPA	OEPA Project Coordinator	
Patrick S. Steerman	Project Coordinator	Steerman Environmental Management & Consulting, LLC	Project Coordinator	
James A. Peeples, P.E.	Project Manager, Supervising Engineer	Brown and Caldwell	Project Manager	B.S., Biology; M.S., Agricultural Engineering; 20 years experience
Michael L. Watkins, P.G.	Supervising Geologist	Brown and Caldwell	QA Manager, Field Team Leader	B.S., M.S., Geology; 24 years experience
Scott Blanchard, P.G.	Geologist III	Brown and Caldwell	Health and Safety Officer	B.S., M.S. Geology; 19 years experience
Dorothy Leeson	QA Officer	TestAmerica	QA Officer	B.S. Chemistry, Ohio University, 25 years experience
Alesia Danford	Project Manager -	TestAmerica	Project Management	26 years experience; coursework Kent State

Special Personnel Training Requirements Table

Project Function	Specialized Training – Title or Description of Course	Training Providers	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates[1]
HAZWOPER	40-hour initial training as per 29 CFR 1910.120	Various certified out-sourced providers	Various	Field personnel, Project Manager, and other key staff	Various BC staff and drilling subcontractor field staff	BC corporate office, Walnut Creek, CA and BC's Columbus, OH office
HAZWOPER	8-hour annual refresher as per 29 CFR 1910.120	Certified in-house and out-sourced providers	Various	Field personnel, Project Manager, and other key staff	Various BC staff and drilling subcontractor field staff	BC corporate office, Walnut Creek, CA and BC's Columbus, OH office
HAZWOPER	CPR and first aid training as per 29 CFR 1910.120	Out-sourced to certified trainers	Various	Site-Safety Officer on site during fieldwork	Various BC field staff	BC corporate office, Walnut Creek, CA and BC's Columbus, OH office

[1]If training records and/or certificates are on file elsewhere, document their location in this column. If training records and/or certificates do not exist or are not available, then this should be noted.

Project Scoping Session Participants Sheet

Project Name: Remedial Design Projected Date(s) of Sampling: 2010-2013 Project Manager: James Peeples	Site Name: CRS Site Site Location: Elyria, Lorain County, Ohio

Date of Session: 8/1/10 through 9/15/10
Scoping Session Purpose: Define DQOs, Content/Status of RDWP and Develop HASP

Name	Title	Affiliation	Phone No.	E-mail Address	Project Role
James Peeples	Project Manager/ Supervising Engineer	BC	614-410-6144	jpeeples@brwncald.com	Project Manager
James Krebs	Project Engineer III	BC	614-653-5474	jkrebs@brwncald.com	Field Team Leader, QAPP, FSP
Bryant Kiedrowski	Project Engineer III	BC	614-923-0853	bkiedrowski@brwncald.com	HASP

Consensus Decisions:

- RDWP/QAPP, other appendices etc. for work activities for design of the remedy for the Site to be performed as set forth in the CD, ROD, and, SOW.
- Health and Safety Plan to be developed - project specific to the CRS Site.
- Selected TestAmerica as the analytical lab.

Comments/Decisions:

- Add a "List of Acronyms" page at the front of each document.
- Standardize nomenclature in the RDWP/appendices

Action Items: Complete RDWP, Appendicies and associated documents for the Performing Parties review by September 15, 2010.

Problem Definition

DATA QUALITY OBJECTIVES (DQOs)

- The data quality objectives (DQOs) needs are established in the Remedial Design Work Plan to be submitted to the USEPA.

Project Quality Objectives/Systematic Planning Process Statements

Who will use the data?

- Data will be used by the CRS Performing Parties Group, its contractors, USEPA, and OEPA.

What will the data be used for?

- The data will be used to evaluate the aforementioned DQOs and to develop a RD that meets the requirements of the CD, SOW and ROD. The project Performance Standards are those described in the SOW;
- Analytical sensitivity must be adequate to detect and quantify the concentrations of each of the listed constituents to the concentrations of their respective action levels;
- The data will be used in the evaluation of Site impact extents, disposition of waste for disposal, documentation of contaminants left in place.

What types of data are required?

- Groundwater and surface water quality data are needed for the parameters, locations, and monitoring frequency as stated in the FSP.
- Groundwater/LNAPL/DNAPL elevation data are needed from the wells and at the frequency as stated in the FSP.
- Soil/waste quality data are needed for the parameters and locations described in the FSP.
- Waste characterization data are needed for the parameters and wastes described in the FSP.
- Survey data are needed for the locations described in the FSP.

How good do the data need to be?

- 90 percent of the data from a given sampling or measurement event will be useable.
- A primary Measurement Performance Criterion is adequate analytical sensitivity to meet the Performance Standards listed on Worksheet No. 15

How much data are needed?

- The spatial distribution of the data is specified in the FSP.
- The temporal distribution of the data is specified in the FSP.
- The specific analytical parameters to be tested are specified in the FSP.

Project Quality Objectives/Systematic Planning Process Statements

How will the data be collected?

- The aqueous media samples (e.g., groundwater, surface water, etc.) destined for chemical analysis will be collected using the procedures specified in the FSP.
- The specified aqueous media samples will be analyzed by TestAmerica following SW846 analytical methodology as described in the RDWP, FSP, Additional Groundwater Studies (AGWS) Work Plan and AGWS Supplemental Work Plan.
- Groundwater and LNAPL/DNAPL thickness and elevation data will be determined from depth to fluid field measurements using electronic water level meters and interface probes.
- The solid media samples (soil, waste, sediment, etc.) destined for chemical analysis will be collected using the procedures specified in the FSP.
- The specified solid and aqueous media samples will be analyzed by TestAmerica following SW846 analytical methodology as described in the RDWP, FSP,AGWS Work Plan and AGWS Supplemental Work Plan.
- Survey data will be obtained by a licensed Ohio surveyor in accordance with the required horizontal and vertical accuracy.

Who will generate the data?

- Qualified personnel from BC will collect the environmental media samples.
- Qualified personnel from BC will perform the liquid level measurements.
- Qualified personnel from the designated surveying company will conduct the horizontal and vertical survey of sampling locations.
- Qualified personnel from the designated drilling company will obtain the rock cores from the subsurface, then the cores will be transferred to qualified BC personnel for core inspection and preparation for additional testing and analysis.

How will the data be reported?

- Field data will be recorded and reported as specified in the FSP.
- Laboratory analytical data will be reported to BC in two formats:
    1. Laboratory summary report including sample results, a case-narrative, and a QA summary report.
    2. Electronic data deliverable (EDD) of the aforementioned in a BC-specified format.
- Survey data will be reported to BC in both hard copy and electronic format, and will include the following data: the northing coordinate, the easting coordinate, and the elevation (MSL-mean sea level).
- Data will be reported to USEPA as appendices to the Remedial Design Report, with the data summarized in tables contained within the body of the report. An electronic version of the complete report, in PDF format, will also be provided.

Project Quality Objectives/Systematic Planning Process Statements

How will the data be archived?

- Aforementioned electronic files will be maintained in their native formats on a server located in BC's Columbus, Ohio office.
- Tape backups of the files on the servers are made periodically which are stored at an off-site location for ten years.
- A paper copy of the complete Remedial Design Report will be maintained either at the BC office in Columbus, Ohio or an off-site document repository for a time period of 10 years following USEPA's approval of the work, as specified in the Consent Decree.

Measurement Performance Criteria Table

Matrix	Aqueous
Analytical Group	VOCs
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Groundwater and Surface Water Sampling	SW-846 8260 B	Precision/ Laboratory	RPD < 30% or the laboratory-specified recovery limits	Laboratory Duplicate	A
Groundwater and Surface Water Sampling	SW-846 8260 B	Precision/Sample Collection	RPD < 30% or the laboratory-specified recovery limits	Field Duplicate	S & A
Groundwater and Surface Water Sampling	SW-846 8260 B	Accuracy/Bias	RPD < 30% or the laboratory-specified recovery limits	MS/MSD	S & A
Groundwater and Surface Water Sampling	SW-846 8260 B	Accuracy/Bias	RPD < 30% or the laboratory-specified range	Laboratory Control Sample	A
Groundwater and Surface Water Sampling	SW-846 8260 B	Accuracy/Bias	Percent recovery within laboratory-specified recovery limits: (DBF 73-122%) (1,2-DCA-d4 61-128%) (Toluene-d8 76-110%) (4-BFB 74-116)	Surrogate	A
Groundwater and Surface Water Sampling	SW-846 8260 B	Accuracy/ Lab contamination	No Target Compounds>RL; no common lab contaminants >5XRL.	Method Blank	A
Groundwater and Surface Water Sampling	SW-846 8260 B	Accuracy/ Transport contamination	No Target Compounds>RL; no common lab contaminants >5XRL.	Trip Blank	A
Groundwater and Surface Water Sampling	SW-846 8260 B	Accuracy/ Field contamination	No Target Compounds>RL; no common lab contaminants >5XRL.	Field Blank	S & A

Measurement Performance Criteria Table

Matrix	Aqueous
Analytical Group	VOCs
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Groundwater and Surface Water Sampling	SW-846 8260 B	Accuracy/ Holding Time	< 14 days until analysis	Reported sample data	A
Groundwater and Surface Water Sampling	SW-846 8260 B	Completeness	>90% field samples >90% lab analyses	Reported sample data	S & A
Groundwater and Surface Water Sampling	SW-846 8260 B	Representativeness	RPD <30% for detected data, ND comparison	Field Duplicate	S & A
Groundwater and Surface Water Sampling	SW-846 8260 B	Sensitivity	QL ≤ Associated Performance Standards	Reported sample data	A

[1] Reference number from QAPP Worksheet No. 21.
[2] Reference number from QAPP Worksheet No. 23.

Measurement Performance Criteria Table

Matrix	Soil
Analytical Group	VOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Soil Sampling	SW-846 8260 B	Precision/Laboratory	RPD < 30% or the laboratory-specified recovery limits	Laboratory Duplicate	A
Soil Sampling	SW-846 8260 B	Precision/Sample Collection	RPD < 30% or the laboratory-specified recovery limits	Field Duplicate	S & A
Soil Sampling	SW-846 8260 B	Accuracy/Bias	RPD < 30% or the laboratory-specified recovery limits	MS/MSD	S & A
Soil Sampling	SW-846 8260 B	Accuracy/Bias	RPD < 30% or the laboratory-specified range	Laboratory Control Sample	A
Soil Sampling	SW-846 8260 B	Accuracy/Bias	Percent recovery within laboratory-specified recovery limits: (DBF 59-138%) (1,2-DCA-d4 61-130%) (Toluene-d8 60-143%) (4-BFB 47-158)	Surrogate	A
Soil Sampling	SW-846 8260 B	Accuracy/ Lab contamination	No Target Compounds>RL; no common lab contaminants >5XRL.	Method Blank	A
Soil Sampling	SW-846 8260 B	Accuracy/ Transport contamination	No Target Compounds>RL; no common lab contaminants >5XRL.	Trip Blank	A
Soil Sampling	SW-846 8260 B	Accuracy/ Field contamination	No Target Compounds>RL; no common lab contaminants >5XRL.	Field Blank	S & A
Soil Sampling	SW-846 8260 B	Accuracy/ Holding Time	< 14 days until analysis	Reported sample data	A
Soil Sampling	SW-846 8260 B	Completeness	>90% field samples >90% lab analyses	Reported sample data	S & A

Measurement Performance Criteria Table

Matrix	Soil
Analytical Group	VOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Soil Sampling	SW-846 8260 B	Representativeness	RPD <30% for detected data, ND comparison	Field Duplicate	S & A
Soil Sampling	SW-846 8260 B	Sensitivity	QL ≤ Associated Performance Standards	Reported sample data	A

[1] Reference number from QAPP Worksheet No. 21.
[2] Reference number from QAPP Worksheet No. 23.

Measurement Performance Criteria Table

Matrix	Aqueous
Analytical Group	SVOCs
Concentration Level	Standard

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Groundwater and Surface Water Sampling	SW-846 8270C	Precision/Laboratory	RPD <30%	Laboratory Duplicate	A
Groundwater and Surface Water Sampling	SW-846 8270C	Precision/Sample Collection	RPD <30%	Field Duplicate	S & A
Groundwater and Surface Water Sampling	SW-846 8270C	Accuracy/Bias	RPD < 30% or the laboratory-specified recovery limits	MS/MSD	S & A
Groundwater and Surface Water Sampling	SW-846 8270C	Accuracy/Bias	RPD < 30% or the laboratory-specified range	Laboratory Control Sample	A
Groundwater and Surface Water Sampling	SW-846 8270C	Accuracy/Bias	Percent recovery within laboratory-specified recovery limits: (2-FBP 28-110%) (2-FP 10-110%) (246-TBP 22-120%) (NB-d5 27-111%) (Phenol-d5 10-110% (TP-d14 37-119%)	Surrogate	A
Groundwater and Surface Water Sampling	SW-846 8270C	Accuracy/ Lab contamination	No Target Compounds>RL; no common lab contaminants >5xRL	Method Blank	A
Groundwater and Surface Water Sampling	SW-846 8270C	Accuracy/ Transport contamination	No Target Compounds>RL; no common lab contaminants >5xRL	Trip Blank	A
Groundwater and Surface Water Sampling	SW-846 8270C	Accuracy/ Field contamination	No Target Compounds>RL; no common lab contaminants >5xRL	Field Blank	S & A
Groundwater and Surface Water Sampling	SW-846 8270C	Accuracy/ Holding Time	< 14 days until extraction; 40 to analysis	Reported sample data	A
Groundwater and Surface Water Sampling	SW-846 8270C	Completeness	>90% field samples >90% lab analyses	Reported sample data	S & A

Measurement Performance Criteria Table

Matrix	Aqueous
Analytical Group	SVOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Groundwater and Surface Water Sampling	SW-846 8270C	Representativeness	RPD <30% for detected data, ND comparison	Field Duplicate	S & A
Groundwater and Surface Water Sampling	SW-846 8270C	Sensitivity	QL ≤ Associated Performance Standards	Reported sample data	A

Measurement Performance Criteria Table

Matrix	Soil
Analytical Group	SVOCs
Concentration Level	Standard

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Soil Sampling	SW-846 8270 C	Precision/Laboratory	RPD <30%	Laboratory Duplicate	A
Soil Sampling	SW-846 8270C	Precision/Sample Collection	RPD <30%	Field Duplicate	S & A
Soil Sampling	SW-846 8270C	Accuracy/Bias	RPD < 30% or the laboratory-specified recovery limits	MS/MSD	S & A
Soil Sampling	SW-846 8270C	Accuracy/Bias	RPD < 30% or the laboratory-specified range	Laboratory Control Sample	A
Soil Sampling	SW-846 8270C	Accuracy/Bias	Percent recovery within laboratory-specified recovery limits: (2-FBP 34-110%) (2-FP 26-110%) (246-TBP 10-118%) (NB-d5 24-225%) (Phenol-d5 28-110% (TP-d14 41-119%)	Surrogate	A
Soil Sampling	SW-846 8270C	Accuracy/ Lab contamination	No Target Compounds>RL; no common lab contaminants >5xRL	Method Blank	A
Soil Sampling	SW-846 8270C	Accuracy/ Transport contamination	No Target Compounds>RL; no common lab contaminants >5xRL	Trip Blank	A
Soil Sampling	SW-846 8270C	Accuracy/ Field contamination	No Target Compounds>RL; no common lab contaminants >5xRL	Field Blank	S & A
Soil Sampling	SW-846 8270C	Accuracy/ Holding Time	< 7 days until extraction; 40 to analysis	Reported sample data	A
Soil Sampling	SW-846 8270C	Completeness	>90% field samples >90% lab analyses	Reported sample data	S & A

Measurement Performance Criteria Table

Matrix	Soil
Analytical Group	SVOCs
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Soil Sampling	SW-846 8270C	Representativeness	RPD <30% for detected data, ND comparison	Field Duplicate	S & A
Soil Sampling	SW-846 8270C	Sensitivity	QL ≤ Associated Performance Standards	Reported sample data	A

Measurement Performance Criteria Table

Matrix	Aqueous
Analytical Group	PCBs
Concentration Level	Standard

Sampling Procedure[1]	Analytical Method/SOP [2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Groundwater and Surface Water Sampling	SW-846 8082	Precision/Laboratory	RPD < 30%	Laboratory Duplicate	A
Groundwater and Surface Water Sampling	SW-846 8082	Precision/Sample Collection	RPD < 30%	Field Duplicate	S & A
Groundwater and Surface Water Sampling	SW-846 8082	Accuracy/Bias	RPD < 30% or the laboratory-specified recovery limits	MS/MSD	S & A
Groundwater and Surface Water Sampling	SW-846 8082	Accuracy/Bias	RPD < 30% or the laboratory-specified range	Laboratory Control Sample	A
Groundwater and Surface Water Sampling	SW-846 8082	Accuracy/Bias	Percent recovery within laboratory-specified recovery limits:(DSBP 10-199%) (TCMX 10-196%)	Surrogate	A
Groundwater and Surface Water Sampling	SW-846 8082	Accuracy/ Lab contamination	No Target Compounds>RL	Method Blank	A
Groundwater and Surface Water Sampling	SW-846 8082	Accuracy/ Transport contamination	No Target Compounds>RL	Trip Blank	A
Groundwater and Surface Water Sampling	SW-846 8082	Accuracy/ Field contamination	No Target Compounds>RL	Field Blank	S & A
Groundwater and Surface Water Sampling	SW-846 8082	Accuracy/ Holding Time	< 7 days until extraction; 40 to analysis	Reported sample data	A
Groundwater and Surface Water Sampling	SW-846 8082	Completeness	>90% field samples >90% lab analyses	Reported sample data	S & A

Measurement Performance Criteria Table

Matrix	Aqueous
Analytical Group	PCBs
Concentration Level	Standard

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Groundwater Sampling	SW-846 8082	Representativeness	RPD <30% for detected data, ND comparison	Field Duplicate	S & A
Groundwater Sampling	SW-846 8082	Sensitivity	QL ≤ Associated Performance Standards	Reported sample data	A

Measurement Performance Criteria Table

Matrix	Soil
Analytical Group	PCBs
Concentration Level	Standard

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Soil Sampling	SW-846 8082	Precision/ Laboratory	RPD <30%	Laboratory Duplicate	A
Soil Sampling	SW-846 8082	Precision/Sample Collection	RPD <30%	Field Duplicate	S & A
Soil Sampling	SW-846 8082	Accuracy/Bias	RPD < 30% or the laboratory-specified recovery limits	MS/MSD	S & A
Soil Sampling	SW-846 8082	Accuracy/Bias	RPD < 30% or the laboratory-specified range	Laboratory Control Sample	A
Soil Sampling	SW-846 8082	Accuracy/Bias	Percent recovery within laboratory-specified recovery limits: (DSBP 10-127%) (TCMX 27 -130%)	Surrogate	A
Soil Sampling	SW-846 8082	Accuracy/ Lab contamination	No Target Compounds>RL	Method Blank	A
Soil Sampling	SW-846 8082B	Accuracy/ Transport contamination	No Target Compounds>RL	Trip Blank	A
Soil Sampling	SW-846 8082	Accuracy/ Field contamination	No Target Compounds>RL	Field Blank	S & A
Soil Sampling	SW-846 8082	Accuracy/ Holding Time	< 14 days until extraction; 40 to analysis	Reported sample data	A
Soil Sampling	SW-846 8082	Completeness	>90% field samples >90% lab analyses	Reported sample data	S & A

Measurement Performance Criteria Table

Matrix	Aqueous
Analytical Group	PCBs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Groundwater and Surface Water Sampling	SW-846 8082	Representativeness	RPD <30% for detected data, ND comparison	Field Duplicate	S & A
Groundwater and Surface Water Sampling	SW-846 8082	Sensitivity	QL ≤ Associated Performance Standards	Reported sample data	A

Measurement Performance Criteria Table

Matrix	Aqueous
Analytical Group	Metals
Concentration Level	Standard

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Ground/Surface Water Sampling	SW-846 6010B/ 7470A	Precision/ Laboratory	RPD <20%	Laboratory Duplicate	A
Ground/Surface Water Sampling	SW-846 6010B/ 7470A	Precision/Sample Collection	RPD <20%	Field Duplicate	S & A
Ground/Surface Water Sampling	SW-846 6010B/ 7470A	Accuracy/Bias	RPD < 20%	MS/MSD	S & A
Ground/Surface Water Sampling	SW-846 6010B/ 7470A	Accuracy/Bias	RPD < 20%	Laboratory Control Sample	A
Ground/Surface Water Sampling	SW-846 6010B/ 7470A	Accuracy/ Lab contamination	No target analytes > RL For common laboratory contaminants, no analytes >2x RL.. If MB is biased high and associated samples are below the RL or 20X the blank the data is accepted.	Method Blank	A
Ground/Surface Water Sampling	SW-846 6010B/ 7470A	Accuracy/ Transport contamination	No Target Compounds>RL	Trip Blank	A
Ground/Surface Water Sampling	SW-846 6010B/ 7470A	Accuracy/ Field contamination	No Target Compounds>RL	Field Blank	S & A
Ground/Surface Water Sampling	SW-846 6010B/ 7470A	Accuracy/ Holding Time	180 Days / Hg in 28 days	Reported sample data	A
Ground/Surface Water Sampling	SW-846 6010B/ 7470A	Completeness	>90% field samples >90% lab analyses	Reported sample data	S & A

Measurement Performance Criteria Table

Matrix	Aqueous
Analytical Group	Metals
Concentration Level	Standard

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Groundwater and Surface Water Sampling	SW-846 6010B/ 7470A	Representativeness	RPD <20% for detected data, ND comparison	Field Duplicate	S & A
Groundwater and Surface Water Sampling	SW-846 6010B/ 7470A	Sensitivity	QL ≤ Associated Performance Standards	Reported sample data	A

Measurement Performance Criteria Table

Matrix	Soil
Analytical Group	Metals
Concentration Level	Standard

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Soil Sampling	SW-846 6010B/ 7470A	Precision/Laboratory	RPD <20%	Laboratory Duplicate	A
Soil Sampling	SW-846 6010B/ 7470A	Precision/Sample Collection	RPD <20%	Field Duplicate	S & A
Soil Sampling	SW-846 6010B/ 7470A	Accuracy/Bias	RPD < 20%	MS/MSD	S & A
Soil Sampling	SW-846 6010B/ 7470A	Accuracy/Bias	RPD < 20%	Laboratory Control Sample	A
Soil Sampling	SW-846 6010B/ 7470A	Accuracy/ Lab contamination	No target analytes > RL For common laboratory contaminants, no analytes >2x RL.. If MB is biased high and associated samples are below the RL or 20X the blank the data is accepted.	Method Blank	A
Soil Sampling	SW-846 6010B/ 7470A	Accuracy/ Transport contamination	No Target Compounds>RL	Trip Blank	A
Soil Sampling	SW-846 6010B/ 7470A	Accuracy/ Field contamination	No Target Compounds>RL	Field Blank	S & A
Soil Sampling	SW-846 6010B/ 7470A	Accuracy/ Holding Time	180 Days / Hg in 28 days	Reported sample data	A
Soil Sampling	SW-846 6010B/ 7470A	Completeness	>90% field samples >90% lab analyses	Reported sample data	S & A

Measurement Performance Criteria Table

Matrix	Soil
Analytical Group	Metals
Concentration Level	Standard

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Soil Sampling	SW-846 6010B/ 7470A	Representativeness	RPD <20% for detected data, ND comparison	Field Duplicate	S & A
Soil Sampling	SW-846 6010B/ 7470A	Sensitivity	QL ≤ Associated Performance Standards	Reported sample data	A

Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/Collection Dates)	How Data Will Be Used	Limitations on Data Use
Analytical data contained in the 2006 RI/FS Report prepared by Parsons.	RI/FS Reports, Revision 3 (Parsons, 2006)	Parsons collected data from 2003 to 2005	Data were used to evaluate remedial alternatives for the Site.	Data are used as reference.

Summary of Project Tasks

Field Sampling Tasks:
- Liquid level measurements.
- Collection of groundwater samples.
- Collection of surface water samples.
- Collection of soil samples.

Analysis Tasks:
- Environmental media samples will be subjected to analytical testing as needed.

Quality Control Tasks:
- Collect field duplicates, trip blanks, field blanks, and MS/MSD samples as appropriate.

Secondary Data:
- See Worksheet No. 13

Data Management Tasks:
- BC will manage the analytical data in data spreadsheets and/or a relational database.

Documentation and Records:
- Records will be prepared while sampling in the form of field logbook entries and completion of field forms, as appropriate.
- Chain-of-custody forms will be maintained.
- Data documentation and reporting by the laboratories will be in accordance with the relevant ASTM methods, laboratory SOPs, and QA Manuals.

Data Deliverables:
- Analytical data will be provided as electronic data deliverable (EDD) and data summary reports completed by TestAmerica.

Field Data Review;
- Field data review records reviewed for completeness and accuracy within one week after sample collection.

Data Review Tasks:
- Verification of data completeness.
- Review of samples data reports and QA summary reports to verify compliance with the MPCs on Worksheet No. 12.

Summary of Project Tasks

Data Reporting:

- Paper copies of laboratory summary report and QA report.
- EDD for analytical data from TestAmerica.
- Summary tables and figures in reports to USEPA, as well as paper copy of the raw data.

Reference Limits and Evaluation Table

Matrix:	Soil
Analytical Group:	VOCs
Concentration Level:	Low

Analyte	CAS Number	Project Action Limit[1] (mg/kg)	Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
				MDL	RL
1,1,2,2-Tectrachlorethane	630-20-6	0.930	.005	0.00034	.005
1,1,2-Trichloroethane	79-00-5	1.6	.005	0.00039	.005
1,2 Dichloroethane	107-06-2	0.600	.005	0.00034	.005
1,2-Dichloropropane	78-87-5	0.740	.005	0.00069	.005
Benzene	71-43-2	1.3	.005	0.00023	.005
Bromodichloromethane	75-27-4	1.8	.005	0.00028	.005
Bromomethane	74-83-9	13	.005	0.00054	.005
Carbon Tetrachloride	56-23-5	0.550	.005	0.00037	.005
Chloroethane	75-00-3	6.5	.005	0.00086	.005
Chloroform	67-66-3	12	.005	0.00029	.005
Chloromethane	74-87-3	2.6	.005	0.00041	.005
Dibromochloromethane	124-48-1	2.6	.005	0.00055	.005
Ethylbenzene	100-41-4	20	.005	0.00026	.005
Methylene Chloride	75-09-2	21	.005	0.00067	.005
Tetrachloroethene (PCE)	127-18-4	3.4	.005	0.00052	.005
Trichloroethene (TCE)	79-01-6	0.110	.005	0.00042	.005
Vinyl Chloride	75-01-4	0.750	.005	0.00039	.005
Xylenes (total)	1330-207	420	.010	0.00067	.010

MDL = Minimum detection limit
RL = Reporting limit
1 = Based on Project Action Limits defined in the RI/FS and ROD

Reference Limits and Evaluation Table

Matrix:	Soil
Analytical Group:	SVOCs
Concentration Level:	Low

Analyte	CAS Number	Project Action Limit[1] (mg/kg)	Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
				MDL	RL
1,3-Dichlorobenzene	541-73-1	63	0.05	0.011	0.05
1,4-Dichlorobenzene	106-46-7	7.9	0.05	0.02	0.05
2,4,6-Trichlorophenol	88-06-2	62	0.15	0.08	0.15
2-Nitroaniline	88-74-4	18	0.2	0.0091	0.2
3,3-Dichlorobenzidine	91-94-1	3.8	0.1	0.018	0.1
Benzo (a) anthracene	56-55-3	2.1	0.0067	0.0033	0.0067
Benzo (a) pyrene	50-32-8	0.210	0.0067	0.0033	0.0067
Benzo (b) fluoranthene	205-99-2	2.1	0.0067	0.0033	0.0067
Benzo (k) fluoranthene	207-08-9	21	0.0067	0.0033	0.0067
Bis (2-Chloroethyl) ether	111-44-4	0.550	0.1	0.002	0.1
Bis (2-Ethylhexyl) phthalate	117-81-7	120	0.05	0.019	0.05
Dibenzo (a,h) anthracene	53-70-3	0.210	0.0067	0.0033	0.0067
Hexachlorobenzene	118-74-1	1.1	0.0067	0.0021	0.0067
Hexachlorobutadiene	87-68-3	22	0.05	0.027	0.05
Indeno (1,2,3-cd) pyrene	193-39-5	2.1	0.0067	0.0033	0.0067
N-Nitrsodi-n-propylamine	621-64-7	0.250	0.05	0.027	0.05
Pentachlorophenol	87-86-5	9	0.15	0.08	0.15

MDL = Minimum detection limit
RL = Reporting limit
1 = Based on Project Action Limits defined in the RI/FS and ROD

Reference Limits and Evaluation Table

Matrix:	Soil
Analytical Group:	TAL Metals
Concentration Level:	Low

Analyte	CAS Number	Project Action Limit[1] (mg/kg)	Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
				MDL	RL
Antimony	7440-36-0	410	1	0.39	1
Arsenic	7440-38-2	1.6	1	0.3	1
Iron	7439-89-6	100,000	10	4.9	10
Lead	7439-92-1	750	0.3	0.19	0.3

Matrix:	Soil
Analytical Group:	PCBs
Concentration Level:	Low

Analyte	CAS Number	Project Action Limit[1] (mg/kg)	Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
				MDL	RL
Aroclor-1221	11104-28-2	0.74	0.033	0.016	0.033
Aroclor-1232	11141-16-5	0.74	0.033	0.014	0.033
Aroclor-1242	53469-21-9	0.74	0.033	0.013	0.033
Aroclor-1248	12672-29-6	0.74	0.033	0.017	0.033
Aroclor-1254	11097-69-1	0.74	0.033	0.017	0.033
Aroclor-1260	11096-82-5	0.74	0.033	0.017	0.033

MDL = Minimum detection limit
RL = Reporting limit
1 = Based on Project Action Limits defined in the RI/FS and ROD

Reference Limits and Evaluation Table

Matrix:	Aqueous
Analytical Group:	VOCs
Concentration Level:	Low

Analyte	CAS Number	Project Action Limit[1] (µg/l)	Project Quantitation Limit (µg/l)	Achievable Laboratory Limits (µg/l)	
				MDL	RL
1,1,1-Trichlorethane	71-55-6	200	1	0.22	1
1,1,2,2-Tectrachlorethane	79-34-5	0.055	1	0.18	1
1,1,2-Trichloroethane	79-00-5	5	1	0.27	1
1,1-Dichloroethane	75-34-3	810	1	0.15	1
1,1-Dichloroethene	75-35-4	7	1	0.19	1
1,2 Dichloroethane	107-06-2	5	1	0.22	1
1,2-Dichloropropane	78-87-5	5	1	0.18	1
Acetone	67-64-1	610	10	1.1	10
Benzene	71-43-2	5	1	0.13	1
Bromodichloromethane	75-27-4	0.18	1	0.15	1
Bromoform	75-25-2	8.5	1	0.64	1
Bromomethane	74-83-9	8.7	1	0.41	1
Carbon Disulfide	75-15-0	1000	1	0.13	1
Carbon Tetrachloride	56-23-5	5	1	0.13	1
Chlorobenzene	108-90-7	100	1	0.15	1
Chloroethane	75-00-3	4.6	1	0.29	1
Chloroform	67-66-3	6.2	1	0.16	1
Chloromethane	74-87-3	1.5	1	0.3	1
cis-1,2-Dichloroethene	156-59-2	70	1	0.17	1
Dibromochloromethane	124-48-1	0.13	1	0.18	1
Ethylbenzene	100-41-4	700	1	0.17	1
Methylene Chloride	75-09-2	4.3	1	0.33	1
Styrene	100-42-5	1600	1	0.11	1
Tetrachloroethene (PCE)	127-18-4	5	1	0.29	1

Reference Limits and Evaluation Table

Matrix:	Aqueous
Analytical Group:	VOCs
Concentration Level:	Low

Analyte	CAS Number	Project Action Limit[1] (µg/l)	Project Quantitation Limit (µg/l)	Achievable Laboratory Limits (µg/l)	
				MDL	RL
Toluene	108-88-3	1000	1	0.13	1
Trans-1,2-Dichloroethene	156-60-5	100	1	0.19	1
Trichloroethene (TCE)	79-01-6	5	1	0.17	1
Vinyl Chloride	75-01-4	2	1	0.22	1
Xylenes (total)	1330-20-7	10000	2	0.28	2

MDL = Minimum detection limit
RL = Reporting Limit
1 = Based on the MCLs, SMCLs, and Risk-Based Preliminary Remediation Goals from the RI/FS and ROD

Reference Limits and Evaluation Table

Matrix:	Aqueous
Analytical Group:	SVOCs
Concentration Level:	Low

Analyte	CAS Number	Project Action Limit[1] (µg/l)	Project Quantitation Limit (µg/l)	Achievable Laboratory Limits (µg/l)	
				MDL	RL
1,2,4-Trichlorobenzene	120-82-1	70	1	0.24	1
1,2-Dichlorobenzene	95-50-1	370	1	0.29	1
1,3-Dichlorobenzene	541-73-1	5.5	1	0.8	1
1,4-Dichlorobenzene	106-46-7	0.5	1	0.34	1
2,4,6-Trichlorophenol	88-06-2	3.6	5	0.8	5
2,4-Dichlorophenol	120-83-2	110	2	0.8	2
2,4-Dimethylphenol	105-67-9	730	2	0.8	2
2,4-Dinitrophenol	51-28-5	73	5	2.4	5
2,4-Dinitrotoluene	121-14-2	73	5	0.27	5
2,6-Dinitrotoluene	606-20-2	36	5	0.8	5
2-Chloronaphthalene	91-58-7	490	1	0.1	1
2-Chlorophenol	95-57-8	30	1	0.29	1
2-Methylphenol	95-48-7	1800	1	0.8	1
2-Nitroaniline	88-74-4	1	2	0.8	2
3,3-Dichlorobenzidine	91-94-1	0.15	5	0.37	5
4-Chloroaniline	106-47-8	150	2	0.8	2
4-Methylphenol	8001-28-3	180	1	0.8	1
Acenaphthene	83-32-9	370	0.2	0.1	0.2
Benzo (a) anthracene	56-55-3	0.092	0.2	0.1	0.2
Benzo (a) pyrene	50-32-8	0.2	0.2	0.1	0.2
Benzo (b) fluoranthene	205-99-2	0.092	0.2	0.1	0.2
Benzo (k) fluoranthene	207-08-9	0.92	0.2	0.1	0.2
Bis (2-Chloroethyl) ether	111-44-4	0.0098	1	0.1	1

Reference Limits and Evaluation Table

Matrix:	Aqueous
Analytical Group:	SVOCs
Concentration Level:	Low

Analyte	CAS Number	Project Action Limit[1] (µg/l)	Project Quantitation Limit (µg/l)	Achievable Laboratory Limits (µg/l)	
				MDL	RL
Carbazole	86-74-8	3.4	1	0.28	1
Chrysene	218-01-9	9.2	0.2	0.1	0.2
Dibenzo (a,h) anthracene	53-70-3	0.0092	0.2	0.1	0.2
Dibenzofuran	132-64-9	24	1	0.1	1
Di-n-octyl phthalate	117-84-0	1500	1	0.8	1
Fluoranthene	206-44-0	1500	0.2	0.1	0.2
Fluorene	86-73-7	240	0.2	0.1	0.2
Hexachlorobenzene	118-74-1	1	0.2	0.1	0.2
Hexachlorobutadiene	87-68-3	0.86	1	0.27	1
Hexachlorocyclopentadiene	77-47-4	50	10	0.8	10
Hexachloroethane	67-72-1	4.8	1	0.8	1
Indeno (1,2,3-cd) pyrene	193-39-5	0.092	0.2	0.1	0.2
Isophorone	78-59-1	71	1	0.27	1
Naphthalene	91-20-3	6.2	0.2	0.1	0.2
Nitrobenzene	98-95-3	3.4	1	0.04	1
N-Nitrosodi-n-propylamine	621-64-7	0.0096	1	0.8	1
N-Nitrosodiphenylamine	86-30-6	14	1	0.31	1
Pyrene	129-00-0	180	0.2	0.1	0.2

MDL = Minimum detection limit
RL = Reporting limit
1 = Based on the MCLs, SMCLs, and Risk-Based Preliminary Remediation Goals from the RI/FS and ROD

Reference Limits and Evaluation Table

Matrix:	Aqueous
Analytical Group:	TAL Metals
Concentration Level:	Low

Analyte	CAS Number	Project Action Limit[1] (µg/l)	Project Quantitation Limit (µg/l)	Achievable Laboratory Limits (µg/l)	
				MDL	RL
Antimony – Dissolved	7440-39-0	6	6	1.8	6
Arsenic – Dissolved	7440-38-2	10	10	3.2	10
Cadmium – Dissolved	7440-43-9	5	2	.66	2
Iron – Dissolved	7439-89-6	11000	100	81	100
Lead – Dissolved	7439-92-1	15	3	1.9	3
Manganese – Dissolved	7439-96-5	880	15	0.41	15

MDL = Minimum detection limit
RL = Reporting limit
1 = Based on the MCLs, SMCLs, and Risk-Based Preliminary Remediation Goals from the RI/FS and ROD

Reference Limits and Evaluation Table

Matrix:	Aqueous
Analytical Group:	PCBs
Concentration Level:	Low

Analyte	CAS Number	Project Action Limit[1] (µg/l)	Project Quantitation Limit (µg/l)	Achievable Laboratory Limits (µg/l)	
				MDL	RL
Aroclor-1016	12674-11-2	0.5	0.5	0.17	0.5
Aroclor-1221	11104-28-2	0.5	0.5	0.13	0.5
Aroclor-1232	11141-16-5	0.5	0.5	0.16	0.5
Aroclor-1242	53469-21-9	0.5	0.5	0.22	0.5
Aroclor-1248	12672-29-6	0.5	0.5	0.1	0.5
Aroclor-1254	11097-69-1	0.5	0.5	0.16	0.5
Aroclor-1260	11096-82-5	0.5	0.5	0.17	0.5

MDL = Minimum detection limit
RL = Reporting limit
1 = Based on the MCLs, SMCLs, and Risk-Based Preliminary Remediation Goals from the RI/FS and ROD

Project Schedule/Timeline Table

Activities	Organization	Anticipated Date(s) of Initiation	Anticipated Date of Completion	Deliverable	Deliverable Due Date
Liquid Level Monitoring	Brown and Caldwell	To be determined	To be determined	Summary table Remedial Design Report	As per the CD and SOW
Soil,Groundwater, and Surface Water Sampling	Brown and Caldwell	To be determined	To be determined	To be determined	Defined in the RDWP

Sampling Design and Rationale

The objectives of the pre-design activities include the following:

1. To further define the limits of the soil removal area, if needed;
2. To obtain groundwater quality data from more areas of the site as defined in the SOW.

Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ID Number	Matrix	Depth (feet)	Analytical Group	Concentration Level	Frequency	Sampling SOP Reference	Rationale for Sampling Location
MW-A through MW-F	GW	TBD	VOC, SVOC, Metals	Low	Per Well	SOP #2 (FSP)	Well placement per SOW
MW-6R	GW	TBD	VOC, SVOC, Metals	Low	Depth Series	FSP and SOP #2	Well placement per SOW
Soil Removal Area	Soil	TBD	VOC	Low	TBD	see FSP	Perimeter of soil removal area
Surface Water; various locations	SW	TBD	VOC, SVOC	Low	TBD	See AGWS-S Work Plan and FSP	Along shoreline of Site

TBD – To be determined based on specific boring/well, SOW requirements, and field judgment.

Analytical SOP Requirements Table

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Container (number, size, and type)	Sample Volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Aqueous	TCL VOCs	SW-846 5030B, 8260B / NC-MS-019	(3) 40 mL vials		pH <2 with HCl, Cool to 4°C	14 days
Aqueous	TCL SVOCs	SW-846 5030B, 8270C / NC-MS-018 / NC-OP-32	(2) 1 liter ambers		Cool to 4°C	7 days extract/ 40 days analysis
Aqueous	PCBs	SW-846 8082 / NC-GC-038	(2) 1 liter ambers		Cool to 4°C	7 days extract/ 40 days analysis
Aqueous	TAL Metals	SW-846 3010A, 6010B/7470A / NC-MT-012 / NC-MT-013 / NCIP011	1 liter plastic	500 mLs	Cool to 4°C, pH <2 with HNO_3	180 days; 28 days for Hg
Soil	TCL VOCs	SW-846 5030B, 8260B / NC-MS-019	Encores / Terracores / jars		Cool to 4°C	14 days
Soil	TCL SVOCs	SW-846 5030B, 8270C / NC-MS-018 / NC-OP-32	4 oz jar		Cool to 4°C	14 days to extract/ 40 days analysis
Soil	PCBs	SW-846 8082 / NC-GC-038	4 oz jar		Cool to 4°C	14 days to extract/ 40 days analysis
Soil	TAL Metals	SW-846 3010A, 6010B/7470A / NC-MT-011 / NC-MT-013 / NCIP010	4 oz jar		Cool to 4°C	180 days; 28 days for Hg

Field Quality Control Sample Summary Table

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Container (number, size, and type)	Sample Volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)	Matrix
Aqueous	TCL VOCs	SW-846 5030B, 8260B / NC-MS-019	(3) 40 mL vials		pH <2 with HCl, Cool to 4°C	14 days	Groundwater
Aqueous	TCL SVOCs	SW-846 5030B, 8270C / NC-MS-018 / NC-OP-32	(2) 1 liter ambers		Cool to 4°C	7 days extract/ 40 days analysis	Groundwater
Aqueous	PCBs	SW-846 8082 / NC-GC-038	(2) 1 liter ambers		Cool to 4°C	7 days extract/ 40 days analysis	Groundwater
Aqueous	TAL Metals	SW-846 3010A, 6010B/7470A / NC-MT-012 / NC-MT-013 / NCIP011	1 liter plastic	500 mLs	Cool to 4°C, pH <2 with HNO_3	180 days; 28 days for Hg	Groundwater
Soil	TCL VOCs	SW-846 5030B, 8260B / NC-MS-019	Encores / Terracores / jars		Cool to 4°C	14 days	Soil
Soil	TCL SVOCs	SW-846 5030B, 8270C / NC-MS-018 / NC-OP-32	4 oz jar		Cool to 4°C	14 days to extract/ 40 days analysis	Soil
Soil	PCBs	SW-846 8082 / NC-GC-038	4 oz jar		Cool to 4°C	14 days to extract/ 40 days analysis	Soil
Soil	TAL Metals	SW-846 3010A, 6010B/7470A / NC-MT-011 / NC-MT-013 / NCIP010	4 oz jar		Cool to 4°C	180 days; 28 days for Hg	Soil

Field Quality Control Sample Summary Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference	No. of Sampling Locations	No. of Field Duplicate Pairs (1)	No. of MS (1)	No. of Field Blanks (1)	No. of Equip. Blanks (1)	No. of Trip Blank Samples (1)	Total No. of Samples to Lab
Aqueous and non-aqueous environmental samples	VOCs, SVOCS, PCBs	Inclusive	TestAmerica SOP Nos. NCMS018, NCOP32, NCMS019, NCGC038	TBD	5% of environmental samples per matrix	5% of environmental samples per matrix	5% of environmental samples per matrix	5% of environmental samples per matrix	One per shipment with VOC samples	TBD
Aqueous and non-aqueous environmental samples	Metals	Inclusive	TestAmerica SOP Nos. NCMT012, NCMT013, NCMT011, NCIP011, NCIP010	TBD	5% of environmental samples per matrix	5% of environmental samples per matrix	5% of environmental samples per matrix	5% of environmental samples per matrix	5% of environmental samples per matrix	TBD
Solid	TCLP extraction	Inclusive	TestAmerica SOP No. NCOP033	TBD	NA	NA	NA	NA	NA	TBD

1. – Minimum of one sample per matrix per sample delivery group
TBD – To be determined
NA – Not applicable

Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments

Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Photo-ionization Detector	Internal and against known standard	As per Owner's Manual	As per Owner's Manual	As per Owner's Manual	Daily	As per Owner's Manual	As per Owner's Manual	Field Team	Owner's Manual
Multi-Gas Meter	Internal and against known standard	As per Owner's Manual	As per Owner's Manual	As per Owner's Manual	Daily	As per Owner's Manual	As per Owner's Manual	Field Team	Owner's Manual
Multi-Parameter Groundwater Field Instrument	Against known standard	As per Owner's Manual	As per Owner's Manual	As per Owner's Manual	Daily	As per Owner's Manual	As per Owner's Manual	Field Team	Owner's Manual
Water Level Meter	None	As per Owner's Manual	As per Owner's Manual	As per Owner's Manual	NA	As per Owner's Manual	As per Owner's Manual	Field Team	Owner's Manual
Interface Probe	None	As per Owner's Manual	As per Owner's Manual	As per Owner's Manual	NA	As per Owner's Manual	As per Owner's Manual	Field Team	Owner's Manual

NA = Not applicable

Analytical SOP References Table

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
NC-MS-018	Revision 1, 12/16/2008 GC/MS Analysis based on Method 8270C	Definitive	Semi-Volatiles	GC/MS	TestAmerica	N
NC-OP-32	Revision 1.0, 01/07/2009 Extraction of Organic Compounds from Waters and Soils , based on SW846 3500 Series and 600 Series Methods	NA	Organic Prep	NA	TestAmerica	N
NC-OP-033	Revision 0, 03/31/2008 Toxicity Characteristic Leaching Procedure and Synthetic Precipitation Leaching Procedure	NA	TCLP extraction	NA	TestAmerica	N
NC-MS-019	Revision 10, 01/07/2009 Determination of Volatile Organics by GC/MS based on Methods 8260B and 8260A.	Definitive	Volatiles	GC/MS	TestAmerica	N
NC-MT-012	Revision 1, 01/07/2009 Inductively Coupled Plasma – Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis	Definitive	Metals	ICP	TestAmerica	N
NC-GC-038	Revision 1, 01/15/2009 Gas Chromatographic Analysis Based on Methods 8000B, 8021B, 8081A, 8082, 8151A, 615 and 8015B.	Definitive	Pests, Herbs, PCBs	GC	TestAmerica	N

Analytical SOP References Table

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?1 (Y/N)
NC-MT-013	Revision 0, 01/07/2009 Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW846 7470A and MCAWW 245.1	Definitive	Mercury	CVAA	TestAmerica	N
NCMT011	Rev 1, 01/07/2009 Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy	Definitive	Mercury	CVAA	TestAmerica	No
NCIP011	Rev 0, 01/07/2009 Acid Digestion for Aqueous Samples	Definitive	Metals Prep	NA	TestAmerica	No
NCIP010	Rev 1, 01/07/2009 Acid Digestion for Soil Samples	Definitive	Metals Prep	NA	TestAmerica	No

NA – Not applicable

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS	Check of mass spectral ion intensities (tuning procedure) using BFB (8260B) and DFTPP (8270C)	Prior to initial calibration and calibration verification (every 12 hours)	Must meet the method requirements before samples are analyzed.	Retune instrument and verify the tune acceptability	QA Manager/Group Leader/Analyst	NC-MS-018, NC-MS-019
	Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit	Initial calibration prior to sample analysis	8260B: The minimum average SPCC RF for Chloromethane, 1,1-Dichloroethane, Bromoform is 0.1; for Chlorobenzene and 1,1,2,2-Tetrachloroethane is 0.3 8270C: The minimum average SPCC RF is 0.050. 8260B and 8270C: RSD is less than or equal to 15% or R2 > 0.99 for target analytes, and is less than or equal to 30% for CCC	Correct problem, then repeat initial calibration	QA Manager/Group Leader/Analyst [b]	NC-MS-018, NC-MS-019
	Second-source calibration verification	Once per five-point initial calibration	Less than 20% for CCC compounds, less than or equal to 50% for non-CCC compounds. Allowance for 6 compounds above 50%	Correct problem, then repeat second source verification. If it still fails, then repeat initial calibration	QA Manager/Group Leader/Analyst [b]	NC-MS-018, NC-MS-019

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS	Daily calibration verification	Before sample analysis and at beginning of every 12 hours of analysis time .	8260B: The minimum average SPCC RF for Chloromethane, 1,1-Dichloroethane, Bromoform is 0.1; for Chlorobenzene and 1,1,2,2-Tetrachloroethane is 0.3 8270C: The minimum average SPCC RF is 0.050. 8260B and 8270C[a]: The percent drift/difference for RF is less than or equal to 20% for CCC analytes and Gasoline.	Correct problem, then repeat CCV. If still fails, repeat initial calibration	QA Manager/Group Leader/Analyst [b]	NC-MS-018, NC-MS-019
Metals (ICP)	Internal standards	During acquisition of calibration standard	Areas within -50% to +100% of last ICAL mid-point for each CCV	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	Lab Manager / Analyst [b]	NC-MS-018, NC-MS-019
Metals (ICP)	Initial calibration per manufacturer's instructions, with a minimum of one standard and a calibration blank	Initial calibration prior to sample analysis	Accepted if the initial calibration verification (ICV) passes	Correct problem, then repeat initial calibration .	Group Leader / Analyst	NC-MT-012

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	Low concentration standard at or near the reporting limit	After one point calibration	Advisory limits of +/-50	Evaluate run based on advisory limits.	Group Leader / Analyst	NC-MT-012
	Second-source (ICV) calibration verification	Once per initial calibration	Within 10%	Correct problem, then repeat. If still fails, repeat initial calibration	Group Leader / Analyst	NC-MT-012
Metals ICPMS	Continuing calibration verification (CCV)	After every 10 samples and the end of the sequence .	Within 10%	Correct problem, then repeat. If still fails, repeat initial calibration. If CCV is biased high and associated samples are below the RL the data can be reported	Group Leader / Analyst	NC-MT-012
	Initial calibration per manufacturer's instructions, with a minimum of one standard and a calibration blank	Initial calibration prior to sample analysis	Correlation coefficient >0.995 (if more than one point); accepted if the initial calibration verification (ICV) passes	Correct problem, then repeat initial calibration	Group Leader / Analyst	NC-MT-002
	Second-source (ICV) calibration verification	Once per initial calibration	Within 10%	Correct problem, then repeat. If still fails, repeat initial calibration	Group Leader / Analyst	NC-MT-002
Metals ICPMS	Continuing calibration verification (CCV)	After every 10 samples and the end of the sequence .	Within 10%	Correct problem, then repeat. If still fails, repeat initial calibration If CCV is biased high and associated samples are below the RL the data can be reported.	Group Leader / Analyst	NC-MT-012

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Metals (CVAA)	Initial calibration (IC) per manufacturer's instructions, with a minimum of five standards and a calibration blank	Initial calibration prior to sample analysis	Correlation coefficient >0.995; accepted if the initial calibration verification (ICV) passes	Correct problem, then repeat initial calibration	Group Leader / Analyst	NC-MT-011 NC-MT-013
	Second-source ICV, prepared at the calibration midpoint	Once per initial calibration	Within 10%	Correct problem, then repeat. If still fails, repeat initial calibration	Group Leader / Analyst	NC-MT-011 NC-MT-013
PCBs (GC)	Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit	Initial calibration prior to sample analysis	A minimum of 5 points for % RSD. Must pass <20%. Quadratic requires 6 points passing by a minimum Coefficient of 0.990 Linear must pass by a coefficient of,0.990.	Correct problem, then repeat initial calibration	QA Manager/Group Leader/Analyst [b]	NC-GC-038
PCBs (GC)	Second-source calibration verification	Once per five-point initial calibration	ICV Must pass <15% for TCL compounds. Non TCL compounds must pass by ,30%	Correct problem, then repeat second source verification. If it still fails, then repeat initial calibration	QA Manager/Group Leader/Analyst [b]	NC-GC-038

Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[2]	SOP Reference
GC	Change septum, clean injection port, change or clip column, install new liner	Detector signals and chromatogram review	Instrument performance and sensitivity	As needed	CCV passes criteria	Reinspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Test America Chemist	NC-GC-038
GC-MS	Clean sources, maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year, other maintenance as needed	Tune and CCV pass criteria	Recalibrate instrument	Test America Chemist	NC-MS-018, NC-MS-019
GC-MS	Change septum, clean injection port, change or clip column, install new liner, change trap	Response factors and chromatogram review	Instrument performance and sensitivity	As needed	Tune and CCV pass criteria	Reinspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Test America Chemist	NC-MS-018, NC-MS-019
ICP	Replace disposables, flush lines, clean injector and torch	Intensity of 1PPM Manganese STD within criteria	Check connections	Daily or as needed	Intensity of 1PPM Manganese STD within criteria	Replace, investigate injector, reanalyze	Test America Chemist	NC-MT-002
ICP	Replace pump windings	Monitor ISTD counts for variation	Instrument performance and sensitivity	As needed	Monitor ISTD counts for variation	Replace windings, recalibrate and reanalyze	Test America Chemist	NC-MT-002
CVAA	Replace disposables, flush lines	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Test America Chemist	NC-MT-013
ICPMS	Change tubing	Monitor Indium intensity counts	Instrument performance and	As needed	Pass performance report	Replace tubing, perform	Test America Chemist	NC-MT-002

Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[2]	SOP Reference
			sensitivity			performance report		
ICPMS	Clean cones	Monitor Indium intensity counts	Instrument performance and sensitivity	As needed	Pass performance report	Clean, replace perform performance report	Test America Chemist	NC-MT-002

Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Lead Field Geologist/BC Support Field Staff, Brown and Caldwell
Sample Packaging (Personnel/Organization): Lead Field Geologist/BC Support Field Staff, Brown and Caldwell
Coordination of Shipment (Personnel/Organization): Lead Field Geologist/BC Support Field Staff, Brown and Caldwell
Type of Shipment/Carrier: Hand delivery, laboratory courier and/or FedEx
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Receiving counter of analytical laboratories as per QA Manual (Attachment A)
Sample Custody and Storage (Personnel/Organization): Analytical laboratories as per QA Manual (Attachment A)
Sample Preparation (Personnel/Organization): Aanalytical laboratories as per QA Manual (Attachment A) and test method SOP/ASTM standard
Sample Determinative Analysis (Personnel/Organization): Analytical laboratories as per QA Manual (Attachment A) and test method SOP/ASTM std.
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Analytical laboratories as per QA Manual (Attachment A) and method SOP
Sample Extract/Digest Storage (No. of days from extraction/digestion): Analytical laboratory as per QA Manual (Attachment A) and method SOP
Biological Sample Storage (No. of days from sample collection): Analytical laboratory as per QA Manual (Attachment A) and method SOP
SAMPLE DISPOSAL
Personnel/Organization: Analytical laboratories as per QA Manual (Attachment A) and method SOP
Number of Days from Analysis: Analytical laboratories as per QA Manual (Attachment A) and method SOP

Sample Custody Requirements

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):

Each environmental sample will be properly identified and individually labeled. Labels will be filled out in indelible ink with at least the following information: BC sample identification, date and time of sample collection, initials of sampler(s), analysis/test required, and preservation (as appropriate). The sample label will be securely attached to the sample container.

Environmental samples being analyzed by the analytical laboratory will be properly packaged and shipped for analysis. Following labeling, the sample bottles will be sealed with custody seals, and enclosed in clear sealable plastic bags (e.g., Ziploc® bags), through which the identifying label is visible.

The sample containers, sealed in bags, will then be placed in a hard plastic cooler, an effort will be made to fill in voids with either ice or packaging material (e.g. bubble wrap). Samples are to be packed with sufficient ice (enclosed in double-bagged sealable plastic bags) to cool the samples to 4±2°C. Additionally, each iced cooler will be packed with a cooler temperature blank. Lastly, the cooler will be filled with adequate cushioning material to minimize the possibility of container breakage.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):

Laboratory custody procedures are described in the attached analytical laboratory QA Manual in Attachments A.

Sample Identification Procedures:

The method of identification of a sample depends on the type of measurement or analysis performed. When field screening measurements (e.g., pH, dissolved oxygen, or turbidity) are made, data will be recorded directly in logbooks or on field investigation forms. Identifying information such as project name, station number, station location, date and time, name of sampler, field observations, remarks, or other pertinent information will be recorded.

Samples collected for laboratory analysis during the field investigation will be specifically designated by BC personnel for unique identification. Each sample will be designated by an alpha-numeric code which will identify the sampling location, type or as necessary depth.

Sample Custody Requirements

The three codes that make up the sample identifier are described as follows:

1. The Site name – CRS (Chemical Recovery Systems, Inc. Site);
2. The medium/sample type codes are listed below:

   MW - Groundwater sample taken from a monitoring well;
   SW – Surface water sample;
   SP – Seep sample;
   SED – Sediment sample;
   S – Soil sample; **DW** – Drummed waste sample;
   W – Waste sample;
   SB – Soil boring sample;
   TB – Trip Blank;
   FB – Field Blank sample;
   MS/MSD – Matrix spike/matrix spike duplicate sample;
   DUP – Duplicate sample; and

3. The location code will be keyed to the specific sample designation.

The following is an example of a sample identifier that will be used for samples collected for laboratory analysis: **CRS-SB-10A-2-4:** indicating that the soil sample was collected from the first boring at soil boring location at CRS-SB-10 from a depth of 2 to 4 feet below ground surface.

Sample Custody Requirements

Chain-of-custody Procedures:

A completed COC form must be included with all sample transfers/shipments.

When analytical samples are being shipped by an overnight delivery service to the laboratory, the COC form and any other paperwork will be checked against the sample labels and field documentation, and then placed in a waterproof sealable plastic bag and taped securely to the inside lid of the cooler. The cooler must then be secured, with custody seals affixed over the lid opening in at least two locations, and the cooler wrapped with strapping tape (without obscuring the custody seals). Orientation "this end up" arrows will be drawn or attached on two sides of the cooler, and a completed overnight delivery service shipping label will be attached to the top or handle of the cooler. Wide, clear tape should be used to secure the label to the lid of the cooler to prevent the shipping address label from being accidentally peeled off the cooler top.

Samples to be shipped by an overnight delivery service will be shipped within 24 hours of sample collection and arrive at the laboratory within 24 hours of sample shipment. A member of the field team will contact the laboratory directly, to notify them of a sample shipment, and again the next day to confirm receipt. In addition, an electronic version of the COCs will be sent to the laboratory either electronically or by facsimile.

If the laboratory is going to courier the samples directly from the site or the samples are being delivered directly to the laboratory by BC, the COC form will not be placed inside the cooler. The COC form must be signed by the receiver (e.g., the laboratory courier, or the laboratory sample custodian) when he/she accepts possession of the samples.

QC Samples Table

Matrix	Water/Soil
Analytical Group	GC semi-volatiles
Analytical Method / SOP Reference	EPA 8082

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>RL; no common lab contaminants >RL.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >20x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>RL; no common lab contaminants >RL.
Instrument Blank	Not required for PCB analysis	.				
Laboratory Control Sample	LCS if prepping an MS/MSD LCS/LCSD if no MS/MSD	Refer to Attachment B for LCS control limits.	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
Matrix Spike/Matrix Spike Duplicate	1/Batch (20 samples)	Refer to Attachment B for MS control limits.	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits
Surrogates	Every sample	Refer to Attachment B for surrogate control limits.	Check calculations and instrument performance; recalculate, reanalyze.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits

QC Samples Table

Matrix	Water/Soil
Analytical Group	GC Semi
Analytical Method / SOP Reference	8081/8151

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>RL; no common lab contaminants >RL.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >20x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>RL; no common lab contaminants >RL.
Laboratory Control Sample	1/Batch (20 samples) LCSD required if no MS/MSD	Refer to Attachment B for LCS control limits.	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
Matrix Spike/Matrix Spike Duplicate	1/Batch (20 samples)	Refer to Attachment B for MS control limits.	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits
Surrogates	Every sample	Refer to Attachment B for surrogate control limits.	Check calculations and instrument performance; recalculate, reanalyze.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits

QC Samples Table

Matrix	Water/Soil
Analytical Group	ICP Metals
Analytical Method / SOP Reference	EPA 6010 / NC-MT-012

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Calibration blank (CCB)	After CCV, after every 10 samples, and at the end of the sequence	No target analytes > RL If CCB is biased high and associated samples are below the RL the data can be reported. .	Correct the problem and reanalyze the blank and previous samples associated with CCB	Group Leader / Analyst	Accuarcy	No target analytes > 2x MDL
MDL study	After initial setup and and once per 12-months; otherwise quarterly MDL verification checks shall be performed.	MDLs will be below the RLs	.	Group Leader / Analyst	Sensitivity	MDLs will be below the RLs
Method blank	One per digestion batch	No target analytes > RL For common laboratory contaminants, no analytes >2x RL.. If MB is biased high and associated samples are below the RL or 20X the blank the data is accepted.	Re-analyze. Correct problem, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank	Group Leader / Analyst	Accuracy/Bias Contamination	No target analytes > ½ RL
Interference check solution (ICS)	At the beginning of an analytical run	**ICSA:** For non-interfering elements with RL's less than 10 , must fall withing 2x the RL from zero. For RL's >10 must fall within 1x RL from zero. If not evaluate run based on lab SOP	Terminate analysis, correct problem, then reanalyze ICS and all affected samples	Group Leader / Analyst	Accuracy	Within ±20% of expected value

QC Samples Table

Matrix	Water/Soil					
		ICSAB Within ±20% of expected value.expected value.				
MS	One MS per preparation batch	QC acceptance criteria: 80% to 120% accuracy, 20% precision; or laboratory statistically derived control limits	Flag data with N	Group Leader / Analyst	Accuracy	QC acceptance criteria: 80% to 120% accuracy, 20% precision or laboratory statistically derived control limits
MSD or sample duplicate	One per preparation batch	RPD < or equal to 20%	Flag data with N	Group Leader / Analyst	Precision	RPD < or equal to 20%
LCS	One LCS per each preparation batch	Laboratory statistically derived control limits : 80% to 120% accuracy. Or in house limits If the LCS is biased high and the associated samples are ND the data can be reported.,	Terminate analysis, identify and correct the problem, then re-prepare and reanalyze all affected samples and QC	Group Leader / Analyst	Accuracy	QC acceptance criteria: 80% to 120% accuracy, 20% precision or laboratory statistically derived control limits
Dilution test	Each preparation batch or when a new sample matrix is incountered.	1:5 dilution must agree within ±10% of the original determination	Flag date with an E	Group Leader / Analyst	Accuracy	10% Difference

QC Samples Table

Matrix	Water / Soil
Analytical Group	Semi Volatile Organics
Analytical Method / SOP Reference	EPA 8270C/ NC-MS-018,

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>RL; no common lab contaminants >RL.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >20x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>RL; no common lab contaminants >RL.
Laboratory Control Sample	1/Batch(20samples)_ LCSD if no MS/MSD	Refer to Attachment B for LCS control limits.	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
Matrix Spike/Matrix Spike Duplicate	1/Batch (20 samples)	Refer to Attachment B for MS control limits.	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits
Surrogates	Every sample	Refer to Attachment B for surrogate control limits.	Check calculations and instrument performance; recalculate, reanalyze.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits

QC Samples Table

Matrix	Water/Soil
Analytical Group	Volatile Organics
Analytical Method / SOP Reference	EPA 8260B/ NC-MS-019, Rev 10

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>RL; no common lab contaminants >5XRL.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >20x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>RL; no common lab contaminants >RL.
Instrument Blank	Once per 12 hours if method blank is not run	No Target Compounds>RL; no common lab contaminants >5XRL.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >20x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>1/2RL; no common lab contaminants >RL.
Laboratory Control Sample	1/Batch (20 samples)	Refer to Attachment B for LCS control limits.	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
Matrix Spike/Matrix Spike Duplicate	1/Batch (20 samples)	Refer to Attachment B for MS control limits.	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits
Surrogates	Every sample	Refer to Attachment B for surrogate control limits.	Check calculations and instrument performance; recalculate, reanalyze.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits

Project Documents and Records Table

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
• Field logbooks • COC forms • Field sample forms	• COC forms • Logbooks (instrument calibration, trouble-shooting, sample log-in) • Field sample forms	• Sample receipt logs • Internal and external COC forms • Equipment maintenance logs • Corrective action documentation	• Field inspection checklist(s) • Data review documentation • Review forms for electronic entry of data into database • Corrective action documentation	• Preliminary Design Report • Final Design Report

Analytical Services Table

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Water / Soil	VOCs	CRS Site	8260B	14 Calendar days	TestAmerica North Canton, 4101 Shuffel Street, NW, North Canton, Ohio 44720 Contact: Alesia Danford Phone: (330) 497-0772	NA
Water / Soil	Metals	CRS Site	6010B / 6020 / 7470 / 7471	14 Calendar days	TestAmerica North Canton, 4101 Shuffel Street, NW, North Canton, Ohio 44720 Contact: Alesia Danford Phone: (330) 497-0772	NA
Water / Soil	PCBs	CRS Site	PCB	14 Calendar Days	TestAmerica North Canton, 4101 Shuffel Street, NW, North Canton, Ohio 44720 Contact: Alesia Danford Phone: (330) 497-0772	NA
Water / Soil	SVOCs	CRS Site	8270C	14 Calendar Days	TestAmerica North Canton, 4101 Shuffel Street, NW, North Canton, Ohio 44720 Contact: Alesia Danford Phone: (330) 497-0772	NA

NA – Not applicable

Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Data review	Upon Receipt of all data	Internal	TestAmerica	Dorothy Leeson, QA Manager for TestAmerica	Senior Chemist for affected laboratory procedure - TestAmerica	Opal Davis-Johnson, Laboratory Manager, TestAmerica	Dorothy Leeson, QA Manager, TestAmerica
Data review	Upon Receipt of Data	External	BC	Michael Watkins, P.G.., QA Officer, BC	James Peeples, P.E., Project Manager, BC	Michael Watkins, P.G., QA Officer, BC	Michael Watkins, P.G., QA Officer, BC

Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, Org.)	Timeframe for Response
Review field records	Sample collection methods will yield unusable result	Patrick Steerman (SEMC), CRS Settling Performing Parties, Michelle Kerr (USEPA RPM)	14 days after finding	In writing via e-mail	Patrick Steerman (SEMC), CRS Settling Performing Parties, Michelle Kerr (USEPA RPM)	As indicated in CA response
Review field records	Scheduled sample not collected	Patrick Steerman (SEMC), CRS Settling Performing Parties, Michelle Kerr (USEPA RPM)	14 days after finding	In writing via e-mail	Patrick Steerman (SEMC), CRS Settling Performing Parties, Michelle Kerr (USEPA RPM)	As indicated in CA response
Qualitative laboratory data review	Inappropriate analytical/testing methods yield unusable result	Patrick Steerman (SEMC), CRS Settling Performing Parties, Michelle Kerr (USEPA RPM)	14 days after finding	In writing via e-mail	Patrick Steerman (SEMC), CRS Settling Performing Parties, Michelle Kerr (USEPA RPM)	As indicated in CA response

QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Preliminary Remedial Design	Draft and Final	As per the CD, SOW, and RD Work Plan	James Peeples P.E., Project Manager, BC	Michelle Kerr (USEPA) Lawrence Antonelli (OEPA) Patrick Steerman (SEMC) CRS Settling Performing Parties
Final Remedial Design	Draft and Final	As per the CD, SOW, and RD Work Plan	James Peeples P.E., Project Manager, BC	Michelle Kerr (USEPA) Lawrence Antonelli (OEPA) Patrick Steerman (SEMC) CRS Settling Performing Parties

Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Field logbook entries	Field notes will be reviewed for completeness.	I	Michael Watkins, BC
Chains-of-custody	COC forms will be reviewed for completeness.	I	Michael Watkins, BC
Laboratory analytical/testing data package	Data packages will be reviewed for completeness.	I	Michael Watkins, BC
Laboratory QA/QC data package	Data packages will be reviewed for completeness.	I	Michael Watkins, BC
Database	Database will be reviewed for completeness and technical accuracy.	I	Michael Watkins, BC

Sampling and Analysis Validation (Steps IIa and IIb) Process Table

Step IIa/Iib	Validation Input	Description	Responsible for Validation (Name, Organization)
Iia	SOPs	Verify that sampling methods/procedures were followed, and that deviations were noted/approved.	Michael Watkins, BC
Iib	SOPs	Determine potential impacts from noted/approved deviations, in regard to PQOs.	Michael Watkins, BC
Iia	Chains of custody	Examine COC forms against QAPP and laboratory contract requirements (e.g., analytical methods, sample identification, etc.).	Michael Watkins, BC
Iia	Laboratory data package	Examine packages against QAPP and laboratory contract requirements, and against COC forms (e.g., holding times, sample handling, analytical methods, sample identification, data qualifiers, QC samples, etc.).	Michael Watkins, BC
Iib	Laboratory data package	Determine potential impacts from noted/approved deviations, in regard to PQOs. Examples include PQLs and QC sample limits (precision/accuracy).	Michael Watkins, BC
Iib	Field and lab duplicates	Compare results of field duplicate (or replicate) analyses with RPD criteria.	Michael Watkins, BC

Step IIa – Compliance with methods, procedures, and contracts
Step IIb – Compliance with QAPP quality objectives

Sampling and Analysis Validation (Steps IIa and IIb) Summary Table

Step IIa/Iib	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa / Iib	Inclusive	Inclusive	Inclusive	Qualitative data review according to Worksheets No. 11, 12, 19, 28	Michael Watkins, BC

Step IIa – Compliance with methods, procedures, and contracts
Step IIb – Compliance with QAPP quality objectives

Evaluation Procedure:

- Liquid level measurement results will be reviewed to determine the presence/absence of LNAPL/DNAPL.
- Analytical results will be evaluated with respect to QA assessment and their ability to satisfy project objectives.

Personnel responsible for performing the usability assessment:

- Michael Watkins, P.G., Quality Assurance Officer, BC.

Usability Assessment Documentation:

- Laboratory summary reports
- Laboratory QA reports
- Brief narrative describing data review and data usability will be presented in the Remedial Design Report

ATTACHMENT A

Cover Page:

Quality Assurance Manual

TestAmerica North Canton
4101 Shuffel Street NW
North Canton, OH 44720
Phone: 330-497-9396
Fax: 330-497-0772

www.testamericainc.com

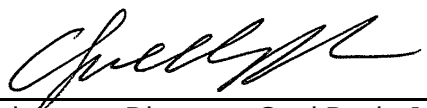
Copyright Information:
This documentation has been prepared by TestAmerica Analytical Testing Corp. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:
©COPYRIGHT 2010 TESTAMERICA ANALYTICAL TESTING CORP. ALL RIGHTS RESERVED

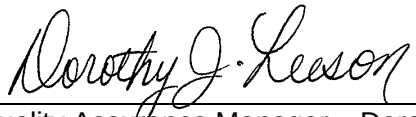
Facility Distribution No. _____ **Distributed To:_____**

Title Page:

Quality Assurance Manual
Approval Signatures



Laboratory Director – Opal Davis-Johnson — Date: 07/21/10



Quality Assurance Manager – Dorothy Leeson — Date: 07/21/10

Technical Director – Dr. Mark Bruce — Date: 07/15/10

SECTION 2

TABLE OF CONTENTS

Section No.	Title	ISO/IEC 17025:2005 Reference	Page No.	Effective Date
-	COVER PAGE		COVER	08/01/10
1.0	TITLE PAGE		1-1	08/01/10
2.0	TABLE OF CONTENTS		2-1	08/01/10
3.0	INTRODUCTION		3-1	08/01/10
3.1	Introduction And Compliance References	4.1.2; 4.2.4	3-1	08/01/10
3.2	Terms And Definitions	4.2.4	3-1	08/01/10
3.3	Scope / Fields Of Testing	4.1.2; 4.2.4	3-2	08/01/10
3.4	Management Of The Manual	4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	3-2	08/01/10
4.0	ORGANIZATION AND MANAGEMENT *(NELAC 5.4.1)*		4-1	08/01/10
4.1	Overview	4.1.1; 4.1.3; 4.1.5; 4.2.Z2	4-1	08/01/10
4.2	Roles And Responsibilities	4.1.3; 4.1.5; 4.1.Z1; 4.1.6; 4.2.1; 4.2.Z2; 4.2.6; 5.2.4	4-1	08/01/10
4.3	Deputies	4.1.5; 4.2.Z2	4-2	08/01/10
5.0	QUALITY SYSTEM *(NELAC 5.4.2)*		5-1	08/01/10
5.1	Quality Policy Statement	4.1.5; 4.2.2; 4.2.3	5-1	08/01/10
5.2	Ethics and Data Integrity	4.1.5; 4.2.2	5-1	08/01/10
5.3	Quality System Supportnig Documentation	4.2.2; 4.2.5	5-2	08/01/10
5.4	QA/QC Objectives for the Measurement of Data	4.1.5; 4.2.2	5-3	08/01/10
5.5	Criteria for Quality Indicators		5-5	08/01/10
5.6	Statistical Quality Control		5-5	08/01/10
5.7	Quality System Metrics		5-6	08/01/10
6.0	DOCUMENT CONTROL *(NELAC 5.4.3)*	4.2.7; 4.3.1; 4.3.2.2; 4.3.3.3; 4.3.3.4	6-1	08/01/10
6.1	Overview		6-1	08/01/10
6.2	Document Approval And Issue	4.3.2.1; 4.3.2.2; 4.3.2.3; 4.3.3.1	6-1	08/01/10
6.3	Procedures For Document Control Policy	4.3.2.1; 4.3.2.2; 4.3.3.1	6-2	08/01/10
6.4	Obsolete Documents	4.3.2.1; 4.3.2.2	6-2	08/01/10
7.0	SERVICE TO THE CLIENT *(NELAC 5.4.7*	4.4.1; 4.4.2; 4.4.3; 4.4.4	7-1	08/01/10
7.1	Overview	4.4.5; 5.7.1	7-1	08/01/10
7.2	Review Sequence And Key Personnel	4.4.5	7-2	08/01/10
7.3	Documentation	5.7.1	7-3	08/01/10
7.4	Special Services	4.7.1; 4.7.2	7-4	08/01/10
7.5	Client Communication	4.7.1; 4.7.2	7-4	08/01/10
7.6	Reporting	4.7.1; 4.7.2	7-5	08/01/10
7.7	Client Surveys	4.7.1; 4.7.2	7-5	08/01/10
8.0	SUBCONTRACTING OF TESTS *(NELAC 5.4.5)*	4.7.1; 4.7.2	8-1	08/01/10

Section No.	Title	ISO/IEC 17025:2005 Reference	Page No.	Effective Date
8.1	Overview	4.4.3; 4.5.4	8-1	08/01/10
8.2	Qualifying and Monitoring Subcontractors	4.5.1; 4.5.2; 4.5.3; 5.3.1	8-2	08/01/10
8.3	Oversight and Reporting	4.5.1; 4.5.2; 4.5.3	8-3	08/01/10
8.4	Contingency Planning		8-4	08/01/10
9.0	PURCHASING SERVICES AND SUPPLIES *(NELAC 5.4.6)*		9-1	08/01/10
9.1	Overview	4.6.1	9-1	08/01/10
9.2	Glassware	4.6.2; 4.6.3; 4.6.4	9-1	08/01/10
9.3	Reagents, Standards, and Supplies		9-1	08/01/10
9.4	Purchase of Equipment / Instruments / Software	4.6.2; 4.6.3; 4.6.4	9-3	08/01/10
9.5	Services		9-4	08/01/10
9.6	Suppliers		9-4	08/01/10
10.0	COMPLAINTS *(NELAC 5.4.8)*		10-1	08/01/10
10.1	Overview	4.8	10-1	08/01/10
10.2	External Complaints		10-1	08/01/10
10.3	Internal Complaints		10-2	08/01/10
10.4	Management Review		10-2	08/01/10
11.0	CONTROL OF NON-CONFORMING WORK *(NELAC 5.4.9)*	4.9.1; 5.10.Z.10	11-1	08/01/10
11.1	Overview	4.9.1; 4.11.3; 4.11.5	11-1	08/01/10
11.2	Responsibilities and Authorities	4.9.1; 4.11.3; 4.11.5	11-1	08/01/10
11.3	Evaluations of Significance and Action Taken	4.9.1; 4.11.3; 4.11.5	11-2	08/01/10
11.4	Prevention of NonConforming Work	4.9.2; 4.11.2	11-2	08/01/10
11.5	Method Suspension / Restriction (Stop Work Procedure)	4.9.1; 4.9.2; 4.11.5	11-2	08/01/10
12.0	CORRECTIVE ACTION *(NELAC 5.4.10)*		12-1	08/01/10
12.1	Overview	4.9.2; 4.11.1; 4.11.2	12-1	08/01/10
12.2	General	4.11.2; 4.11.3	12-1	08/01/10
12.3	Closed Loop Corrective Action Process	4.11.2; 4.11.3; 4.11.4; 4.12.2	12-1	08/01/10
12.4	Technical Corrective Actions		12-3	08/01/10
12.5	Basic Corrections	4.11.1; 4.13.2.3	12-4	08/01/10
13.0	PREVENTIVE ACTION *(NELAC 5.4.11)*	4.10; 4.12.1; 4.12.2	13-1	08/01/10
13.1	Overview	4.10; 4.12.1;	13-1	08/01/10
13.2	Management of Change		13-2	08/01/10
14.0	CONTROL OF RECORDS *(NELAC 5.4.12)*		14-1	08/01/10
14.1	Overview	4.2.7; 4.13.1.1	14-1	08/01/10
14.2	Technical and Analytical Records	4.13.1.1; 4.13.1.2; 4.13.1.3; 4.13.1.4; 4.13.2.1; 4.13.2.2; 4.13.2.3	14-5	08/01/10
14.3	Laboratory Support Activities	4.13.2.2; 4.13.2.3	14-6	08/01/10

Section No.	Title	ISO/IEC 17025:2005 Reference	Page No.	Effective Date
14.4	Administrative Records		14-6	08/01/10
14.5	Records Management, Storage, and Disposal		14-6	08/01/10
15.0	AUDITS *(NELAC 5.4.13)*		15-1	08/01/10
15.1	Internal Audits	4.14.1; 4.14.2; 4.14.3; 5.9.1; 5.9.A.15	15-1	08/01/10
15.2	External Audits	4.14.2; 4.14.3; 4.14.4	15-3	08/01/10
15.3	Audit Findings		15-3	08/01/10
16.0	MANAGEMENT REVIEWS *(NELAC 5.4.14)*	4.1.6; 4.15.1; 4.15.2	16-1	08/01/10
16.1	Quality Assurance Report		16-1	08/01/10
16.2	Annual Management Review	4.2.2	16-1	08/01/10
16.3	Potential Integrity Related Managerial Reviews		16-2	08/01/10
17.0	PERSONNEL *(NELAC 5.5.2)*	5.2.1	17-1	08/01/10
17.1	Overview	5.2.2; 5.2.3; 5.2.5	17-1	08/01/10
17.2	Education and Experience Requirements for Technical Personnel	5.2.1; 5.2.3; 5.2.4	17-1	08/01/10
17.3	Training	5.2.5	17-3	08/01/10
17.4	Data Integrity and Ethics Training Program		17-4	08/01/10
18.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS *(NELAC 5.5.3)*		18-1	08/01/10
18.1	Overview	5.3.1; 5.3.3; 5.3.4; 5.3.5	18-1	08/01/10
18.2	Environment	5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	18-1	08/01/10
18.3	Work Areas	5.3.3; 5.3.4; 5.3.5	18-2	08/01/10
18.4	Floor Plan		18-2	08/01/10
18.5	Building Security	5.3.4	18-2	08/01/10
19.0	TEST METHODS AND METHOD VALIDATION *(NELAC 5.5.4)*	5.4.1	19-1	08/01/10
19.1	Overview	5.4.1; 5.4.5.1	19-1	08/01/10
19.2	Standard Operating Procedures (SOPs)	4.3.3.1; 5.4.2	19-1	08/01/10
19.3	Laboratory Methods Manual		19-1	08/01/10
19.4	Selection of Methods	5.4.1; 5.4.2; 5.4.3; 5.4.4; 5.4.5.1; 5.4.5.2; 5.4.5.3	19-2	08/01/10
19.5	Laboratory Developed Methods and Non-Standard Methods	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3; 5.4.Z.3	19-16	08/01/10
19.6	Validation of Methods	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3; 5.4.Z.3	19-16	08/01/10
19.7	Method Detection Limits (MDL) / Limits of Detection (LOD)	5.4.Z.3	19-18	08/01/10
19.8	Instrument Detection Limits (IDL)		19-19	08/01/10
19.9	Verification of Detection and Reporting Limits		19-19	08/01/10
19.10	Retention Time Windows		19-19	08/01/10
19.11	Evaluation of Selectivity		19-20	08/01/10

Section No.	Title	ISO/IEC 17025:2005 Reference	Page No.	Effective Date
19.12	Estimation of Uncertainty of Measurement	5.1.1; 5.1.2; 5.4.6.1; 5.4.6.2; 5.4.6.3; 5.4.Z.4	19-20	08/01/10
19.13	Control of Data	5.4.7.1; 5.4.7.2; 5.9.1;	19-21	08/01/10
20.0	EQUIPMENT (AND CALIBRATIONS) *(NELAC 5.5.5)*	5.5.4; 5.5.5; 5.5.Z.5; 5.5.6; 5.5.Z.6	20-1	08/01/10
20.1	Overview	5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10; 5.6.1; 5.6.Z.8	20-1	08/01/10
20.2	Preventive Maintenance	5.5.1; 5.5.3; 5.5.7; 5.5.9; 5.6.1; 5.6.Z.8	20-1	08/01/10
20.3	Support Equipment	5.5.10; 5.5.11; 5.6.2.1.2; 5.6.2.2.1; 5.6.2. 5.5.8; 5.5.Z.6; 5.5.10; 5.6.1; 5.6.Z.8; 5.6.3.12.2	20-2	08/01/10
20.4	Instrument Calibrations		20-5	08/01/10
20.5	Tentatively Identified Compounds (TICs) – GC/MS Analysis		20-7	08/01/10
20.6	GC/MS Tuning		20-8	08/01/10
21.0	MEASUREMENT TRACEABILITY *(NELAC 5.5.6)*		21-1	08/01/10
21.1	Overview	5.6.2.1.2; 5.6.2.2.2; 5.6.3.1	21-1	08/01/10
21.2	NIST-Traceable Weights and Thermometers	5.6.3.1; 5.6.3.2	21-1	08/01/10
21.3	Reference Standards / Materials	5.6.3.1; 5.6.3.2; 5.6.3.3; 5.6.3.4; 5.9.1	21-1	08/01/10
21.4	Documentation and Labeling of Standards, Reagents, and Reference Materials		21-2	08/01/10
22.0	SAMPLING *(NELAC 5.5.7)*		22-1	08/01/10
22.1	Overview	5.7.1; 5.7.3	22-1	08/01/10
22.2	Sampling Containers		22-1	08/01/10
22.3	Definition of Holding Time		22-1	08/01/10
22.4	Sampling Containers		22-2	08/01/10
22.5	Sample Aliquots / Subsampling	5.7.1	22-2	08/01/10
23.0	HANDLING OF SAMPLES *(NELAC 5.5.8)*	5.8.1	23-1	08/01/10
23.1	Chain of Custody (COC)	5.7.2; 5.8.4; 5.9.1	23-1	08/01/10
23.2	Sample Receipt	5.8.2; 5.8.3	23-2	08/01/10
23.3	Sample Acceptance Policy		23-5	08/01/10
23.4	Sample Storage	5.8.4	23-5	08/01/10
23.5	Hazardous Samples and Foreign Soils		23-5	08/01/10
23.6	Sample Shipping	5.8.2	23-5	08/01/10
23.7	Sample Disposal		23-5	08/01/10
24.0	ASSURING THE QUALITY OF TEST RESULTS *(NELAC 5.5.9)*		24-1	08/01/10
24.1	Overview	5.9.2	24-1	08/01/10
24.2	Controls	5.9.2	24-1	08/01/10

Section No.	Title	ISO/IEC 17025:2005 Reference	Page No.	Effective Date
24.3	Negative Controls	5.9.2	24-1	08/01/10
24.4	Positive Controls	5.9.2	24-2	08/01/10
24.5	Sample Matrix Controls	5.9.2	24-4	08/01/10
24.6	Acceptance Criteria (Control Limits)		24-4	08/01/10
24.7	Additional Procedures to Assure Quality Control		24-7	08/01/10
25.0	REPORTING RESULTS *(NELAC 5.5.10)*		25-1	08/01/10
25.1	Overview	5.10.1; 5.10.2; 5.10.8	25-1	08/01/10
25.2	Test Reports	5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8	25-1	08/01/10
25.3	Negative Controls	5.10.1; 5.10.7; 5.10.8	25-3	08/01/10
25.4	Supplemental Information for Test	5.10.1; 5.10.3.1; 5.10.5	25-4	08/01/10
25.5	Environmental Testing Obtained from Subcontractors	5.10.1; 5.10.6	25-4	08/01/10
25.6	Client Confidentiality	4.1.5; 5.10.7	25-5	08/01/10
25.7	Format of Reports	5.10.8	25-5	08/01/10
25.8	Amendments to Test Reports	5.10.9; 5.10.Z.10	25-5	08/01/10
25.9	Policies on Client Requests for Amendments	5.9.1; 5.10.Z.10	25-6	08/01/10

LIST OF TABLES

Table No.	Title	ISO/IEC 17025:2005 Reference	Page	Effective Date
12-1	General Corrective Action Procedures	4.11.2; 4.13.2.3	12-5	08/01/10
14-1	Records Index	4.13.1.1	14-1	08/01/10
14-2	Special Record Retention Requirements		14-3	08/01/10
15-1	Types of Internal Audits and Frequency	4.14.1	15-1	08/01/10
19-1	Wet Chemistry Methods	5.5.4; 5.5.5	19-3	08/01/10
19-2	Methods for Mercury by Cold Vapor Atomic Absorption		19-7	08/01/10
19-3	Methods for Mercury by Cold Vapor Atomic Fluororescence		19-7	08/01/10
19-4	Methods for Metals by ICP and ICPMS		19-7	08/01/10
19-5	Metals Sample Preparation Methods		19-11	08/01/10
19-6	Organic Sample Preparation Methods		19-12	08/01/10
19-7	Organic Method of Analysis		19-14	08/01/10
20-1	Laboratory Equipment & Instrumentation		20-9	08/01/10
20-2	Schedule of Routine Maintenance		20-13	08/01/10
20-3	Preventive Maintenance Procedures		20-23	08/01/10
22-1	Inorganic Sample Containers, Preservatives, and Holding Times		22-3	08/01/10
22-2	Organic Sample Containers, Preservatives, and Holding Times		22-13	08/01/10
22-3	Sample Containers, Preservatives, and Holding Times for TCLP and SPLP		22-23	08/01/10
24-1	Example - Negative Controls		24-1	08/01/10
24-2	Sample Matrix Control		24-4	08/01/10

LIST OF FIGURES

Figure No.	Title	ISO/IEC 17025:2005 Reference	Page	Effective Date
4-1	Corporate and Laboratory Organizational Charts	4.1.3; 4.1.5; 4.2.Z2	4-3	08/01/10
8-1	Example - Client-Approved Subcontractor Form		8-5	08/01/10
19-1	Example – Demonstration of Capability Documentation		19-27	08/01/10
19-2	Work Flow		19-28	08/01/10
23-1	Example: Chain of Custody (COC)		23-7	08/01/10
23-2	Example: Custody Seals		23-8	08/01/10
23-3	Example: Internal Chain of Custody (COC)		23-9	08/01/10
23-4	Example: Cooler Receipt Form	5.8.3	23-10	08/01/10

LIST OF APPENDICES

Appendix No.	Title	Page	Effective Date
1	Laboratory Floor Plan	Appendix 1-1	08/01/10
2	Laboratory Method Listing	Appendix 2-1	08/01/10
3	Glossary/Acronyms	Appendix 3-1	08/01/10
4	Laboratory Certifications, Accreditations, Validations	Appendix 4-1	08/01/10

SOPs AND POLICIES REFERRED TO IN THE QA MANUAL

SOP/Policy Reference	Title
CA-C-S-001	Work Sharing Process
CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-003	Management of Change Procedure
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CA-Q-S-008	Management Systems Review
CA-T-P-001	Qualified Products List
CW-E-M-001	Corporate Environmental Health & Safety Manual
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-Q-M-002	Corporate Quality Management Plan
NC-QA-015	Equipment Monitoring and Thermometer Calibration
NC-QA-018	Statistical Evaluation of Data and Development of Control Charts
NC-QA-019	Records Information Management
NC-QA-027	Preparation and Management of Standard Operating Procedures
NC-QA-028	Employee Orientation and Training
NC-QA-029	Nonconformance and Corrective Action System
NC-SC-005	Sample Receiving and Sample Control
NC-SC-006	Sample Procurement Protocol
CA-Q-T-005	Laboratory Documentation
NC-QA-021	Evaluation of Method Detection Limits for Chemical Tests

SECTION 3

INTRODUCTION
(NELAC 5.1 - 5.3)

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica North Canton's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Guide 17025 (2005). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plant, CA-Q-M-002, (CQMP) and the various accreditation and certification programs listed in Appendix 7 (3). The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations. The relevant NELAC section is included in the heading of each QAM section.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- U.S. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.1, April 2009.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Final Version 3, January 2006.
- Toxic Substances Control Act (TSCA).

3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 6 (2) for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among effluent water, groundwater, hazardous waste, sludge, wipes, and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical process, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 4. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory must abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director/Manager , the Quality Assurance (QA) Manager, and the Technical Director. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

- Specific requirements delineated in project plans may supersede general quality requirements described in this manual. Ohio VAP requirements are listed throughout the document.

3.4 MANAGEMENT OF THE MANUAL

3.4.1 Review Process

This manual is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager must review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates must be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control and Updating Procedures (refer to SOP NC-QA-27 Preparation and Management of SOPs).

SECTION 4

ORGANIZATION AND MANAGEMENT
(NELAC 5.4.1)

4.1 OVERVIEW

TestAmerica North Canton is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities, and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, Corporate Quality Assurance, Corporate Quality Director, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate and TestAmerica North Canton is presented in Figure 4-1 Employee names are provided to demonstrate range and size of departments however the actual staff members may vary over time. The most current Organization Chart may be obtained from Quality Assurance Manager or Laboratory Director. .

4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program. More extensive job descriptions are maintained by laboratory management.

4.2.1 Quality Assurance Program

The responsibility for quality lies with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for knowing the content of this manual and upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica North Canton laboratory.

North Canton Laboratory Key Personnel

Name	Position
Rusty Vicinie	General Manager, NE Region
Opal Davis-Johnson	Laboratory Director
Raymond Risden	Operations Manager
Dorothy Leeson	Quality Assurance Manager
Mark Bruce	Technical Director

Rebecca Strait	Client Services Director
Steve Jackson	Regional Safety Director, Waste Management Supervisor
Tammy Tokos	Reporting Group Leader
Olguita Colon	General Chemistry Group Leader
Will Cordell	Field Analytical Group Leader
Steve Earle	Extractions Group Leader
Al Haidet	Shipping Group Leader
Dave Heakin	GC Volatiles Group Leader
Tom Hula	GC/MS Semivolatiles Group Leader
John McFadden	Sample Control Group Leader
Darren Miller	Maintenance Group Leader
Roger Toth	Metals Group Leader
Carolynne Roach	GC Semivolatiles Group Leader
Tom Stiller	GC/MS Volatiles Group Leader
Patrick O'Meara	Project Management Group Leader

4.2.2 Quality Assurance (QA) Manager

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system

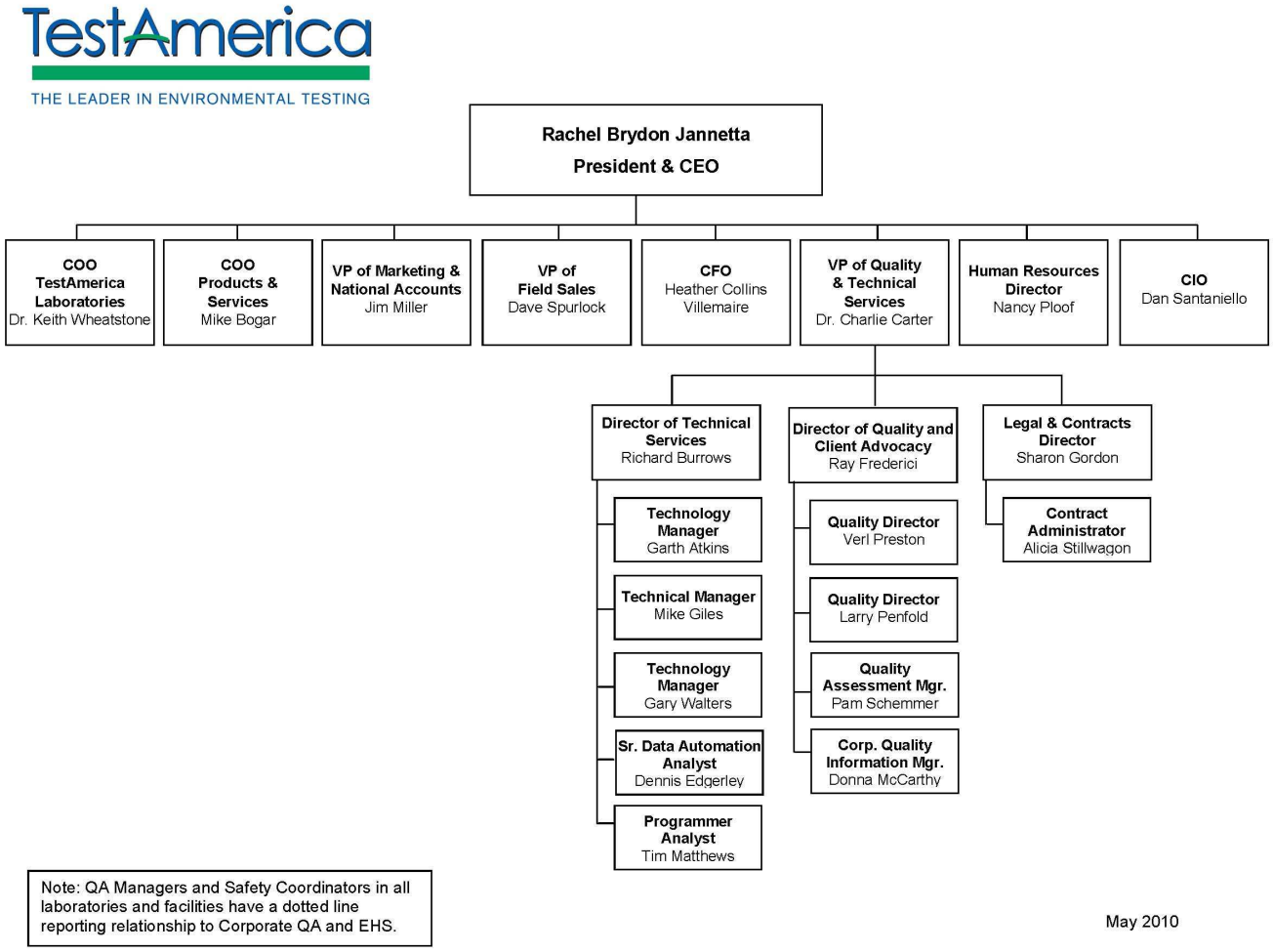
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 12.
- Evaluation of the thoroughness and effectiveness of training.

4.3 DEPUTIES

The following table defines who assumes the responsibilities of key personnel in their absence:

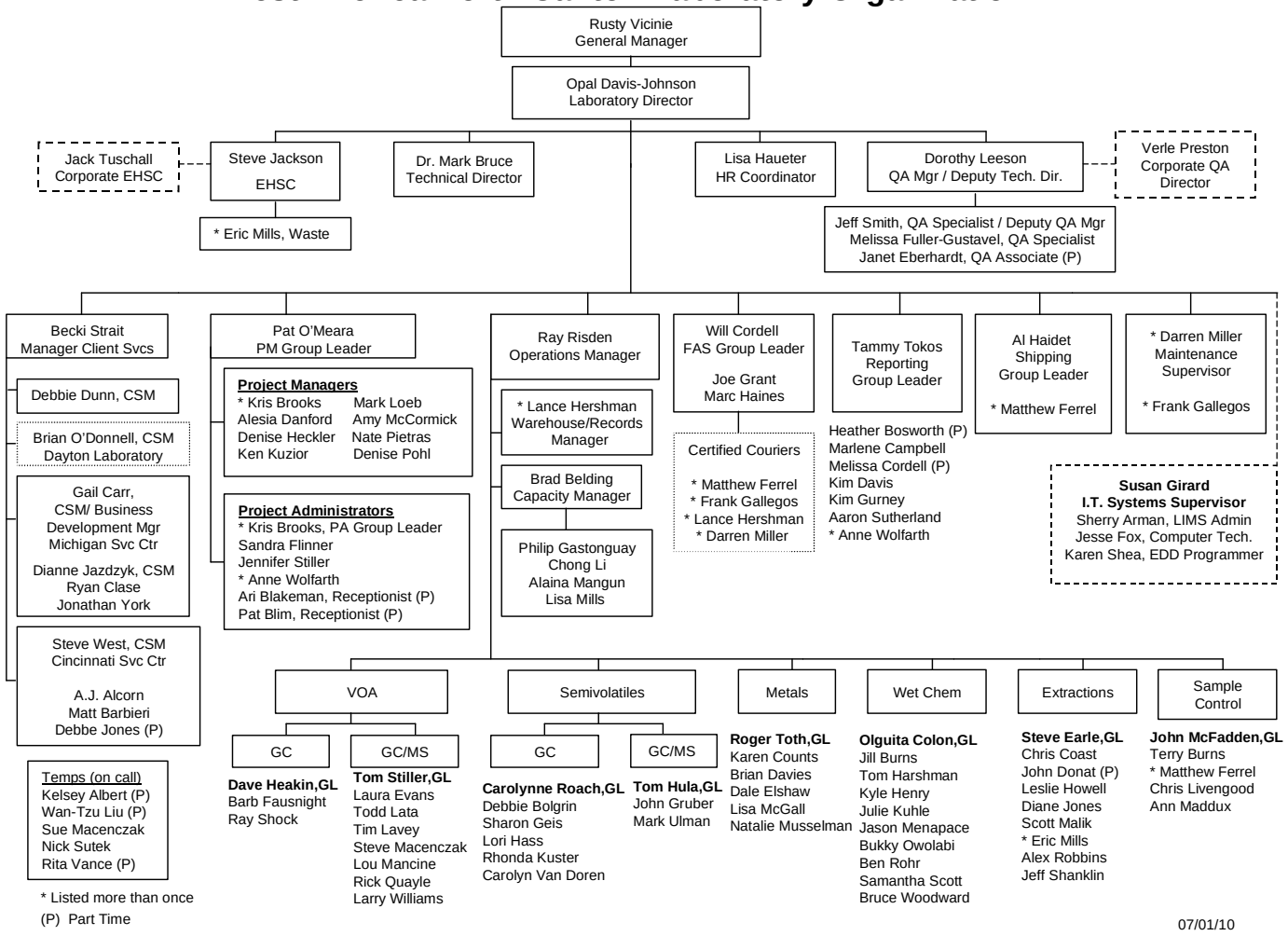
Key Personnel	**Deputy**
Laboratory Director	Technical Director
Quality Assurance Manager	Quality Assurance Specialist
Technical Director	Quality Assurance Manager
EHS Coordinator	Operations Manager

Figure 4-1. Corporate and Laboratory Organization Charts



North Canton Lab Org Chart

TestAmerica North Canton Laboratory Organization



SECTION 5

QUALITY SYSTEM
(NELAC 5.4.2)

5.1 QUALITY POLICY STATEMENT

It is TestAmerica's policy to:

- Provide data of known qualty to its clients by adhering to approved methodologies, regulatory requirements, and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative, and managerial activities. TestAmerica recognizes that the implementation of a Quality Assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.
- To comply with the ISO/IEC 17025:2005 International Standard and to continually improve the effectiveness of the management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 ETHICS AND DATA INTEGRITY

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of the TestAmerica Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy CA-L-P-001) and Employee Ethics Statements (Appendix 1)
- Ethics and Compliance Officers (ECOs)
- A training program
- Self-governance through disciplinary action for violations
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct (Corporate SOP CA-L-S-001)
- Procedures and guidance for recalling data if necessary (Corporate SOP CA-L-S-001)

- Effective external and internal monitoring system that includes procedures for internal audits (Section 16)
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 QUALITY SYSTEM SUPPORTING DOCUMENTATION

The laboratory's Quality System is communicated through a variety of documents

- Quality Assurance Manual – Each laboratory has a lab-specific Quality Assurance Manual.
- Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions - A subset of procedural steps, tasks or forms associated with an operation of a management system, e.g., checklists, preformatted bench sheets, forms.
- Laboratory SOPs – General and technical
- Corporate Quality Policy Memorandums
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Policy Memorandum –
- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies

- Other: Work Instructions (WI), memos, flow charts, etc.

Note: The laboratory's has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's (QAM) shall take precedence over the CQMP in those cases. Any regulatory requirements (e.g.; Ohio VAP, CT RCP, etc) provided in the laboratory specific documents (i.e., QAM and SOPs) take precedence over any policies provided in corporate documents.

5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory must provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS). Equations to derive relevant QC objectives can be found in the method specific SOPs.

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other

regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project) expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability must be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), inter-element corrections (separation), use of matrix modifiers (separation),

specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc.

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory maintains a Control Limit Report that summarizes the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated, and are managed by the laboratory's QA Department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where U.S. EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in NC-QA-018 Statistical Evaluation of Data and Development of Control Charts and in Section 24).

5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Group Leader and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance Department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 25. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

Company Confidential & Proprietary

5.6.1 QC Charts

In-house limits for all QC data must be evaluated and compared to the limits published in the methods for applicable matrices. Method limits must be employed until sufficient QC data are acquired. A minimum of 20 to 30 data points are recommended to establish the in-house QC limits. Calculated results of the QC (LCS) samples are evaluated by comparing against control limits (3-sigma).

Control charts are used to develop control limits, trouble-shoot analytical problems, and, in conjunction with the non-conformance system, to monitor for trends. Program-specific data analysis requirements for control charts are followed as required for data generated under those programs. These additional requirements shall be documented in a QAPP or QAS.

5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6

DOCUMENT CONTROL
(NELAC 5.4.3)

6.1 OVERVIEW

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled at each laboratory Facility:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers, and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the company intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP NC-QA-027, Preparation and Management of Standard Operataing Procedures.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. The laboratory also maintains instrument manuals (hard or electronic copies).

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and Corrective Action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a document control system for each document include a unique document title and number, the number of pages of the item, the effective date, revision number, and the laboratory name. The QA Department is responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a staff member submits an electronic draft to the QA Department

for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document, and retains the official document on file. The official document is provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures must be reviewed at a minimum of every 24 months, and revised as appropriate. For procedures associated with DoD and Ohio VAP project work, applicable SOPs and Policies are reviewed every 12 months. Changes to documents occur when a procedural change warrants.

6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOPs NC-QA-019 and CW-Q-S-001. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA/QC Department.

For changes to SOPs, refer to Corporate SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP), and SOP NC-QA-027, Preparation and Management of Standard Operating Procedures.

Forms, worksheets, Work Instructions, and information are organized by department in the QA office. Electronic versions are kept on a hard drive in the QA department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP NC-QA-027.

6.4 OBSOLETE DOCUMENTS

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP NC-QA-027.

SECTION 7

SERVICE TO THE CLIENT
(NELAC 5.4.7)

7.1 OVERVIEW

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (Percent Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time must be checked for feasibility.

Electronic or hard-copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this must be documented and discussed with the client prior to contract approval (refer to Section 8 for Subcontracting Procedures).

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the

contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 REVIEW SEQUENCE AND KEY PERSONNEL

Appropriate personnel must review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the opportunity is forwarded to a Customer Service Manager (CSM) for review. The CSM contacts the appropriate Sales Executive (National Account Manager, Key Account Executive, Regional Account Executive, and/or Program Manager) to determine which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, reporting specifications, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP CA-L-P-002**,** Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- Laboratory Customer Service Manager
- Laboratory Operations Manager
- Laboratory and/or Corporate Technical Director
- Laboratory and/or Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality Assurance Managers
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director/Manager reviews the formal laboratory quote, and makes final acceptance for their facility.
- Based on the level of discount extended for the project, approval of the General Manager or Sales Director may also be required.

The Customer Service Manager or local Account Executive then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her backup will fulfill the review requirements.

The Legal & Contracts Director (or their designee) maintains copies of all signed contracts. The Laboratory Director also maintains an electronic copy of any contract signed at the local level.

7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. Documents are reviewed by the Laboratory Director and stored on the laboratory's public drive.

The contract must be distributed to and maintained by the Corporate Contracts Department and the applicable Account Executive. A copy of the contract must be filed electronically by the Laboratory Director. Quotes must be archived electronically in the laboratory quote module (TALs) or in the public shared drive if an off-TALs quote is submitted.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps email records or a phone log of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract-specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition,

project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes, e.g., use of a non-standard method or modification of a method, and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory. Project-specific changes made after samples are in-house are communicated through Change Order forms.

Programmatic and/or method changes are communicated via email transmittal and/or in meetings with the applicable Operations Managers. If the modification includes use of a non-standard method, or significant modification of a method, documentation of the modification is made in the case narrative of the applicable data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 SPECIAL SERVICES

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO 17025/NELAC 2003 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third-party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 CLIENT COMMUNICATION

Customer Service Managers (CSMs) and Project Managers (PMs) are the primary communication link to the clients. They must inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project Management must maintain ongoing client communication throughout the entire client project.

Technical Directors, Operation Manager, or Group Leaders are available to discuss any technical questions or concerns the client may have.

7.6 REPORTING

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 CLIENT SURVEYS

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica Sales and Marketing teams periodically develop lab and client-specific surveys to assess client satisfaction.

SECTION 8

SUBCONTRACTING OF TESTS
(NELAC 5.4.5)

8.1 OVERVIEW

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to theTestAmerica Laboratories. The phrase “work sharing” refers to internal transfers of samples between the TestAmerica Laboratories. The term “outsourcing” refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and must meet the same commitments we have made to the client. Refer to TestAmerica’s Corporate SOPs on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process SOP (CA-C-S-001).

When outsourcing analytical services, the laboratory must assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client’s Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client’s analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation must be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work must be identified in the final report, as must non-NELAC accredited work, where required.

For DOD projects, the subcontractor laboratories used must have an established and documented laboratory quality system that complies with DoD QSM requirements. The subcontractor laboratories are evaluated following the procedures outlined below and as seen in Figure 8-2. The subcontractor laboratory must receive project-specific approval from the DoD client before any samples are analyzed.

The QSM has five specific requirements for subcontracting:

1. Subcontractor laboratories must have an established laboratory quality system that complies with the QSM.

2. Subcontractor laboratories must be approved by the specific DoD component laboratory approval process.

3. Subcontractor laboratories must demonstrate the ability to generate acceptable results from the analysis of PT samples, subject to availability, using each applicable method, in the specified matrix, and provide appropriate documentation to the DoD client.

4. Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.

5. Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives.

Project Managers (PMs) or Customer Service Managers (CSM) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory must advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client must be retained in the project folder.

Note: In addition to the client, some regulating agencies, such as the US Army Corps of Engineers and the USDA, require notification prior to placing such work.

8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM or Customer Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory
- Firms specified by the client for the task. (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder.)
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica. A listing of all approved subcontracting laboratories and supporting documentation is available on the TestAmerica intranet site. Verify necessary accreditation, where applicable (e.g., on the subcontractors NELAC, A2LAA accreditation or State Certification.
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses
- NELAC or A2LA-accredited laboratories
- In addition, the firm must hold the appropriate certification to perform the work required

All TestAmerica Laboratories are pre-qualified for work sharing, provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. Refer to Corporate SOP CA-C-S-001, Work Sharing Process.

When the potential subcontract laboratory has not been previously approved, CSMs or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director/Manager. The Laboratory Director/Manager requests that the QA Manager begin the process of approving the subcontract

laboratory as outlined in Corporate SOP CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility. (An e-mail is sufficient documentation; or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented.)

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They must add the lab to the approved list on the intranet site, along with the associated documentation and notify the Finance Group for J.D.Edwards.

8.2.2 The client must assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list, and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors must be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified must be brought tothe attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints must be investigated. Documentation of the complaint, investigation, and corrective action must be maintained in the subcontractor file on the intranet site. Complaints are posted using the Vendor Performance Report**.**
- Information must be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing must be retained on the intranet listing. The QA Manager must notify all TestAmerica laboratories, Corporate Quality, and Corporate Contracts if any laboratory requires removal from the intranet site. This notification must be posted on the intranet site and e-mailed to all Lab Directors/Managers, QA Managers, and Sales Personnel.

8.3 OVERSIGHT AND REPORTING

The CSM or PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM or CSM responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. For TestAmerica Laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control Department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within the TestAmerica.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory EDD, i.e., imported, the report must explicitly indicate the specific lab that produced the data and identify the specific methods and samples.

Note: The results submitted by a TestAmerica work-sharing laboratory may be transferred electronically and the results reported by the TestAmerica work-sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 CONTINGENCY PLANNING

The Laboratory Director/Manager may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, the QA Manager will be required to verify certifications. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1. Example - Client-Approved Subcontractor Form

Client Information:

Client Name & Account Number:_____

Client Contact: _____

Client Address: _____

_____

Project Information: (Please choose all applicable.)

- **Certification required: □ State □ NELAC □ A2LA □ Method___**

  □ Target compound_____ □ Other_____ □ N/A

- **Required Turn around time (method provisional)**_____

Subcontractor's Information:

Subcontractor's Name: _____

Subcontractor's Contact: _____

Subcontractor's Email: _____

Subcontractor's Address: _____

_____

Subcontractor's Phone Number: _____

Analytical Test/Compound/Method to be subcontracted: _____

Certification Statement:

I hereby give ***[Insert Lab Name]*** permission to use the above noted subcontractor for the above noted testing procedures/methods. I realize that the above subcontractor will be held liable for the validity of the above mentioned testing procedures/methods. All subcontractors shall meet the requirements as spelled out in project information and will follow all analytical holding times and turn around times for analytical reports. The subcontract laboratory, and not TestAmerica, will be held liable for liquidated damages for delays in subcontracted analytical reports and/or electronic data deliverables.

Client Signature **Date**

SECTION 9

PURCHASING SERVICES AND SUPPLIES
(NELAC 5.4.6)

9.1 OVERVIEW

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with the TestAmerica's Corporate Controlled Purchases Procedure, SOP CW-F-S-007.

Contracts must be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy CW-F-P-002. Request for Proposals (RFP's) must be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 GLASSWARE

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass must be used where possible. For safety purposes, thick-wall glassware must be used where available.

9.3 REAGENTS, STANDARDS & SUPPLIES

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent and Acid Lot Testing and Approval, SOP CA-Q-S-001.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any

determination. This information is contained in the method SOP. The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use

9.3.2 Receiving

It is the responsibility of the Warehouse Manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to date the material when received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDSs) are kept on a backup disc located in the Wet Chemistry bullpen and available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent. Specifications are listed in SOP NC-QA-017, Reagents and Standards.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five-year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals must not be used past themanufacturer's or SOPs expiration date unless "verified" (refer to Item 3 listed below).

- An expiration date cannot be extended if the dry chemical is discolored or appears otherwise physically degraded, the dry chemical must be discarded.

- Expiration dates can be extended if the dry chemical is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).

- If the dry chemical is used for the preparation of standards, the expiration dates can be extended six months if the dry chemical is compared to an unexpired independent source in performing the method and the performance of the dry chemical is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained in the Standard Logbook in each laboratory group.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 500 psig or the tank must be replaced. The quality of the gases must

meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a conductivity of less than 1 mmho/cm (or specific resistivity of greater than 1.0 megaohm-cm) at 25^{o}C. The specific conductivity is checked and recorded daily. If the water's conductivity is greater than the specified limit, the Operations Manager and appropriate Department Managers/ Group Leaders must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased VOA vials must be certified clean and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage condictions are per the Corporate Environmental Health & Safety Manual (Corporate Document CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or group leader makes a supply request to the Operations Manager and/or the Laboratory Director/Manager. If they agree with the request the procedures outlined in TestAmerica's Corporate Policy CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed, and Purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned, such as HP-20, added to the equipment list described in Section 21 that is maintained by the QA Department, and I.T. must be notified so they can synchronize the instrument for backups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Department Managers or Operations Manager.

9.6 SUPPLIERS

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP CW-F-S-018) and Procurement and Contracts Policy (Policy CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report (CW-F-WI-009).

The Corporate Purchasing Group must work through the appropriate channels to gather the information required to clearly identify the problem and must contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports must be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their

financial stability. The QA Department and/or the Laboratory Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10

COMPLAINTS
(NELAC 5.4.8)

10.1 OVERVIEW

The laboratory considers an effective client complaint handling process to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and improving client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services, (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following SOPs NC-QA-029, Nonconformance and Corrective Action System, and CA-C-S-002, Complaint Handling and Service Recovery.

10.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to CA-C-S-002, Complaint Handling and Service Recovery.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints must be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery

- Process Improvement

The laboratory must inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and must follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing, and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the Corrective Action system described in Section 12.

10.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16)

SECTION 11

CONTROL OF NON-CONFORMING WORK
(NELAC 5.4.9)

11.1 OVERVIEW

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies, and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a Corrective Action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the Corrective Action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's Corrective Action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed.

Note: The laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all Corrective Actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit**.**

11.2 RESPONSIBILITIES AND AUTHORITIES

TestAmerica's Corporate SOP entitled Internal Investigation of Potential Data Discrepancies and Determination for Data Recall (CA-L-S-001) outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of the TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances the Laboratory Director/Manager, Operations Manager, Project Manager, or a member of the QA team may exceptionally authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client must be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's Corrective Action procedures described in Section 13. This information may also need to be documented in logbooks and/or data review as appropriate**.** Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24 hours. The Senior Management staff is compromised of the Laboratory Director, QA Manager, Customer

Service Manager, Operations Manager, I.T. Manager, H.R. Manager, and Technical Director. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality and Client Advocacy, and the laboratory's Corporate Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director/Manager, QA Manager, ECOs, Corporate Quality Director, the COO, General Managers, and the Corporate Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation and Recall Procedure (SOP CA-L-S-001) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECOs and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/Corrective Action reporting in lieu of the data recall determination form contained in TestAmerica Corporate SOP CA-L-S-001.

11.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's Corrective Action system. On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's Corrective Action process may be followed.

11.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality must be respected, and the problem with the required corrective and preventive action must be stated in writing and presented to the Laboratory Director/Manager.

The Laboratory Director/Manager must arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting must be held to confirm that there is a problem, that suspension/restriction of the method is required and must be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target, or test fully back on line.

The QA Manager must also initiate a Corrective Action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed-upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the Internet. It is the responsibility of the Laboratory Director/Manager to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction, i.e., Project Management, Log-in, etc. Clients must NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager must determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Director, QA Manager, Group Leader) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed Corrective Action report.

SECTION 12

CORRECTIVE ACTION
(NELAC 5.4.10)

12.1 OVERVIEW

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the Corrective Action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Nonconformance Memos (NCM).

12.2 GENERAL

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a Corrective Action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution (see more on client complaints in Section 11).

12.2.1 Non-Conformance Memo (NCM)

An NCM is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non matrix related)
- Isolated Reporting / Calculation Errors
- Client Complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips

12.3 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a Corrective Action. There are four main components to a closed-loop Corrective Action process once an issue has been identified--Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

Upon discovery of a non-conformance event, the event must be defined and documented. An NCM must be initiated, someone is assigned to investigate the issue, and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment. SOP NC-QA-029, Nonconformance and Corrective Action System, establishes procedures for the identification and documentation of nonconformances and the corrective actions taken as a result of these events.

The cause analysis step is the key to the process as a long-term corrective action cannot be determined until the cause is determined.

If the cause is not readily obvious, the Group Leader, Lab Director, QA Manager, or designee is consulted.

12.3.2 Selection and Implementation of Corrective Actions

Where corrective action is needed, the laboratory must identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.

Corrective actions must be, to a degree, appropriate to the magnitude of the problem identified through the cause analysis.

Whatever corrective action is determined to be appropriate, the laboratory must document and implement the changes. The NCM is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

- Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Department Manager/Group Leader and QA Manager is responsible to ensure the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM is entered into a database for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and must be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

Also refer to Section 15.2.4, Special Audits.)

12.4 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11 for information regarding the control of non-conforming work). The documentation of these procedures is through the use of an NCM.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, and QAM Sections 19 and 20. The QA Manager reviews all corrective actions monthly, at a minimum, and highlights are included in the QA monthly report.

To the extent possible, samples must be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data must be reported with an appropriate data qualifier and/or the deficiency must be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by a written NCM and appropriate corrective action (e.g., re-analysis) is taken and documented**.**

12.5 BASIC CORRECTIONS

When mistakes occur in records, each mistake must be crossed-out, and [not obliterated (e.g. no White-Out)], and the correct value entered alongside. All such corrections must be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original “uncorrected” file must be maintained intact and a second “corrected” file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) must also be documented.

Table 12-1 .
General Corrective Action Procedures

*** For the Ohio EPA Voluntary Action Program (VAP), please refer to the SOPs for the acceptable criteria, corrective actions, and exceptions.**

INORGANIC LABORATORY QUALITY CONTROL SAMPLES

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Alkalinity	* Method Blank	310.1 2320B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	310.1 2320B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	310.1 2320B	Total alkalinity: 1 per batch of 20 samples	—	N/A
	Matrix Spike Duplicate	310.1 2320B	Total alkalinity: 1 per batch of 20 samples	—	N/A
	Duplicate	310.1 2320B	For carbonate, bicarbonate, hydroxide, alkalinity only. Frequency: 1 per batch of 10 samples Criteria 310.1: ≤ 20 % RPD(3) Criteria 2320B: ≤ 25 % RPD(3) Corrective Action: Flag data outside of limit.	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Ammonia	* Method Blank	350.1 350.2 SM4500 NH-E,F	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	350.1 350.2 SM4500 NH-E,F	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	—	N/A
	Matrix Spike	350.1 350.2 SM4500 NH-E,F	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Ammonia (Cont'd)	Matrix Spike Duplicate	350.1 350.2 SM4500 NH-E,F	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	350.1 350.2 SM4500 NH-E,F	N/A	—	N/A
Ammonia (TKN)	* Method Blank	351.2 351.3 SM4500 NO_3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Ammonia (TKN) (Cont'd)	Laboratory Control Sample	351.2 351.3 SM4500 NO_3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	351.2 351.3 SM4500 NO_3	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	351.2 351.3 SM4500 NO_3	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	351.2 351.3 SM4500 NO_3	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
BOD	* Method Blank	405.1 SM5210B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample and Laboratory Control Sample Duplicate	405.1 SM5210B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	405.1 SM5210B	N/A	—	N/A
	Matrix Spike Duplicate	405.1 SM5210B	N/A	—	N/A
	Duplicate	405.1 SM5210B	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Bromide	* Method Blank	300.0A[(5)]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample and Laboratory Control Sample Duplicate	300.0A[(5)]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples
	Matrix Spike	300.0A[(5)]	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with MS outside of limit
	Matrix Spike Duplicate	300.0A[(5)]	N/A	9056A	N/A
	Duplicate	300.0A[(5)]	N/A	9056A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chemical Oxygen Demand (COD)	* Method Blank	410.4 SM5220D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample and Laboratory Control Sample Duplicate	410.4 SM5220D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	410.4 SM5220D	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	410.4 SM5220D	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	410.4 SM5220D	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chloride	* Method Blank	300.0A [5] 325.2 325.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A [5] 325.2 325.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0A(5)	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Methods 9251Corrective Action: If not within laboratory control limits, rerun all associated samples Method 9056/9253 Corrective Action: Flag data associated with MS outside of limits

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chloride (cont'd)	Matrix Spike Duplicate	325.2 325.3	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits/< 20 % RPD(3) Corrective Action: Flag data outside of limit Method 9056: MSD is not applicable
	Duplicate	300.0A(5) 325.2 325.3	N/A	9056A	N/A
Chlorine, Residual	* Method Blank	330.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	330.5	N/A	—	N/A
	Matrix Spike	330.5	N/A	—	N/A
	Matrix Spike Duplicate	330.5	N/A	—	N/A
	Duplicate	330.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD(3) Corrective Action: Flag data outside of limit.	—	Water

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chromium (Cr^{+6})	* Method Blank	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank unless the blank is above RL, and samples are ND.	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank unless the blank is above RL, and samples are ND.
	Laboratory Control Sample	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples prepped Criteria: percent recovery for water must be within ± 15 % and for solids must be within ± 20% Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	3060A 7196A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chromium (Cr^{+6}) (Cont'd)	Matrix Spike Duplicate	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	3500 Cr-D	N/A	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD(3) limit Corrective Action: Flag data outside of limit.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Conductivity, Specific	* Method Blank	120.1 SM2510B	N/A	9050A	Not Applicable
	Laboratory Control Sample	120.1 SM2510B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9050A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	120.1 SM2510B	N/A	9050A	N/A
	Matrix Spike Duplicate	120.1 SM2510B	N/A	9050A	N/A
	Duplicate	120.1 SM2510B	Frequency: 1 with each batch of samples processed not to exceed 10 samples Criteria: ≤ 20 % RPD(3) Corrective Action: Flag data outside of limit.	9050A	Frequency: 1 with each batch of samples processed not to exceed 10 samples

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Cyanide (Amenable)	* Method Blank	335.1 SM4500C N-G	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	335.1 SM4500C N-G	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.1 SM4500C N-G	N/A	9012A	N/A
	Matrix Spike Duplicate	335.1 SM4500C N-G	N/A	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within lab control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	335.1 SM4500C N-G	N/A	9012A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Cyanide (Total)	* Method Blank	335.2 335.3 335.4 4500-CN E 335.2-CLP-M	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank unless the blank is above RL, and samples are ND.	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank unless the blank is above RL, and samples are ND.
	Laboratory Control Sample	335.2 335.3 335.4 4500-CN E 335.2-CLP-M	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.2 335.3 335.4 4500-CN E 335.2-CLP-M	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Cyanide (Total) (cont'd)	Matrix Spike Duplicate	335.2 335.3 335.4 4500-CN E 335.2-CLP-M	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	335.2 335.3 335.4 335.2-CLP-M	N/A	9012A	N/A
Flashpoint	* Method Blank	—	N/A	1010 ASTM D93-9	N/A
	Laboratory Control Sample	—	N/A	1010 ASTM D93-9	N/A
	Matrix Spike	—	N/A	1010 ASTM D93-9	N/A
	Matrix Spike Duplicate	—	N/A	1010 ASTM D93-9	N/A
	Duplicate	—	Frequency: 1 per batch of ≤20 samples per matrix Criteria: RPD(3) must be ≤ 20% Corrective Action: Flag data associated with unacceptable Duplicate	1010 ASTM D93-9	Frequency: 1 per batch of ≤20 samples per matrix Criteria: RPD(3) must be ≤ 20% Corrective Action: Flag data associated with unacceptable Duplicate

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Fluoride	* Method Blank	300.0A[(5)] 340.2 SM4500F-C,ISE	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable
	Laboratory Control Sample	300.0A[(5)] 340.2 SM4500F-C,ISE	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples
	Laboratory Control Sample Duplicate	300.0A[(5)] SM4500F-C,ISE	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Flouride (Cont'd)	Matrix Spike	300.0A[(5)] 340.2 SM4500F-C,ISE	Frequency: 1 per 10 samples by IC Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with outside of limit
	Matrix Spike Duplicate	340.2 SM4500F-C,ISE	Frequency: 1 per 20 samples by IC Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	340.2	N/A
	Duplicate	300.0A[(5)] 340.2 SM4500F-C,ISE	N/A	9056A	Frequency: 1 with each batch of samples processed Criteria: RPD[(3)] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[(3)] limits

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Hardness	* Method Blank	130.2 2340B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	130.2 2340B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	130.2 2340B	Method 130.2: 1 per 20 samples Method 2340B: Frequency , Criteria, and Corrective Action: See ICP Metals Method 200.7 Requirements	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Hardness (cont'd)	Matrix Spike Duplicate	130.2 2340B	Method 130.2: 1 per 20 samples Method 2340B: Frequency, Criteria, and Corrective Action: See ICP Metals Method 200.7 Requirements	—	N/A
	Duplicate	130.2 2340B	N/A	—	N/A
Iron, Ferrous & Ferric	* Method Blank	3500-Fe D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	3500-Fe D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Iron, Ferrous & Ferric (Cont'd)	Matrix Spike	3500-Fe D	Frequency: 1 every 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit	—	N/A
	Matrix Spike Duplicate	3500-Fe D	Frequency: 1 every 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit	—	N/A
	Duplicate	3500-Fe D	N/A	—	N/A
Nitrate	* Method Blank	300.0A(5) 353.2 SM4500-NO_3-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Nitrate (Cont'd)	Laboratory Control Sample	300.0A[(5)] 353.2 SM4500-NO_3-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0A[(5)] 353.2 SM4500-NO_3-E	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0A[(5)] 353.2 SM4500-NO_3-E	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Nitrate (cont'd)	Duplicate	300.0A(5) 353.2 SM4500-NO_3-E	N/A	9056A	Frequency: 1 per 10 samples Criteria: RPD(3) must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples
Nitrite	* Method Blank	300.0A(5) 353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A(5) 354.1 353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Nitrite (Cont'd)	Matrix Spike	300.0A[(5)] 354.1 353.2	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0A[(5)] 354.1 353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	N/A
	Duplicate	300.0A[(5)] 354.1 353.2	N/A	9056A	Frequency: 1 per 10 samples Criteria: RPD[(3)] must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Nitrate-Nitrite	* Method Blank	353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	353.2	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
pH	* Method Blank	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Laboratory Control Sample	150.1 SM4500H-B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9040B 9045C	N/A
	Matrix Spike	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Matrix Spike Duplicate	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Duplicate	150.1 SM4500H-B	Frequency: 1 with each batch of samples processed not to exceed 10 samples per matrix Criteria: ≤ 20 % RPD(3) limit Corrective Action: Flag data outside of limit.	9040B 9045C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are ≤ 20% RPD(3) Corrective Action: Flag data associated with unacceptable Duplicate
Phenolics	* Method Blank	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Phenolics (Cont'd)	Laboratory Control Sample	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable matrix spike	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Phosphorus (Total and Ortho-phosphate)	* Method Blank	300.0 (4,5) 365.3 365.1 SM4500P-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0(4,5) 365.3 365.1 SM4500P-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0(4,5) 365.3 365.1 SM4500P-E	Frequency: 1 per 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data associated with MS outside of limits
	Matrix Spike Duplicate	365.3 365.1 SM4500P-E	Frequency: 1 per 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	9056A	**N/A**

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Phosphorus (Total and Ortho-phosphate) (Cont'd)	Duplicate	300.0(4,5) 365.3 365.1 SM4500P-E	N/A	9056A	Frequency: 1 with each batch of samples processed Criteria: RPD(3) must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD(3) limits
Solids	* Method Blank	160.1 160.2 160.3 160.4 160.5 SM2540E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: If analyte level in method blank is ≥ RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte are reprepared and reanalyzed.	—	N/A
	Laboratory Control Sample	160.1 160.2 160.3 160.4 160.5 SM2540E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, reprepare and rerun all associated samples	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Solids (Cont'd)	Matrix Spike	160.1 160.2 160.3 160.4 160.5 SM4500P-E	N/A	—	N/A
	Matrix Spike Duplicate	160.1 160.2 160.3 160.4 160.5 SM4500P-E	N/A	—	N/A
	Duplicate	160.1 160.2 160.3 160.4 160.5 SM4500P-E	Frequency: 1 with each batch of samples processed not to exceed 10 samples Criteria: Sample results should agree within 20% if both the sample and sample duplicate results are > 5 X RL Corrective Action: Flag data outside of limit	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Sulfate	* Method Blank	300.0A[(5)] 375.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9038 9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A[(5)] 375.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9038 9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Method 9038 Criteria: Percent recovery must be within ± 15 % Method 9056 Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Sulfate (cont'd)	Matrix Spike	300.0A[(5)] 375.4	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9038 9056A	Frequency: 1 with each batch of samples processed not to exceed 10 samples (9038) or 20 samples (9056) Method 9038 Criteria: Limits are 75% - 125% recovery Method 9056 Criteria: Percent recovery must be within laboratory 0control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	300.0A[(5)] 375.4	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9038 9056A	N/A
	Duplicate	300.0A[(5)] 375.4	N/A	9038 9056A	N/A
Sulfide	* Method Blank	376.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Sulfide (Cont'd)	Laboratory Control Sample	376.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike	376.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	376.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data Method 9034: Not Applicable
	Duplicate	376.1	N/A	9030A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Total Organic Carbon (TOC)	* Method Blank	415.1 SM5310D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	415.1 SM5310D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	415.1 SM5310D	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Total Organic Carbon (TOC) (cont'd)	Matrix Spike Duplicate	415.1 SM5310D	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike Duplicate
	Duplicate	415.1 SM5310D	N/A	9060 Walkley-Black	Not Applicable
Total Organic Halides (TOX)	* Method Blank	450.1 (5)	Frequency: 1 with each set of 8 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9020B	Frequency: Run in duplicate between each group of 8 analytical determinations Criteria: Concentration less than reporting limit or less than 2 X MDL or RL whichever is lower Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	450.1 (5)	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery of analyte must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)	9020B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery of analyte must be within 90-110% Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Total Organic Halides (TOX) (cont'd)	Matrix Spike	450.1 (5)	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data with unacceptable Matrix Spike	9020B	Frequency: 1 per batch of 10 samples Criteria: Must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-WC-0001
	Matrix Spike Duplicate	450.1(5)	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data with unacceptable Matrix Spike	9020B	N/A
	Duplicate	450.1(5)	**N/A**	9020B	N/A
Turbidity	* Method Blank	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Turbidity (cont'd)	Laboratory Control Sample	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	180.1	N/A	—	N/A
	Matrix Spike Duplicate	180.1	N/A	—	N/A
	Duplicate	180.1	Frequency: 1 per 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit Not Applicable.	—	N/A
Mercury by CVAA & CVAFS	* Method Blank	200 series 1631E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank, unless the blank is above RL, and samples are ND.	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank, unless the blank is above RL and samples are ND.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Mercury by CVAA & CVAFS (Cont'd)	Laboratory Control Sample	200 series 1631E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be within ± 20 %. 1631E is +/- 23% Corrective Action: Rerun all samples associated with unacceptable LCS, unless samples are ND, results are reported.	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS, unless MT-0003. Exception: If samples are ND, results are reported.
	Matrix Spike	200 series 1631E	Frequency: with each batch of samples processed not to exceed 20 samples. 1631E frequency is 1 in 10 samples, 71-125% Criteria: Recovery should be within 75-125 % Corrective Action: Flag data associated with unacceptable MS.	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery should be within 75-125 % Corrective Action: Flag data associated with unacceptable MS.
	Matrix Spike Duplicate	200 series 1631E	Frequency: 1 with each batch of samples processed not to exceed 20 samples. 1631E frequency is 1 in 10 samples, 71-125% RPD 24% Criteria: Recovery should be within 75-125 % , RPD(3) should be within 20% Corrective Action: Flag data associated with unacceptable MSD	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery should be within 75-125 % , RPD(3) should be within 20% Corrective Action: Flag data associated with unacceptable MSD

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Mercury by CVAA & CVAFS (Cont'd)	Duplicate	200 series 1631E	N/A	7000A series	N/A
ICP Metals	* Method Blank	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results <RL are also valid. Corrective Action: Rerun all samples associated with unacceptable blank unless the blank is above RL, and samples are ND. SOP NC-MT-012	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results <RL are also valid. Corrective Action: Rerun all samples associated with unacceptable blank unless the blank is above RL, and samples are ND. SOP NC-MT-012
	Laboratory Control Sample	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be ± 85-115%. If LCS is biased high and samples are <RL, the results are valid. Corrective Action: Rerun all samples associated with unacceptable LCS SOP NC-MT-012	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be ± 20 %. If LCS is biased high and samples are <RL, the results are valid. Corrective Action: Rerun all samples associated with unacceptable LCS SOP NC-MT-012. If samples are ND, results are reported.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
ICP Metals (Cont'd)	Matrix Spike	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125% Corrective Action: Flag data associated with unacceptable matrix spike SOP NC-MT-012	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125% Corrective Action: Flag data associated with unacceptable matrix spike SOP NC-MT-012
	Matrix Spike Duplicate	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125%, RPD(3) must be within 20% Corrective Action: Flag data associated with unacceptable matrix spike SOP NC-MT-012	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125%, RPD(3) must be within 20% Corrective Action: Flag data associated with unacceptable matrix spike SOP NC-MT-012
	Duplicate	200.7 200.8	Not Applicable	6010B 6020	Not Applicable
	Serial Dilution	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: 10% difference. 10% difference only applied if sample results are >50 times IDL. Corrective Action: Flag data associated with unacceptable serial dilution SOP NC-MT-012	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: 10% difference. 10% difference only applied if sample results are >50 times IDL. Corrective Action: Flag data associated with unacceptable serial dilution SOP NC-MT-012

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Footnotes

[1] National Pollutant Discharge Elimination System

[2] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

[3] RPD-Relative Percent Difference

[4] Orthophosphate only

[5] Method not listed in 40 CFR Part 136. Method 300.0 is a proposed 40CFR method. Specific state and/or region approval is required for NPDES.

[6] Current promulgated method is a Guidance Method Only, SW-846, Final Update III, Rev.3, 12/96.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Aromatic Volatiles by GC	* Method Blank	602	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	602	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	602	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Aromatic Volatiles by GC (Cont'd)	Matrix Spike Duplicate	602	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	602	N/A	8021B	N/A
	Surrogates	602	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Reprepare and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	602	Optional: Internal standards are added to the method blank and all samples (QC included).	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Halogenated Volatiles by GC	* Method Blank	--	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	--	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	--	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	--	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Halogenated Volatiles by GC (Cont'd)	Duplicate	--	N/A	8021B	N/A
	Surrogates	--	N/A	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Reprepare and reanalyze samples or flag sample data not meeting surrogate criteria.
	Internal Standards	--	N/A	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Herbicides	Laboratory Control Sample	615 [3]	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract all samples associated with unacceptable LCS	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS
	Matrix Spike	615 [3]	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	615 [3]	N/A	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory control limits Corrective Action: Flag data associated with unacceptable matrix spike sample

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Herbicides (cont'd)	Duplicate	615 [3]	N/A	8151A	N/A
	Surrogates	615 [3]	N/A	8151A	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must fall within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	615 [3]	N/A	8151A	Optional
Pesticides/ PCBs	* Method Blank	608	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Reprepare and reanalyze all samples associated with unacceptable blank

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Pesticides/ PCBs (Cont'd)	Laboratory Control Sample	608	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	608	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Pesticides/ PCBs (cont'd)	Matrix Spike Duplicate	608	N/A	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	608	N/A	8081A 8082	N/A
	Surrogates	608	Not specified in method	8081A 8082	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: Results must fall within laboratory established control limits Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
Petroleum Hydro-carbons	* Method Blank	1664A	Frequency: 1 with each preparation batch Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9071B	Frequency: 1 with each preparation batch Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Petroleum Hydro-carbons (Cont'd)	Laboratory Control Sample	1664A	Frequency: 1 with each analytical batch Criteria: Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS	9071B	Frequency: 1 with each analytical batch Criteria: Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	1664A	Frequency: 1 with every 10 samples per site Criteria: See percent recovery limits in SOP, NC-WC-0084 Corrective Action: See SOP, NC-WC-0084	9071B	Frequency: 1 with every 10 samples per site Criteria: See percent recovery limits in SOP, NC-WC-0084 Corrective Action: See SOP, NC-WC-0084
	Matrix Spike Duplicate	1664A	Frequency: 1 with every 20 samples per site Criteria: See percent recovery and RPD limits in SOP, NC-WC-0084 Corrective Action: See NC-WC-0084	9071B	Frequency: 1 with every 10 samples per site Criteria: See percent recovery and RPD limits in SOP, NC-WC-0084 Corrective Action: See NC-WC-0084
	Duplicate	1664A	N/A	9071B	N/A

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Purgeable Halocarbons by GC	* Method Blank	601	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	601	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	601	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Purgeable Halocarbons by GC (Cont'd)	Matrix Spike Duplicate	601	N/A	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	601	N/A	8021B	N/A
	Surrogates	601	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	601	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Semivolatiles	Matrix Spike	625	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	625	N/A	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Semivolatiles (Cont'd)	Duplicate	625	N/A	8270C	N/A
	Surrogates	625	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8270C	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	625	Frequency: Internal standards spiked into method blank and all samples (QC included) Criteria: All internal standard recoveries must be within laboratory control limits Corrective Action: Flag sample data not meeting internal standard recovery requirements	8270C	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard. The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Volatiles by GC/MS	* Method Blank	624	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	624	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Volatiles by GC/MS (Cont'd)	Matrix Spike	624	Frequency: 1 per ≤ 20 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	624	N/A	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	624	N/A	8260B	N/A

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Volatiles by GC/MS (cont'd)	Surrogates	624	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8260B	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	624	Frequency: Internal standards spiked into method blank and all samples (QC included) Criteria: All internal standard recoveries must be within laboratory control limits Corrective Action: Flag sample data not meeting internal standard recovery requirements	8260B	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard. The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.

Footnotes

[1] National Pollutant Discharge Elimination System

[2] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996)

[3] Method not listed in 40 CFR Part 136

.

SECTION 13

PREVENTIVE ACTION
(*NELAC 5.4.11)*

13.1 OVERVIEW

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc.

The monthly QA Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, Ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process (Section 13) is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process, and management review.

13.1.2 Any Preventive Actions undertaken or attempted must be taken into account during the Annual Management Review (Section 16). A highly detailed recap is not required; a simple recount of success and failure within the preventive action program will provide management a measure for evaluation.

13.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in SOP CA-Q-S-003, Management of Change.

SECTION 14

CONTROL OF RECORDS
(NELAC 5.4.12)

The laboratory maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

14.1 OVERVIEW

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA Department in a database which is backed up as part of the regular network backup. Records are of two types--either electronic or hard-copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the Records Manager.

Table 14-1. Records Index

Type of Record	Retention	Disposition
General Laboratory Documents		
Instrument output	5 yrs from project completion	Shred or burn
Quality control data	5 yrs from project completion*	Shred or burn
Field sample data	5 yrs from project completion	Shred or burn
Final analytical reports	5 yrs from project completion	Shred or burn
Instrument logbooks	5 yrs from last entry	Shred or burn
Equipment monitoring & maintenance records	5 yrs from last entry	Shred or burn
Instrument calibration records	5 yrs from last entry	Shred or burn
Standard preparation logs	5 yrs from last entry	Shred or burn
Standards certificates	5 yrs from last entry	Shred or burn
Measurement & test equipment logs (e.g., refrig., balances, etc.)	5 yrs from last entry	Shred or burn
Method & instrument validation records	5 yrs from last entry	Shred or burn
Instrument manuals	Retain until superseded	Trash
Project management files	5 yrs from date of archival	Shred or burn
Quotes & proposals	2 yrs from date of expiration	Shred or burn
LQM, policies, & SOPs	5 yrs from date of archiving	Shred or burn

Type of Record	Retention	Disposition
Analyst demonstrations of proficiency	5 yrs from date of archival	Shred or burn
Quality assurance audits	5 yrs from last entry	Shred or burn
Certifications & approvals	5 yrs from last entry	Shred or burn
Employee signature list	5 yrs from date of archival	Shred or burn
MDL Studies	5 yrs from last entry	Shred or burn
Performance testing studies	5 yrs from last entry	Shred or burn
QA reports to management	5 yrs from last entry	Shred or burn
Quality control charts	5 yrs from last entry	Shred or burn
Environment, Health and Safety Records		
Medical records	Retain while active & 30 yrs from last entry	Shred or burn
Employee exposure & monitoring records	Retain while active & 30 yrs from last entry	Shred or burn
Workers compensation files & first report of injury	Retain while active & 30 yrs from last entry	Shred or burn
Accident logs (OSHA Form 200)	5 yrs from last entry	Shred or burn
Accident reports	5 yrs from last entry	Shred or burn
Environmental permits	5 yrs from last entry	Shred or burn
Environmental management, e.g., discharge reports	5 yrs from last entry	Shred or burn
Health & safety audits	5 yrs from last entry	Shred or burn
Chemical Hygiene Plan	5yrs from archival	
Safety Inspections	5 yrs from last entry	Shred or burn
TLD exposure records	5 yrs from last entry	Shred or burn
EH&S training	5 yrs from last entry	Shred or burn
Accounting	See Accounting and Controls Procedures Manual	
Administrative		
Personnel records (not including medical or disability records)	7 years from last entry	Shred or burn

* Exceptions listed in Table 14-2.

All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss**.** All records must be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records and electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention must be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.1 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Note: For the Ohio VAP program the laboratory is required to notify Ohio EPA of its intent to dispose of any records.

Table 14-2. Special Record Retention Requirements

Program	Retention Requirement
Ohio – Drinking Water	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
OSHA - 40 CFR Part 1910	30 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement and others as negotiated.
Ohio Voluntary Action Program	10 years

Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.2 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hardcopy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.12.1 for more information.

14.1.3 The record-keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. (Records stored off site should be accessible within two days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This must include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory copy of the Chain-of-Custody is stored with the invoice and the Work Order sheet generated by LIMS. The Chain-of-Custody would indicate the name of the sampler. If any sampling notes are provided with a Work Order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record-keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes, e.g., set format for naming electronic files, set format for what is included with a given analytical data set. SOP NC-QA-019, Records Information Management, outlines this procedure. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run long or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into LIMS for each method as required.

- Changes to hardcopy records must follow the procedures outlined in Sections 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "Sampled by," "Prepared by," "Reviewed by", or "Analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.

- Hard-copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure no data is lost, and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard-copy which was scanned.

- Also refer to Section 19.14.1, “Computer and Electronic Data Related Requirements”.

14.2 TECHNICAL AND ANALYTICAL RECORDS

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement (refer to Section 15.1). The records for each analysis must contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records must include the identity of laboratory personnel responsible for sample preparation, performance of each analysis and reviewing results.

14.2.2 Observations, data, and calculations are recorded in real-time at the time they are made and are identifiable to the specific task.

14.2.3 Changes to hardcopy records must follow the procedures outlined in Sections 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails. The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- Laboratory sample ID code
- Date of analysis. Time of analysis is also required if the holding time is 72 hours or less, or when time-critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available. Instrument logs may be in electronic format.
- Analysis type
- All manual calculations and manual integrations
- Analyst or operator initials/signature
- Sample preparation, including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents
- Test results
- Standard and reagent origin, receipt, preparation, and use
- Calibration criteria, frequency, and acceptance criteria
- Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions
- Quality control protocols and assessment
- Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries

- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats**.**

14.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- All original raw data, whether hard-copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)
- A written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value
- Copies of final reports
- Archived SOPs
- Correspondence relating to laboratory activities for a specific project
- All Corrective Action reports, audits and audit responses
- Proficiency test results and raw data
- Results of data review, verification, and cross-checking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include, but are not limited to, records pertaining to:

- Sample preservation including appropriateness of sample container and compliance with holding time requirement
- Sample identification, receipt, acceptance or rejection and login
- Sample storage and tracking including shipping receipts, sample transmittal / COC forms
- Procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard-copy form Refer to Table 14-1.

14.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

14.5.1 All records (including those pertaining to test equipment), certificates, and reports are safely stored, held secure, and in confidence to the client. Certification-related records are

available to the accrediting body upon request.

14.5.2 All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

14.5.3 Records that are stored or generated by computers or personal computers have hardcopy, write-protected backup copies, or an electronic audit trail controlling access**.**

14.5.4 The laboratory has a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially.

14.5.5 Transfer of Ownership

In the event the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous five years of such action.

14.5.6 Records Disposal

14.5.6.1 Records are removed from the archive and destroyed after five years, unless otherwise specified by a client or regulatory requirement. On a project-specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration (refer to Tables 14-1 and 14-2).

14.5.6.2 Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

14.5.6.3 If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15

AUDITS
(NELAC 5.4.13)

15.1 INTERNAL AUDITS

.
Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.

Audits are conducted and documented as described in TestAmerica Corporate SOP CA-Q-S-004 on performing Internal Audits. The types and frequency of routine internal audits are shown in Table 16-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems	QA Department or Designee	All areas of the laboratory annually
QA Technical Audits - Evaluate raw data vs. final reports - Analyst integrity - Data authenticity	QA Department or Designee	All methods within a 2-year period, with at least 15% of methods every quarter
SOP Method Compliance	Group Leader	- All SOPs within a 2-year period - All new analysts or new analyst/methods within 3 months of IDOC
Special	QA Department or Designee	Surveillance or spot checks performed as needed
Performance Testing	Analysts with QA oversight	Two successful per year for each NELAC field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, laboratory Data Integrity and Ethics Policies, NELAC quality systems, client and state

requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to, data review, quality controls, preventive action, and corrective action. The completeness of earlier corrective action is assessed. The audit is divided into modules for each operating or support area of the lab, and each module is comprehensive for a given area. The area audits may be done on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

Note: Part of the quality systems audit relates to regulatory compliance. An assessment of the laboratory's compliance to regulatory requirements is performed by Corporate QA through monthly management reports, review of client and regulatory concerns and also through periodic on-site evaluations.

15.1.2 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, MintMiner is used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits must include all methods within a two-year period.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs must be assessed by the Technical Director and the QA department at least every two years. The work of each newly hired analyst is assessed within three months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products must be performed within 3 months of completing the documented training.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies--nonpotable water and soil

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique

request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory group leaders are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. A copy of the audit report and the laboratory's Corrective Action plan must be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2003 NELAC standards.

15.3 AUDIT FINDINGS

Audit findings are documented using the Corrective Action process and database. The laboratory's Corrective Action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must set and agreed to by Operations management and the QA Manager.

Developing and implementing Corrective Action to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. A copy of the audit report and the laboratory's Corrective Action plan must be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory must take timely corrective action, and must notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16

MANAGEMENT REVIEWS
(NELAC 5.4.14)

16.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report must be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director/Manager and their Corporate Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director/Manager, General Manager, or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

16.2 ANNUAL MANAGEMENT REVIEW

The Senior Lab Management Team (Laboratory Director, Technical Director, Operations Manager, QA Manager, HR Supervisor, I.T. Supervisor) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining quality goals and objectives. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director/Manager. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory must summarize any critical findings that cannot be solved by the lab, and report them to Corporate IT.

The Management Systems Review (Corporate SOP CA-Q-S-008 and Work Instruction CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective; therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review
- Prior Monthly QA Reports issues
- Laboratory QA Metrics
- Review of report reissue requests
- Review of client feedback and complaints

- Issues arising from any prior management or staff meetings
- Minutes from prior Senior Lab Management Team meetings. Issues that may be raised from these meetings include:
  - Adequacy of staff, equipment and facility resources
  - Adequacy of policies and procedures
  - Future plans for resources and testing capability and capacity
- The annual internal double blind PT program sample performance (if performed)
- Compliance to the Ethics Policy and Data Integrity Plan, including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and Corporate Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants
- A reference to the existing data quality related documents and topics that were reviewed
- Quality system or operational changes or improvements that will be made as a result of the review, e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)

Changes to the quality systems requiring update to the laboratory QA Manual must be included in the next revision of the QA Manual.

16.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/ Recall SOP CA-L-S-001 must be followed. All investigations that result in finding inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's COO, VP of Client & Technical Services, General Managers and Corporate Quality Directors receive a monthly report from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Managers are also made aware of progress on these issues for their specific labs.

SECTION 17

PERSONNEL
(NELAC 5.5.2)

17.1 OVERVIEW

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training must have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff must be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training must be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL

The laboratory makes every effort to hire analytical staff that posses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. There are competent analysts and technicians in the industry who have not earned a college degree. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet "Human Resources" web-page (also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc. are also considered

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least one year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	Or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience. Or 5 years of prior analytical experience
Technical Directors/Department Managers – General	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Director – Wet Chem only (no advanced instrumentation)	Associate degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 TRAINING

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame*	Employee Type
New Hire Orientation	Immediately	All
Environmental Health & Safety Orientation	Prior to lab work	All
Environmental Health & Safety Orientation Follow-up Test	30-60 days after hire	All
Environmental Health & Safety Training	Refer to EH&S Manual	All
Ethics – New Hires	1 week of hire	All
Ethics - Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in the employee's training file.
- Documentation of proficiency (refer to Section 19)
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training
- A Confidentiality Agreement signed by each staff member signed at the time of employment
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct, e.g., ethics. This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts' knowledge to refer to QA Manual for quality issues
- Analysts following SOPs, i.e., practice matches SOPs
- Analysts regularly communicate to group leaders and QA if SOPs need revision rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP NC-QA-028, Employee Orientation and Training.

17.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica**.** It is a formal part of the initial employee orientation within one week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and annual refresher for all employees. Senior management at each facility performs the Ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times, TestAmerica has established a Corporate Ethics Policy (CA-L-P-001) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement/Agreement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts; and for that reason, TestAmerica has a zero tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping
- Discussion regarding data integrity procedures

- Specific examples of breaches of ethical behavior--peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion
- Internal monitoring. Investigations and data recalls
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient

Additionally, a Data Integrity Hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS
(NELAC 5.5.3)

18.1 OVERVIEW

The laboratory is an 54,440 sq. ft. secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity-controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

18.2 ENVIRONMENT

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. A 225KVA UPS is installed in the main electrical bus to provide at least 15 minutes of backup power in the event of a power failure. This unit also provides voltage and frequency control of lab and office power. A spike/surge arrestor is installed to protect against power surge/sag and lightning strikes. A 30 KW natural gas-fueled backup generator is installed to provide power to the I.T. area in the event of a power failure. Additionally, this generator provides power to two walk-in sample

storage coolers and several other smaller sample storage coolers. Smaller portable generators are available to provide “spot power” where needed in the event of a power failure.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing must be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to, and use of, all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory
- Sample receipt areas
- Sample storage areas
- Chemical and waste storage areas
- Data handling and storage areas
- Sample processing areas
- Sample analysis areas

18.4 FLOOR PLAN

A floor plan can be found in Appendix 1.

18.5 BUILDING SECURITY

Building keys are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor’s logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into

the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

Signs are posted in the laboratory designating employee only areas - "Authorized employees beyond this point".

SECTION 19

TEST METHODS AND METHOD VALIDATION
(NELAC 5.5.4)

19.1 OVERVIEW

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples; and where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 STANDARD OPERATING PROCEDURES (SOPs)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP CW-Q-S-002 entitled Writing a Standard Operating Procedure, or the laboratory's SOP NC-QA-027, Preparation and Management of Standard Operating Procedures.
- SOPs are reviewed at a minimum of every two years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 LABORATORY METHODS MANUAL

For each test method, the laboratory must have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory must demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 SELECTION OF METHODS

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services, e.g., special matrices, non-routine compound lists, etc., the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods must be used.

When clients do not specify the method to be used or methods are not required, the methods used must be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th edition/ on-line edition Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*

- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

TABLE 19-1. Wet Chemistry Methods [1]

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Acidity	Water	305. [2] SM 2310 B	--	--
Alkalinity, Bicarbonate, Carbonate	Water	305. [2] SM 2320 B		
	Solid	EPA 310.1[2] (M)	--	--
Arsenic (ASV) Anodic Stripping Voltammetry	Water	--	EPA 7063	--
Ash Content	Solid	--	--	ASTM D29-74
Biochemical Oxygen Demand, Carbonaceous	Water	EPA 405.1 SM 5210 B	--	--
Bromide	Water	EPA 300.0A	EPA 9056A	--
	Waste	EPA 300.0A	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A	--
Cation-Exchange Capacity	Solid	--	EPA 9081	--
Chemical Oxygen Demand	Water	EPA 410.4 SM 5220D	--	--
	Waste	EPA 410.4	--	--
Chloride	Water	EPA 300.0A EPA 325.2[2]	EPA 9056A EPA 9251	EPA 325.2[2]
	Waste	EPA 300.0A	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A EPA 9251(M)	--
Chromium, Hexavalent	Water	EPA 3500-Cr-D	EPA 7196A	--
	Waste	EPA 3500-Cr-D	EPA 7196A	--

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
	Solid	--	EPA 3060A EPA 7196A	--
Specific Conductance	Water	EPA 120.1 SM 2510B	EPA 9050A	--
	Waste	EPA 120.1	EPA 9050A	--
	Solid	--	EPA 9050A	--
Chlorine, Residual	Water	EPA 330.5[2] SM 3500 CL-G	--	--
Cyanide (Amenable)	Water	EPA 335.1[2] SM 4500 CN-G	EPA 9012A	--
	Solid	--	EPA 9012A	--
Cyanide (Total)	Water	SM 4500-CN E EPA 335.4 335.2-CLP-M	EPA 9012A	---
	Waste	--	EPA 9012A	--
	Solid	335.2-CLP-M	EPA 9012A	--
Cyanide (Weak and Dissociable) (Free)	Water	SM 4500-CN I	--	--
Dissolved Oxygen	Water	360.1[2] SM 4500 O-G	--	--
Flash Point	Waste	--	EPA 1010	ASTM D93-9
	Solid	--	EPA 1010	ASTM D93-9
Fluoride	Water	EPA 300.0A EPA 340.2[2]	EPA 9056A	SM 4500 F-C, ISE
	Waste	EPA 340.2 (M)[2] EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 340.2 (M)[2] EPA 300.0A (M)	EPA 9056A	--
Iron, Ferrous & Ferric	Water	SM 3500 FE D	--	--
Hardness	Water	EPA 130.2[2]	--	SM 2340B
Moisture	Solid	---	EPA 160.3 (M) ASTM D2216-90	---
Nitrogen, Ammonia	Water	EPA 350.1	--	EPA 350.2[2]
	Waste	EPA 350.1	--	EPA 350.2[2]
	Solid	EPA 350.1	--	EPA 350.2[2]
	Water	SM 4500 NH_3-E (Titration)	--	--

Analytical Parameters Matrix	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Nitrogen, Ammonia (cont'd)	Water	SM 4500 NH_3-F (ISE)	--	--
Nitrite (NO_2)	Water	EPA 300.0A	EPA 9056A	--
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A	--
Nitrate (NO_3)	Water	EPA 300.0A	EPA 9056A	SM 4500 NO_3-E
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M)	--	--
Nitrate plus Nitrite NO_2/NO_3	Water	EPA 353.2	--	--
	Waste	EPA 353.2	--	--
Total Kjeldahl Nitrogen (TKN)	Water	EPA 351.3	--	SM 4500 NO_3
	Waste	EPA 351.3	--	--
	Solid	EPA 351.3	--	--
Oil and Grease (Hexane Extractable Material)	Water	EPA 1664A	EPA 9071B	--
	Waste	EPA 1664A	EPA 9071B	--
	Solid	--	EPA 9071B	--
Ortho-phosphate o-PO_4	Water	EPA 300.0A EPA 365.1	EPA 9056A	SM 4500 P-E
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M) EPA 365.1	EPA 9056A	--
pH	Water	EPA 150.1[2]	EPA 9040B	EPA 9041
	Waste	SM 4500 H-B	EPA 9045C	--
	Solid	---	EPA 9045C	--
Paint Filter	Water	--	EPA 9095A	--
Phenolics	Water	EPA 420.1	--	--
	Waste	--	EPA 9065	--
	Solid	--	EPA 9065	--
Phosphorus (Total)	Water	EPA 365.1	--	SM 4500 P-E
	Waste	EPA 365.1	--	--
	Solid	EPA 365.1	--	--
Sulfate (SO_4)	Water	EPA 300.0A EPA 375.4[2]	EPA 9056A EPA 9038	--
	Waste	EPA 300.0A (M) EPA 375.4[2]	EPA 9056A EPA 9038	--
	Solid	EPA 300.0A (M)	EPA 9056A EPA 9038 (M)	--

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA	Other
Sulfide	Water	EPA 376.1[2]	EPA 9030A SM 4500	9030B/9034
Total Organic Carbon (TOC)	Water	EPA 415.1[2]	EPA 9060	SM 5310 D
	Waste	--	EPA 9060	--
	Solid	EPA 415.1 (M)	EPA 9060 (M)	Walkley-Black
Total Organic Halides (TOX)	Water	--	EPA 9020B EPA 9023(EOX)	EPA 450.1
	Waste	--	--	--
	Solid	--	EPA 9020B	--
Total Petroleum Hydrocarbons	Water	EPA 1664A (SGT-HEM)	EPA 9071B	--
	Waste	EPA 1664A (SGT-HEM)	EPA 9071B	--
	Solid	--	EPA 9071B	--
Total Solids	Water	EPA 160.3	--	--
	Waste	EPA 160.3	--	--
	Solid	EPA 160.3 (M)	--	--
Total Dissolved Solids	Water	EPA 160.1	--	2540E
Total Suspended Solids	Water	EPA 160.2	---	2540E
Volatile and Volatile Suspended Solids	Water	EPA 160.4	--	--
Settleable Solids	Water	EPA 160.5	--	--
Turbidity	Water	EPA 180.1	--	--

[1] Any matrix not listed is not applicable for the associated method
[2] Removed from 40CFR

TABLE 19-2. Methods for Mercury by Cold Vapor Atomic Absorption

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Mercury (CVAA)	Water		EPA 245.1	EPA 7470A	--
	TCLP Leachate		--	EPA 7470A	--
	Waste		--	EPA 7471A	--
	Solid		EPA 254.5	EPA 7471A	--

TABLE 19-3. Methods for Mercury by Cold Vapor Atomic Fluororescence

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Mercury, Low Level (CVAFS)	Water		--	--	EPA 1631E

TABLE 19-4. Methods for Metals by ICP and ICPMS

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Aluminum	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Antimony	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Arsenic	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Barium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Beryllium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Boron	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		EPA 200.7	EPA 6010B	---
Calcium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		EPA 200.7	EPA 6010B	---
Cadmium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Cobalt	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Chromium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Copper	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Iron	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Lead	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Magnesium	Water		EPA 200.7	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7	EPA 6010B EPA 6020	---
Manganese	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Molybdenum	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Nickel	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Potassium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		---	EPA 6010B	---
Selenium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Silver	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Sodium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		---	EPA 6010B	---
Tin	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Thallium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Titanium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		---	EPA 6010B	---
Vanadium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		---	EPA 6010B EPA 6020	---
Zinc	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

TABLE 19-5. Metals Sample Preparation Methods

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Toxicity Characteristic Leaching Procedure (TCLP)	Water		---	EPA 1311	---
	Waste		---	EPA 1311	---
	Solid		---	EPA 1311	---
ICP Metals	Water		EPA 200.7	EPA 3005A EPA 3010A	---
	TCLP Leachate		---	EPA 3010A	---
	Waste		---	EPA 3050B	---
	Solid		---	EPA 3050B	---
ICPMS Metals	Water		EPA 200.8	EPA 3010A	---
	TCLP		---	EPA 3010A	---
	Waste		---	EPA 3050B	---
	Solid		---	EPA 3050B	---
CVAA Mercury	Water		EPA 245.1	EPA 7470A	---
	TCLP Leachate		---	EPA 7470A	---
	Waste		---	EPA 7471A	---
	Solid		---	EPA 7471A	
CVAFS Mercury Low Level	Water		---	---	EPA 1631E

TABLE 19-6. Organic Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	EPA 624	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Halogenated Volatiles by GC	Water	EPA 601	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Aromatic Volatiles by GC	Water	EPA 602	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Semivolatiles by GC/MS	Water	EPA 625	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---
Pesticides/PCBs by GC	Water	EPA 608	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Herbicides by GC	Water		EPA 615	EPA 8151A	---
	Waste		---	EPA 8151A	---
	Solid		---	EPA 8151A	---
Total Petroleum Hydrocarbons (Gasoline Range) by GC	Water		---	EPA 5030B	WI GRO
	Waste		---	EPA 5030B EPA 5035	WI GRO
	Solid		---	EPA 5035 EPA 5035	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC	Water		---	EPA 3510C EPA 3520C	WI DRO
	TCLP Leachate		---	EPA 3510C EPA 3520C	---
	Waste		---	EPA 3550B EPA 3580A	WI DRO
	Solid		---	EPA 3550B	WI DRO

TABLE 19-7. Organic Methods of Analysis

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water		EPA 624	EPA 8260B	---
	Waste		---	EPA 8260B	---
	Solid		---	EPA 8260B	---
Halogenated Volatiles by GC	Water		EPA 601	EPA 8021B	---
	Waste		---	EPA 8021B	---
	Solid		---	EPA 8021B	---
Aromatic Volatiles by GC	Water		EPA 602	EPA 8021B	---
	Waste		---	EPA 8021B	---
	Solid		---	EPA 8021B	---
Semivolatiles by GC/MS	Water		EPA 625	EPA 8270C	
	Waste		---	EPA 8270C	---
	Solid		---	EPA 8270C	---
Pesticides/PCBs by GC	Water		EPA 608	Pesticides 8081A PCBs 8082	---
	TCLP Leachate		---	Pesticides 8081A PCBs 8082	---
	Waste		---	Pesticides 8081A PCBs 8082	---
	Solid		---	Pesticides 8081A PCBs 8082	---
Phenoxyacid Herbicides by GC	Water		---	EPA 8151A	---
	TCLP Leachate		---	EPA 8151A	---
	Waste		---	EPA 8151A	---
	Solid		---	EPA 8151A	---
Gasoline Range Organics by GC	Water		---	EPA 8015B (M)	WI GRO
	Waste		---	EPA 8015B (M)	---
	Solid		---	EPA 8015B (M)	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC/FID	Water		---	EPA 8015B (M)	WI DRO
	Waste		---	EPA 8015B (M)	---
Dissolved Gases RSK-175	Water		---	---	SOP
Formaldehyde Carbonyl Compounds	Water		---	EPA 8315	---

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory must inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it must be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory must confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

19.4.2.1 A demonstration of capability is performed (SOP NC-QA-028, Employee Orientation and Training) whenever there is a change in instrument type (e.g., new instrumentation), method, or personnel.

19.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Technical Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

19.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve (low standard at or below the QL)and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported

below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).

Note: For Ohio VAP work, the term Reporting Limit will be used.

- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted as "*Reporting Limit based on the low standard of the calibration curve".*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

19.4.3.1 At least four aliquots must be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.2 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest. Refer to SOP NC-QA-028, Employee Orientation and Training, for details on this procedure.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

19.4.3.3 A certification statement (see Figure 19-1 as an example) must be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

19.5 LABORATORY-DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch-specific QC samples such as LCS, method blanks, or PT samples.

19.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B, or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to Section 19.7.10). Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used.

Refer to the Corporate SOP CA-Q-S-006 or the laboratory's SOP NC-QA-021 for details on the laboratory MDL process.

Note: For Ohio VAP projects, the MDL procedure must also comply with OAC Rule 3745-300-01(A)(78).

19.8 INSTRUMENT DETECTION LIMITS (IDL)

19.8.1 The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

19.8.2 IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either by using seven replicate spike analyses, like MDL but without sample preparation, or by the analysis of ten instrument blanks and calculating three times the absolute value of the standard deviation.

19.8.3 If IDL is > than the MDL, it may be used as the reported MDL.

19.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

19.9.1 Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP NC-QA-021 or Corporate CA-Q-S-006 for further details.

19.9.2 The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept in each department. Complete details are available in the laboratory SOPs.

19.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, atomic absorption, or fluorescence profiles.

19.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

19.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 ± 0.5 mg/l.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement, e.g., 524.2, 525, etc., and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.13.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the QuantIMS LIMS which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes AS400 which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.13.1.1 Maintain the Database Integrity

Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.

- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.

19.13.1.2 Ensure Information Availability

Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.13.1.3 Maintain Confidentiality

Ensure data confidentiality through physical access controls, and encryption of when electronically transmitting data.

19.13.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved, e.g., extractions, dilutions, instrument readings and concentrations. The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by peer review once updated in LIMS. The review checklists are signed by both the analyst and reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it must not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.13.2.1 All raw data must be retained. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.13.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. The units "mg/l" and "mg/kg" are the same as "parts per million (ppm)". The units "µg/l" and "µg/kg" are the same as "parts per billion (ppb)." For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%. Units are defined in each lab SOP.

19.13.2.3 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

19.13.2.4 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file.

19.13.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out in 'real time' and have enough information on them to trace the events of the applicable analysis/task (e.g., calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.).

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA Department. A record is maintained of all logbooks in the lab.

- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.13.4 Review / Verification Procedures

19.13.4.1 Data Recording Procedures

To ensure data integrity, all documentation of data and records generated or used during the process of data generation must be performed in compliance with Section 3 of this document and Policy CA-Q-T-005, Laboratory Documentation.

19.13.4.2 Data Reduction and Verification Procedures

Data review procedures comprise a set of computerized and manual checks applied at appropriate levels of the measurement process. Data review begins with the reduction or processing of data and continues through verification of the data and the reporting of analytical results. Calculations are checked from the raw data to the final value prior to reporting results for each group of samples. Data reduction can be performed by the analyst who obtained the data or by another analyst. Data verification starts with the analyst who performs a 100% review of the data to ensure the work was done correctly the first time. Data verification continues with review by a second reviewer who verifies that data reduction has been correctly performed and that the analytical results correspond to the data acquired and processed.

19.13.4.2.1 Data Reduction and Initial Verification

Data reduction and initial verification may be performed by more than one analyst depending upon the analytical method employed. The preparation and analytical data may be reviewed independently by different analysts. In these instances, each item may not be applicable to the subset of the data verified or an item may be applicable in both instances. It is the responsibility of the analyst to ensure that the verification of data in his or her area is complete. The data reduction and initial verification process must ensure that:

- Sample preparation information is correct and complete including documentation of standard identification, solvent lot numbers, sample amounts, etc.
- Analysis information is correct and complete including proper identification of analysis output (charts, chromatograms, mass spectra, etc.)
- Analytical results are correct and complete including calculation or verification of instrument calibration, QC results, and qualitative and quantitative sample results with appropriate qualifiers
- The appropriate SOPs have been followed and are identified in the project and/or laboratory records
- Proper documentation procedures have been followed
- All non-conformances have been documented
- Special sample preparation and analytical requirements have been met.
- The data generated have been reported with the appropriate number of significant figures as defined by the analytical method in the LIMS or

otherwise specified by the client.

In general, data will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- Input of raw data for computer processing
- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an analyst, all steps in the computation must be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, they may be attached to the data sheets.

Manual integrations are sometimes necessary to correct misintegrations by an automatic data system software program, but must only be performed when necessary. Further discussion of manual integrations and the required documentation is given in Policy CA-Q-S-002, Acceptable Manual Integration Practices.

For data that are input by an analyst and processed using a computer, a copy of the input must be kept and uniquely identified with the project number and other information as needed. The samples analyzed must be clearly identified.

If data are directly acquired from instrumentation or a test procedure and processed, or immediately entered into LIMS, the analyst must verify that the following are correct:

- Project and sample numbers
- Calibration constants and RFs
- Units
- Numerical values used for reporting limits.

Analysis-specific calculations for methods are provided in SOPs. In cases where computers perform the calculations, software must be validated or verified, as described in Section 6.0 of this document, before it is used to process data.

The data reduction is documented, signed and dated by the analyst completing the process. Initial verification of the data reduction by the same analyst is documented on a data review checklist, signed and dated by the analyst.

19.13.4.2.2 Data Verification

Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data that has been fully reduced and checked through Level 1 review is performed by an experienced peer, group leader, or designee. This Level 2 check is performed to ensure that Level 1 review has been completed correctly and thoroughly. The second level

reviewer examines the data signed by the analyst. Any exceptions noted by the analyst must be reviewed. Included in this review is an assessment of the acceptability of the data with respect to:

- Adherence of the procedure used to the requested analytical method SOP
- Correct interpretation of chromatograms, mass spectra, etc.
- Correctness of numerical input when computer programs are used (checked randomly)
- Correct identification and quantitation of constituents with appropriate qualifiers
- Numerical correctness of calculations and formulas (checked randomly)
- Acceptability of QC data (100% review)
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.)
- Documentation of dilution factors, standard concentrations, etc.
- Sample holding time assessment.

This review also serves as verification that the process the analyst has followed is correct in regard to the following:

- The analytical procedure follows the methods and client-specific instructions.
- Nonconforming events have been addressed by corrective action as defined on a nonconformance memo
- Valid interpretations have been made during the examination of the data and the review comments of the initial reviewer are correct
- The package contains all of the necessary documentation for data review and report production and results are reported in a manner consistent with the method used for preparation of data reports.

The specific items covered in the second stage of data verification may vary according to the analytical method, but this review of the data must be documented by signing the same checklist.

19.13.4.2.3 Completeness Verification

A third-level review is performed by the reporting and project management staff. This review is required before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. The items to be reviewed are:

- Analysis results are present for every sample in the analytical batch, reporting group, or sample delivery group (SDG)

- Every parameter or target compound requested is reported with either a value or reporting limit
- All nonconformances, including holding time violations, and data evaluation statements that impact the data quality are accompanied by clearly expressed comments from the laboratory

- The final report contains all the supporting documentation required by the project, and is in either the standard TestAmerica format or in the client-required

format.

- Implement checks to monitor the quality of laboratory results using correlation of results for different parameters of a sample (for example, does the TOC results justify the concentration of organic compounds found by GC/MS.)

- A narrative to accompany the final report must be finalized by the PM. This narrative must include relevant comments collected during the earlier reviews.

The Quality Assurance Department performs data reviews per SOP CA-Q-S-004, Internal Auditing. For DoD work, 10% of all reports must undergo an internal data review.

19.13.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002).

19.13.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.13.5.2 Analysts must not increase or decrease peak areas to for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

19.13.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.13.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate-approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example - Demonstration of Capability Documentation

Analyst Demonstration of Capability
Certification Statement

Analyst Name
Date:

Test Method:
SOP:
Matrix:

TestAmerica North Canton laboratory
4101 Shuffel Drive NW
North Canton, OH 44720
(330) 497-9396

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the TestAmerica Quality Assurance Plan, has met the Initial or Ongoing Demonstration of Capability.

2. The test method was performed by the analyst identified on this certification following the TestAmerica SOP.

3. A copy of the laboratory-specific SOP is available for all personnel on-site.

4. The data associated with the initial/ongoing demonstration of capability are true, accurate, complete and self-explanatory (*). These data are attached to this certification statement.

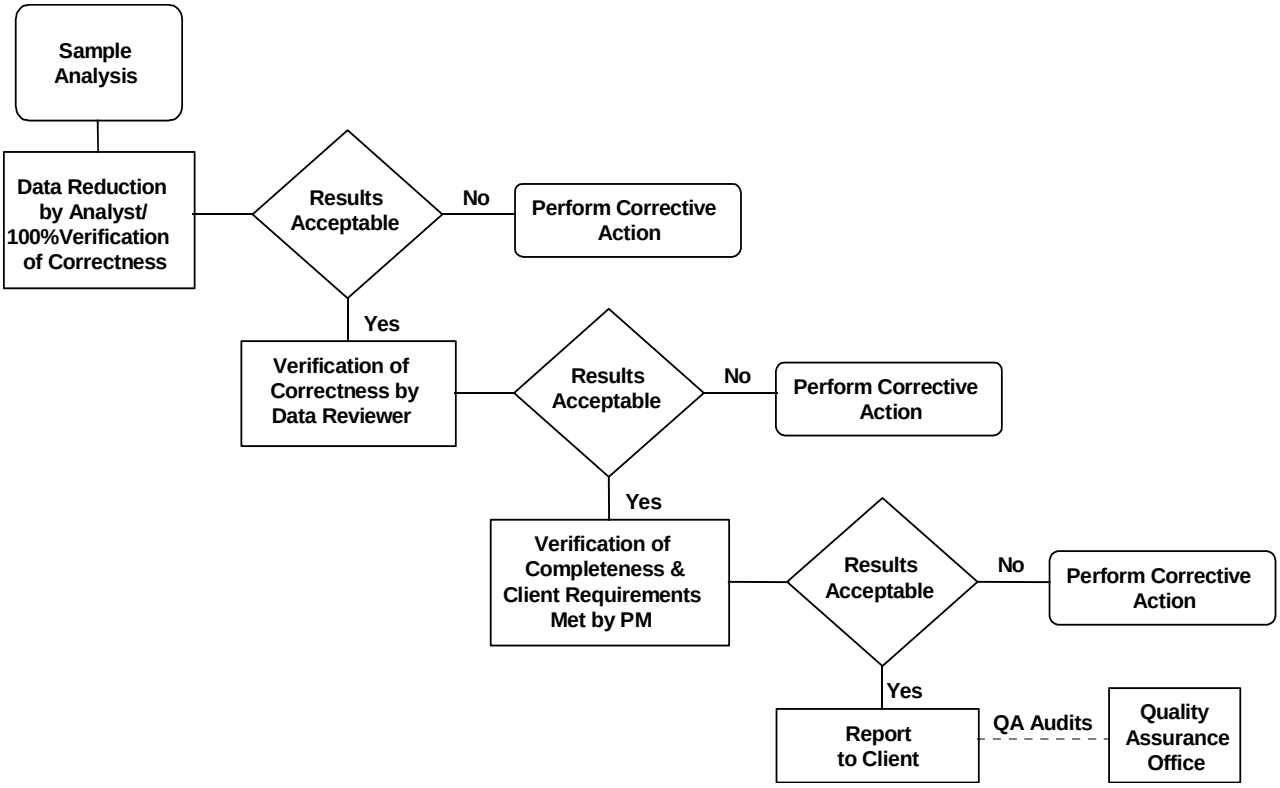
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

Comments/Observations:

Analyst's Name	Signature	Date
Technical Director's Name	Signature	Date
QA Manager's Name	Signature	Date

* *True: Consistent with supporting data.*
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance testing.
Self-explanatory: Data properly labeled and stored so that the results are traceable and require no additional explanation.

Figure 19-2. Work Flow



SECTION 20

EQUIPMENT (AND CALIBRATIONS)
(NELAC 5.5.5)

20.1 OVERVIEW

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 20-1*.*

Equipment is only operated by authorized and trained personnel. Manufacturers instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 PREVENTIVE MAINTENANCE

20.2.1 The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

20.2.2 Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

20.2.3 Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Group Leader to ensure instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also be outlined in analytical SOPs or instrument manuals. (Note: For some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

20.2.4 Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs must be kept for all major pieces of equipment. Instrument Maintenance Logbooks may also be used to specify instrument parameters.

20.2.4.1 Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

20.2.4.2 Each entry in the instrument log includes the Analyst's initials, date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control, e.g., CCV run on 'date' was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

20.2.4.3 When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled-in page must be signed across the page entered and the logbook, so it is clear that a page is missing if only half a signature is found in the logbook.

20.2.5 Instrument Repair

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it must be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory must examine the effect of this defect on previous analyses.

20.2.6 Equipment Malfunction

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Backup instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the backup is not available and the analysis cannot be carried out within the needed timeframe, the samples must be subcontracted.

20.2.7 Instrument Transfer or Send-Out

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

20.3 SUPPORT EQUIPMENT

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, dispensing devices, if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM Type 1 weights spanning its range of use (weights that have been calibrated to ASTM Type 1 weights may also be used for daily verification). ASTM Type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed. They are calibrated at least every five years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM Type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Reference SOP NC-QA-015, Equipment Monitoring and Thermometer Calibration. A list of balances is in Table 21.2.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to $\pm$ 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes, and thermocouples are calibrated quarterly.

The NIST thermometer is recalibrated every three years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s)have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logsheets. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logsheets. More information on this subject can be found in SOP NC-QA-015, Equipment Monitoring and Thermometer Calibration.

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day (seven days a week for DOD labs).

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between or 4 $\pm$ 2^{o}C.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logsheets posted on each unit and method-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are given unique identification numbers and the deliveery volumes are verified gravimetrically, at a minimum, on a quarterly basis. Glass micro-syringes are considered the same as Class A glassware.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy.

The laboratory maintains a sufficient inventory of autopipettors, and dilutors of differing capacities that fulfill all method requirements.

These devices are given unique identification numbers, and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

Any device not regularly verified cannot be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy.

20.3.7 Field Sampling Devices (ISCO Autosamplers)

Each autosampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is recorded on the sampling documentation in a logbook.

The autosampler is calibrated semi-annually by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The autosampler is programmed to run three cycles, and each of the three cycles is measured into a beaker to verify 100 ml are received.

If the RSD (Relative Standard Deviation) between the three cycles is greater than 20%, the procedure is repeated. If the result is still greater than 20%, the following options may be employed:

1) The unit is taken out of service.
2) The unit is used to pull composite samples only over a 24-hour period.

The results of this check are kept in a logbook/binder.

20.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, action is performed and any affected samples are re-analyzed if possible. If the re-analysis is not possible, any data associated with an unacceptable initial calibration must be reported with appropriate data qualifiers (refer to Section 12). For the Ohio EPA Voluntary Action Program (VAP), please refer to the SOPs for the acceptable criteria, corrective actions, and exceptions.

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 CALIBRATION STANDARDS

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to 3 significant figures) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.2 Calibration Verification

The calibration relationship established during the initial calibration must be verified at initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinataive methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS), then bracketing standards are not required. Only daily verifications are needed. The results from these verification standards must meet the calibration veirfication criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples or injections include matrix or batch QC samples.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

20.4.2.1 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. For Ohio VAP samples, results may not be reported when calibration verifications are exceeded low.

20.5 TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this

type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

20.6 GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Laboratory Equipment and Instrumentation

Instrument Type	Manufacturer	Model/Serial No.	Year into Service
GC/MS Volatiles	Hewlett-Packard (UX2)	5971A-5890, S/N 3140A38490	1992
	Hewlett-Packard (HP6)	5973-6890, S/N US00005571	2006
	Hewlett-Packard (UX7)	5973-6890, S/N US00010937	1998
	Hewlett-Packard (UX8)	5973-6890, S/N US00027773	1999
	Hewlett-Packard (UX9)	5973-6890, S/N US00028329	2000
	Hewlett-Packard (UX10)	5973-6890, S/N US00032072	2000
	Agilent (UX11)	5973-6890, S/N US00038093	2000
	Agilent (UX12)	5973-6890, S/N US10202133	2002
	Agilent (UX14)	5973-6890, S/N CN10340027	2003
	Agilent (UX15)	5973-6890, S/N CN10515062	2005
	Agilent (UX16)	5975-6890, S/N CN10539065	2005
GC/MS Volatiles Autosampler	OI Analytical (UX2)	4552	1999
	OI Analytical (HP6)	4552	1998
	OI Analytical (UX7)	4552	1998
	OI Analytical (UX8)	4552	1999
	OI Analytical (UX9)	4552	2000
	OI Analytical (UX10)	4552	2000
	OI Analytical (UX11)	4552	2000
	OI Analytical (UX12)	4552	2002
	OI Analytical (UX14)	4552	2003
	OI Analytical (UX15)	4552	2005
	OI Analytical (UX16)	4552	2005
GC/MS Volatiles Purge and Trap	OI Analytical (UX2)	4560	1999
	OI Analytical (HP6)	Encon	1998
	OI Analytical (UX7)	4660	2004
	OI Analytical (UX8)	4560	2004
	OI Analytical (UX9)	4560	2000
	OI Analytical (UX10)	4660	2003
	OI Analytical (UX11)	4560	2000
	OI Analytical (UX12)	4560	2002
	OI Analytical (UX14)	4560	2003
	OI Analytical (UX15)	4660	2005

Instrument Type	Manufacturer	Model/Serial No.	Year into Service
	OI Analytical (UX16)	4660	2005
	EST (spare)	4560	2002
GC/MS Semivolatiles	Hewlett-Packard HP7	5973-6890, S/N US00009247	1998
	Hewlett-Packard HP8	5973-6890, S/N US00022627	1999
	Hewlett-Packard HP9	5973-6890, S/N US00027943	2000
	Agilent HP10	5973-6890, S/N CN10340002	2003
	Agilent A4AG2	5975-C, S/N CN10721110	2007
GC Volatiles	Agilent (A)	6890 PID/FID, S/N US10402056	2004
	Hewlett-Packard (O)	6890 Dual PID/Hall, S/N US00007206	1997
	Hewlett-Packard (P)	6890 PID/HALL, S/N US00030616	1997
	Hewlett-Packard (Y)	6890N PID/FID, S/N US10337062	2003
	Agilent (Z)	6890 EPC & PDD/FID, S/N 10205072	2000
	Agilent (N)	7890 Atomic Fluorescence, S/N CN10820009	2008
GC Volatiles Auto Sampler	OI Analytical (O)	Archon	2000
	OI Analytical (Y)	4552	1998
	Varian (A)	Archon	1998
	Varian (P)	4552	2000
	Agilent (Z)	7694	2000
	EST (N)	Centurion	2008
GC Volatiles Purge & Trap	Tekmar (O)	4560	2000
	Tekmar (P)	3000	1993
	OI Analytical (A)	4560	1998
	OI Analytical (Y)	3000	1993
	Tekmar (N)	Stratum	2008
GC Semivolatiles	Hewlett-Packard (P1)	6890 EPC & Dual ECD Y-Splitter S/N US00023208	1998
	Hewlett-Packard (P2)	6890 EPC & Dual ECD Y-Splitter S/N US00023512	1998
	Hewlett-Packard (P3)	6890 EPC & Dual ECD Y-Splitter S/N US00023674	1998
	Hewlett-Packard (P4)	6890 EPC & Dual ECD Y-Splitter S/N US00029531	1999
	Hewlett-Packard (P6)	6890 EPC & Dual FID S/N US00032848	2000

Instrument Type	Manufacturer	Model/Serial No.	Year into Service
	Agilent (P7)	6890N EPC & Dual FID S/N US10202132	2000
	Agilent (P9)	6890N EPC & Dual ECD Y-Splitter S/N US10205045	2005
	Agilent (P10)	6890 EPC & Dual ECD Y-Splitter S/N US10151110	1999
	Agilent (P11)	6890N EPC & Dual ECD Y-Splitter S/N CN10517088	2004
	Agilent (P12)	6890N EPC & Dual ECD Y-Splitter S/N CN10512025	2005
	Agilent (P13)	6890N EPC & Dual ECD Y-Splitter S/N CN10435032	2004
GC Semivolatiles HPLC	Hewlett-Packard (L2)	HPLC 1100, S/N US82404153	1998
Extractions Sonicators	Misonix	3000 (self-tuning), S/N R1044	2005
	Misonex	Ultrasonic Processor XL, S/N G4221	
	Heat Systems	XL2020, S/N G1026	2001
Extractions 6-Position Accelerated Soxhlet Extractor	Gerhardt Soxtherm	8 units	2003
Extractions pH Meter	Thermo Orion	420, S/N 074028	1998
	Mettler Toledo	SevenEasy pH (self-calibrating) S/N 1228295055	2008
	Denver Instrument (spare)	UB-5 S/N UB-5093011	2004
Metals ICP	Thermo Jarrell Ash (I-5)	Trace Analyzer 61E, S/N 273490	1994
	Thermo Jarrell Ash (I-6)	Trace Analyzer 61E, S/N 269490	1994
Metals ICP/MS	Thermo (I-8)	Series 2, S/N 01137C	2007
Metals Mercury Analyzer	Leeman (CVAA) (H1)	PS200 II, S/N HG9031	1999
	Leeman (CVAF) Low Level (H3)	Hydra AF Gold+, Model #112-00067-1 S/N AFG1006	2001
	Leeman (CVAA) (H4)	Hydra AA , S/N 6011	2006
WC Arsenic Speciation	Trace Detect	Nano-Band Explorer, S/N NBE00017	2003
WC Autotitrator	Man-Tech	PC – Titrate, S/N MS-9K8-217	2001

Instrument Type	Manufacturer	Model/Serial No.	Year into Service
WC Block Digester	Andrews	2210 Phenol	1999
	Andrews	2205 Ammonia	1999
	Lachat	BD46 TKN	1992
WC BOD	Mantech	BOD, S/N N817947	1999
WC Conductivity	Man-Tech	4310, S/N 1613	1989
WC Cyanide	LabCrest MidiDist	PRG-2520-BL, S/N 1000-99-01	1999
WC Discrete Analyzer	Kone	Konelab 200, Z1718383	2001
	Kone	Konelab 250, A2120021	2005
WC Dissolved Oxygen Meter	YSI	52C E, S/N 99C1094	1993
WC Flashpoint	Herzog	HFP 339, S/N 073390084	2007
	Petrolab (199443)	Petrotest, S/N 0741990603	1999
WC Ion Chromatograph	Dionex	DX-320, S/N 00060187	2001
	Dionex	DX-120, S/N 98110093	1999
WC pH Meter	Orion pH Meter	320, S/N 020032	2007
	Orion (Ammonia ISE)	520A, S/N 48029	1996
WC Residual Chlorine Meter	Hanna	HI 93701	2000
	Hanna	HI 93701	2006
WC TOC	OI Analytical	1010 TOC Analyzer, S/N K503710931	2005
WC TOX	Thermo Electron	1200, S/N 973515	1998
	Thermo Electron	1200, S/N 2001.0174	2005
	Thermo Electron	1200, S/N 2005.0234	2005
WC TRAACS	Bran & Luebbe	800, S/N 890140	1993
WC Turbidimeter	HF Scientific	Micro 100, S/N 200705143	2001
WC UV / VIS	Milton Roy	Spectronic 401, S/N 381A170002	1993
	Genesys	Spectronic 20, S/N 3SGL078016	1998
	Genesys	Spectronic 20, S/N 3SCA174040	2008
	Genesys	Spectronic 20, S/N 3SGL226006 (Model 4001/4)	2008
WC Sulfide	Westco EasyDist		2008

Table 20-2. Schedule of Routine Maintenance
(Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations)

INSTRUMENT MAINTENANCE SCHEDULE
ION CHROMATOGRAPH

As Needed	Daily	Weekly	Monthly
Clean micro-membrane suppressor when decreases in sensitivity are observed.	Check plumbing/leaks.	Check pump heads for leaks.	Check all air and liquid lines for discoloration and crimping, if indicated.
Check fuses when power problems occur.	Check gases.	Check filter (inlet)	Check/change bed supports guard and analytical columns, if indicated.
Reactivate or change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated.	Check pump pressure.		
De-gas pump head when flow is erratic.	Check conductivity meter.		

INSTRUMENT MAINTENANCE SCHEDULE
TOTAL ORGANIC HALIDE ANALYZER

Daily	**As Needed**
Check electrodes for damage; polish the electrodes.	Examine and clean or replace pyrolysis tube.
Replace dehydrating fluid and electrolyte fluid.	Clean titration cell.
Clean quartz boat.	Observe gas flow.
Observe check valves during use for backfeed.	Replace reference electrode fluid.
At end of each day of use, wash out absorption module, empty electrolyte and fill cell with DI water. Empty dehydrator tube	Change quartz wool.
Perform cell performance check.	Replace O-rings and seals.

INSTRUMENT MAINTENANCE SCHEDULE
HIGH PRESSURE LIQUID CHROMATOGRAPH

Daily	**As Needed**
Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse gas and delivery lines to prevent contamination of the new solvent.	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements.
Check gas supply.	Oil autosampler slides when sample does not advance.
Flush with an appropriate solvent to remove all bubbles.	Rinse flow cell with 1N nitric acid if sensitivity low.
Pre-filter all samples.	Change pump seals when flow becomes inconsistent.
	Repack front end of column Backflush column.

INSTRUMENT MAINTENANCE SCHEDULE
ICP AND ICP/MS

Daily	Monthly or As Needed	Semi-Annually	Annually
Check vacuum pump gage. (<10 millitorr)	Clean plasma torch assembly to remove accumulated deposits	Change vacuum pump oil	Notify manufacturer service engineer for scheduled preventive maintenance service
Check cooling water supply system is full and drain bottle is not full. Also drain tubing is clear, tight fitting, and has few bends.	Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance	Replace coolant water filter (may require more or less frequently depending on quality of water)	
Check nebulizer is not clogged	Clean filters on back of power unit to remove dust		
Check capillary tubing is clean and in good condition	Replace when needed: - peristaltic pump tubing - sample capillary tubing - autosampler sipper probe		
Check peristaltic pump windings are secure	- Check yttrium position - Check O-rings - Clean/lubricate pump rollers		
Check high voltage switch is on			
Check torch, glassware, aerosol injector tube, and bonnet are clean			

INSTRUMENT MAINTENANCE SCHEDULE
CVAS AND CVAFS

Daily	As Needed	Annually
Change drying tube	Change pump tubing	Change Hg lamp
Check pump tubing/drain tubing	Check/change Hg lamp	
Check gas pressure	Clean optical cell	
Check aperture reading Check tubing	Lubricate pump	

INSTRUMENT MAINTENANCE SCHEDULE
GAS CHROMATOGRAPH

Daily *	**As Needed**
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance, or when column performance (e.g., peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Quarterly FID: clean detector, only as needed—not quarterly/or semi-annually.
Check temperatures of injectors and detectors. Verify temperature programs by RT shift.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.
Clean injector port weekly for TPH for 8015B, when breakdown fails; otherwise, when RT shift or bad samples run.	Annually FID: replace flame tip, only as needed. Only as needed: ECD--detector cleaning and re-foiling, whenever loss of sensitivity, erratic response, or failing resolution is observed
Check baseline level during analysis of run—not maintenance.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).
Watched weekly: check reactor temperature of electrolytic conductivity detector. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks, when analyzing pesticides; part of analysis—not maintenance. Clip column leader when chromatography looks bad—not daily.	Replace or repair flow controller if constant gas flow cannot be maintained. Replace fuse. Reactivate external carrier gas dryers. Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. ECD: follow manufacturer's suggested maintenance schedule. Reactivate flow controller filter dryers when presence of moisture is suspected. HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.

*No daily maintenance done on any instrument/method. Weekly change IPL on TPH instrument. Everything else is on an "as needed" basis.

INSTRUMENT MAINTENANCE SCHEDULE
MASS SPECTROMETER

Daily	Weekly	As Needed	Quarterly	Annually
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43)	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between maintenance.	Check ion source and analyzer (clean, replace parts as needed)	Replace the exhaust filters on the mechanical rough pump every 1-2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.	Check vacuum, relays, gas pressures and flows	
Check inlets, septa		Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.	Change oil in the mechanical rough pump.	
Check baseline level		Repair/replace jet separator.		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates need for replacement.		

INSTRUMENT MAINTENANCE SCHEDULE
TRAACS AUTO ANALYZER

As Needed	Daily
Replaces air filter when progressive loss of air pressure is observed.	Check air pressure gauge (22 ± 2 psi)
Replace air valve tubing when occlusion in tubing is observed	Use recommended washout procedure (at end of analysis operations)
Change all pump tubes (or after 200 hours of pumping time, or after 1000 hours of pumping time)	
Clean sample probe shaft	
Replace pump platens	
Lightly lubricate the linear sample rails (use semi-fluid lubricant)	
Replace colorimeter lamp (or after 2500 hours of use)	

INSTRUMENT MAINTENANCE SCHEDULE
ANALYTICAL/TOP LOADING BALANCES

Daily	Annually
Check using Class 1-verified weights once daily or before use Clean pan and weighing compartment	Manufacturer cleaning and calibration

INSTRUMENT MAINTENANCE SCHEDULE
REFRIGERATORS/WALK-IN COOLERS

Daily	As Needed
Temperatures checked and logged	Refrigerant system and electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
OVENS

Daily	As Needed
Temperatures checked and logged	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
SPECIFIC DIGITAL ION ANALYZER

Daily	As Needed
Daily when used: • Calibrate with check standards • Inspect electrode daily, clean as needed • Inspect electrode proper levels of filling solutions daily; fill as needed • Clean probe after each use	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
TURBIDIMETER

Daily	Monthly	As Needed
Daily when used: • Adjust linearity on varying levels of NTU standards. Standardize with NTU standards • Inspect cells	Clean instrument housing	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
DISSOLVED OXYGEN METER

Daily	As Needed
Daily when used: • Calibrate with saturated air • Check probe membrane for deterioration • Clean and replace membrane with HCl solution	• Electronics serviced • Clean and replace membrane with HCl solution

INSTRUMENT MAINTENANCE SCHEDULE
CONDUCTANCE METER

Daily	As Needed
Daily when used: • Check probe and cables • Inspect conductivity cell	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
CHEMICAL OXYGEN DEMAND (COD) REACTOR [1]

Daily	As Needed
Daily when used: • Calibrate with check standards	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
SPECTROPHOTOMETER

As Needed	Daily	Monthly	Annually
Dust the lamp and front of the front lens	Check the zero % adjustment	Clean windows	Check instrument manual
	Clean sample compartment		Perform wavelength calibration
	Clean cuvettes		Replace lamp annually or when erratic response is observed
			Clean and align optics

INSTRUMENT MAINTENANCE SCHEDULE
pH METER

As Needed	Daily
Clean electrode	Inspect electrode. Verify electrodes are properly connected and filled
Refill reference electrode	Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer

INSTRUMENT MAINTENANCE SCHEDULE
TOTAL ORGANIC CARBON ANALYZER

Daily	As Needed	Weekly	Monthly
Check: • Oxygen supply • Persulfate supply • Acid supply • Carrier gas flow rate (~ 150 cc/min) • IR millivolts for stability (after 30 min. warm-up) • Reagent reservoirs	Check injection port septum after 50-200 runs Tube end-fitting connections after 100 hours or use Indicating drying tube NDIR zero, after 100 hours of use Sample pump, after 2000 hours for use Digestion vessel/condensation chamber, after 2000 hours of use Permeation tube, after 2000 hours of use NDIR cell, after 2000 hours of use Change pump tubing	Check liquid-flow-rate-pump-tubing conditions on autosampler Check injection port septum	Clean digestion vessel Clean condenser column Do the leak test

Instrument Maintenance Schedule
Digestion Block

Annually
Check temperature with NIST thermometer

Instrument Maintenance Schedule
Flash Point Tester

Daily
Check tubing Clean sample cup each use
Check gas
Clean flash assembly
Check stirrer

Table 20-3. Preventive Maintenance Procedures

SUMMARY OF INORGANIC METHOD CALIBRATIONS

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Acidity	Initial	350.1	Two-level calibration that bracket the expected pH of the sample ± 0.05 pH units of true value	9040B 9045C	2 point calibration ± 0.05 pH units of true value
	Continuing	350.1	One buffer check every 10 samples ± 5% of true value	9040B 9045C	N/A
	Other	350.1	Third point check	9040B 9045C	Third point check
	Ending	350.1	One buffer check ± 5% of true value	9040B 9045C	N/A
Alkalinity, Bicarbonate, Carbonate	Initial	310.1 2320B	2 point calibration of pH meter ± 0.05 pH units of true value	--	N/A
	Continuing	310.1 2320B	N/A	--	N/A
	Ending	310.1 2320B	N/A	--	N/A
Ammonia	Initial	350.1	6 levels including blank, "r" [3] ≥ 0.995	--	N/A
	Continuing	350.1	One level or LCS every 10 samples ± 10% of true value	--	N/A
	Ending	350.1	One level or LCS every 10 samples ± 10% of true value	--	N/A
Arsenic Speciation		N/A	N/A	7063	* Refer to Section 10 of SOP NC-WC-0090

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Biochemical Oxygen Demand (BOD)	Initial	405.1 SM5210B	a. Winkler titration: Iodometric with standard thiosulfate b. Membrane electrode: Read in air and in water with zero dissolved oxygen	--	N/A
	Continuing	405.1 SM5210B	N/A	--	N/A
	Ending	405.1 SM5210B	N/A	--	N/A
Bromide	Initial	300.0A	5 levels plus a blank, "r"[3] ≥ 0.995	9056A	5 levels plus a blank, "r" [3] ≥ 0.995
	Continuing	300.0A	Level every 10 samples ± 10% of true value	9056A	N/A
	Ending	300.0A	N/A	9056A	N/A
Cation Exchange	Initial	N/A	N/A	9081	N/A
	Continuing	N/A	N/A	9081	N/A
	Ending	N/A	N/A	9081	N/A
Chemical Oxygen Demand (COD)	Initial	410.4 SM5220D	5 levels plus a blank"r" [3] ≥ 0.995	--	N/A
	Continuing	410.4 SM5220D	One level every 10 samples ± 10% of true value	--	N/A
	Ending	410.4 SM5220D	One level ± 10% of true value	--	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Chloride	Initial	300.0A 325.2	5 levels plus blank "r" [3] ≥ 0.995	9056A 9251	Method 9056: 3 levels plus a blank Method 9252: 5 levels plus blank "r" [3] ≥ 0.995
	Continuing	300.0A 325.2	One level every 10 samples ± 10% of true value	9056A 9251	Method 9056: One per batch of 20 samples, ± 10% of true value Method 9252: One level every 10 samples, ± 10% of true value
	Ending	300.0A 325.2	One level every 10 samples ± 10% of true value	9056A 9251	Method 9056 : N/A Method 9252: One level ± 10% of true value
Chromium Cr^{+6}	Initial	3500 Cr-D	3 levels plus blank	7196A	5 levels plus blank "r" [3] ≥ 0.995
	Continuing	3500 Cr-D	One level every 10 samples ± 10% of true value	7196A	One level every 10 samples ± 15%
	Ending	3500 Cr-D	One level ± 10% of true value	7196A	One level ± 15%
Chlorine, Residual	Initial	330.5 SM3500CL-G	N/A	--	N/A
	Continuing	330.5 SM3500CL-G	N/A	--	N/A
	Ending	330.5 SM3500CL-G	N/A	--	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Conductivity	Initial	120.1 SM2510B	Standard KCl solution	9050A	One level to determine cell constant
	Continuing	120.1 SM2510B	N/A	9050A	N/A
	Ending	120.1 SM2510B	N/A	9050A	N/A
Cyanide (Amenable)	Initial	335.1 SM4500CN-G	6 levels plus blank "r" [3] ≥ 0.995	9012A	6 levels plus blank "r" [3] ≥ 0.995
	Continuing	335.1 SM4500CN-G	One level every 10 samples ± 10% of true	9012A	One mid-level every 10 samples ± 15% of true value
	Ending	335.1 SM4500CN-G	One level ± 10 % of true value	9012A	± 15% of true value
Cyanide (Total)	Initial	335.1/335.2 335.3/335.4 SM4500CU-E/ 335.2-CLP-M	6 levels plus blank "r" [3] ≥ 0.995	9012A	6 levels plus blank "r" [3] ≥ 0.995
	Continuing	335.1/335.2 335.3/335.4 SM4500CU-E/ 335.2-CLP-M	One mid-level every 10 samples ± 10 % of true value	9012A	One mid-level every 10 samples ± 15% of true value
	Ending	335.1/335.2 335.3/335.4 SM4500CU-E/ 335.2-CLP-M	One mid-level ± 10 % of true value	9012A	± 15% of true value
Flashpoint	Initial	--	N/A	1010 ASTM D93-9	p-Xylene reference standard must have flashpoint of 81°F ±2°F
	Continuing	--	N/A	1010 ASTM D93-9	N/A
	Ending	--	N/A	1010 ASTM D93-9	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Fluoride	Initial	300.0A 340.2	Method 300.0A: 5 levels plus a blank, "r" [3] ≥ 0.995 Method 340.2: 5 levels "r" [3] ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 340.2	One mid-level every 10 samples ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 340.2	One mid-level ± 10% of true value	9056A	N/A
Hardness	Initial	130.2 2340B	Method 130.2: Standardize titrant Method 2340B: See ICP Metals 200.7	--	N/A
	Continuing	130.2 2340B	Method 130.2: N/A Method 2340B: See ICP Metals 200.7	--	N/A
	Ending	130.2 2340B	Method 130.2: N/A Method 2340B: See ICP Metals 200.7	--	N/A
Iron (Ferrous)	Initial	3500-Fe D	3 levels plus a blank, "r" [3] ≥ 0.995	-	N/A
	Continuing	3500-Fe D	One mid-level every 10 samples ± 10% of true value	-	N/A
	Ending	3500-Fe D	One mid-level ± 10% of true value	-	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Nitrate	Initial	300.0A 353.2 SM4500NO_3-E	5 levels plus a blank "r" [3] ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 353.2 SM4500NO_3-E	One mid-level every 10 samples ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 353.2 SM4500NO_3-E	One mid-level ± 10% of true value	9056A	N/A
Nitrite	Initial	300.0A 354.1	5 levels plus a blank "r" [3] ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 354.1	One mid-level every 10 samples ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 354.1	One mid-level ± 10% of true value	9056A	N/A
Nitrate-Nitrite	Initial	300.0A 353.2	5 levels plus blank "r" [3] ≥ 0.995	--	N/A
	Continuing	300.0A 353.2	One level every 10 samples ± 10% of true value	--	N/A
	Ending	300.0A 353.2	One mid-level ± 10% of true value	--	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Phosphorus (Total and Ortho-phosphate)	Initial	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: 3 levels plus a blank Method 365.2: 5 levels plus a blank	--	N/A
	Continuing	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: One level every 10 samples ± 10% of true value Method 365.2: Blank and 2 standards with each series of samples, ± 2% of true value or recalibrate	--	N/A
	Ending	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: ± 10% of true value Method 365.2: N/A	--	N/A
pH	Initial	150.1 SM4500H-B	2 level calibration that bracket the expected pH of the sample ± 0.05 pH units of true value	9040B 9045C	2 point calibration ± 0.05 pH units of true value
	Continuing	150.1 SM4500H-B	One buffer check every 10 samples ± 5% of true value	9040B 9045C	N/A
	Other	150.1 SM4500H-B	Third point check	9040B 9045C	Third point check
	Ending	150.1 SM4500H-B	One buffer check ± 5% of true value	9040B 9045C	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Phenolics	Initial	420.1	5 levels plus a blank "r" [3] ≥ 0.995	9065 9066	5 levels plus a blank "r" [3] 0.995
	Continuing	420.1	One mid-level every 10 samples ± 10% true value	9065 9066	One mid-level ± 15% true value
	Ending	420.1	One mid-level ± 10% true value	9065 9066	One mid-level ± 15% true value
Phosphate	Initial	SM4500P-E	N/A	9056	3 levels plus a blank
	Continuing	SM4500P-E	N/A	9056	One per batch of 20 samples ± 15% of true value
	Ending	SM4500P-E	N/A	9056	N/A
Settleable Solids	Initial	160.5	N/A	--	N/A
	Continuing	160.5	N/A	--	N/A
	Ending	160.5	N/A	--	N/A
Specific Conductance	Initial	120.1 SM2510B	Standardize meter with 0.01 M KCl	9050A	N/A
	Continuing	120.1 SM2510B	One level every 10 samples ± 10% of true value	9050A	N/A
	Ending	120.1 SM2510B	One level ± 10% of true value	9050A	N/A
Sulfate	Initial	300.0A 375.4	Method 300.0A: 5 levels plus blank "r" [3] ≥ 0.995 Method 375.4: 3 levels plus blank "r" [3] ≥ 0.995	9038 9056A	Method 9038: 3 levels plus a blank for every hour of continuous sample analysis. Method 9056: 3 levels plus a blank

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Sulfate (Cont'd)	Continuing	300.0A 375.4	Method 300.0A: One mid-level after every 10 samples ± 10% of true value Method 375.4: One level every 3 or 4 samples ± 10% of true value	9038 9056A	Method 9038: Independent-prepared check standard every 15 samples Method 9056: 1 per batch of 20 samples ± 10% of true value
	Ending	300.0A 375.4	± 10% of true value	9038 9056A	N/A
Sulfide	Initial	376.1	Method 376.1: This is a titration method. Therefore, calibrations are not applicable.	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
	Continuing	376.1	Method 376.1: N/A	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
	Ending	376.1	Method 376.1: N/A	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
Total Dissolved Solids	Initial	160.1 SM2540E	This is a gravimetric determination. Calibrate balance prior to analysis	--	N/A
	Continuing	160.1 SM2540E		--	N/A
	Ending	160.1 SM2540E		--	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Total Kjeldahl Nitrogen (TKN)	Initial	351.3 $SM4500NO_3$	Method 351.3: Titrimetric: Standardize titrant Colorimetric: 7 levels plus blank	--	N/A
	Continuing	351.3 $SM4500NO_3$	Method 351.3: N/A	--	N/A
	Ending	351.3 $SM4500NO_3$	Method 351.3: N/A	--	N/A
Total Organic Carbon (TOC)	Initial	415.1 SM5310D	3 levels plus blank	9060 Walkley Black	3 levels plus blank "r" [3] ≥ 0.995
	Continuing	415.1 SM5310D	1 mid-level every 10 samples ± 15% of true value	9060 Walkley Black	1 mid-level every 10 samples ± 15% of true value
	Ending	415.1 SM5310D	± 15% of true value	9060 Walkley Black	± 15% of true value
Total Organic Halides (TOX)	Initial	450.1	Method 450.1: Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration standard) Verify with independently-prepared check standard	9020B 9023 (EOX)	Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration standard) Verify with independently-prepared check standard –ICV ± 10% SOP CORP-WC-0001

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Total Organic Halides (TOX) (cont'd)	Continuing	450.1	± 10% of true value	9020B 9023 (EOX)	CCV ± 10% of true value SOP CORP-WC-0001
	Ending	450.1	± 10% of true value	9020B 9023 (EOX)	CCV ± 10% of true value SOP CORP-WC-0001
Total Solids	Initial	160.3	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.3		--	N/A
	Ending	160.3		--	N/A
Total Suspended Solids (Nonfilterable)	Initial	160.2 SM2540E	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.2 SM2540E		--	N/A
	Ending	160.2 SM2540E		--	N/A
Turbidity	Initial	180.1	Minimum of 1 level in each instrument range. Follow manufacturer's instructions	--	N/A
	Continuing	180.1	N/A	--	N/A
	Ending	180.1	N/A	--	N/A
Volatile Solids	Initial	160.4	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.4		--	N/A
	Ending	160.4		--	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
ICP & ICP/MS Metals (excludes Hg)	Initial	200.7	One level and blank. ICV RSD <3% from replicate - daily	6010B	One level and blank. ICV RSD <5% from replicate - daily
	Initial	200.8	One level and blank	6020	One level and blank
	Continuing	200.7	Every 10 samples ±10% of true value CCV RSD < 5% from replicate	6010B	Mid-level calibration standard Every 10 samples ± 10% of true value CCV RSD < 5% from replicate
	Continuing	200.8	N/A	6020	N/A
	Ending	200.7	±10% of true value CCV RSD < 5% from replicate	6010B	Mid-level calibration standard ± 10% of true value CCV RSD < 5% from replicate
	Ending	200.8	N/A	6020	N/A
	Other	200.7	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Semi-Annually: ICP interelement correction factors Instrument detection limits	6010B	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Semi-Annually: ICP interelement correction factors Instrument detection limits
	Other	200.8	N/A	6020	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Mercury by CVAA & CVAFS	Initial	245.1 1631E	5 levels plus blank ICV ±10% of true value "r" [3] ≥ 0.995	7470A 7471A	5 levels plus blank ICV ± 10% of true value "r" [3] ≥ 0.995
	Continuing	245.1* 1631E	Daily or every 10 samples, whichever is more frequent ±20% of true value	7470A 7471A	Every 10 samples ±20% of true value
	Ending	245.1 1631E	±20% of true value	7470A 7471A	±20% of original prepared standard
	Other	245.1 1631E	Annually: MDL	7470A 7471A	Annually: MDL

* 245.1 continuing – Initial CCV ±5% of true value

Footnotes

[1] National Pollutant Discharge Elimination System.

[2] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December, 1996).

[3] "r" = correlation coefficient.

SUMMARY OF ORGANIC METHOD CALIBRATIONS

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Aromatic Volatiles by GC	Initial	602	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.
	Continuing	602	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 injections or 12 hrs whichever is greater, % D ≤ 15%, gases 20% D. Evaluate per SOP
	Ending	602	Run closer per NELAC requirement, but no criteria	8021B	Mid-level calibration standard, % D ≤ 15%. Evaluate per SOP, except as noted above.
	Other	602	N/A	8021B	N/A
Herbicides by GC	Initial	615 [9]	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8151A	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	615 [9]	One or more calibration standards analyzed daily % D ± 15% of predicted response	8151A	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	615 [9]	N/A	8151A	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.
	Other	615 [9]	N/A	8151A	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Pesticides/ PCBs by GC	Initial	608	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8081A 8082	Minimum of 5 levels. If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. (See SOP NC-GC-038)
	Continuing	608	One or more calibration standards analyzed daily. % D ± 15% of predicted response	8081A 8082	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	608	N/A	8081A 8082	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.
	Other	608	N/A	8081A 8082	N/A
Petroleum Hydrocarbons/ Oil and Grease	Initial	1664A	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be ± 10% at 2 mg and ± 0.5% at 1000 mg or recalibrate balance	9071B	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be ± 10% at 2 mg and ± 0.5% at 1000 mg or recalibrate balance
	Continuing	1664A	N/A	9071B	N/A
	Ending	1664A	N/A	9071B	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Purgeable Halocarbons by GC	Initial	601	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.
	Continuing	601	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 injections, or 12 hrs, whichever is greater. % D < 15%, gases 20% D Evaluate per SOP
	Ending	601	Run per NELAC, but no requirement to meet	8021B	Mid-level calibration standard, % D <15%. Evaluate per SOP
	Other	601	N/A	8021B	N/A
Halogenated Volatiles by GC	Initial	--	N/A	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	--	N/A	8021B	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	--	N/A	8021B	Mid-level calibration standard % D < 15% of predicted response for any analyte quantitated and reported.
	Other	--	N/A	8021B	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Semivolatiles	Initial	625	Minimum of 3 levels, lowest near but above MDL. If % RSD ≤ 35%, use avg RF. Otherwise calibration curve employed.	8270C	Minimum of 5 levels, % RSD for RF for CCCs[(4)] < 30% SPCCs[(5)]: RF > 0.050
	Continuing	625	One level every 24 ours. Acceptance criteria are found in the method and SOP.	8270C	Mid-level standard every 12 hours (after tuning) %D for CCCs[(4)] < 20 % between RF from standard and avg RF from initial SPCCs[(5)]: RF > 0.050.
	Ending	625	N/A	8270C	N/A
	Other	625	DFTPP[(7)] tuning every 24 hours before standard or sample runs.	8270C	DFTPP[(7)] tuning at the beginning of every 12 hour shift.

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Volatiles	Initial	624	Minimum of 3 levels, lowest near but above MDL. If % RSD ≤ 35%, use avg RF. Otherwise, calibration curve employed.	8260B	Minimum of 5 levels, %RSD for RF for CCCs[4] < 30.0% SPCCs[5]:RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform
	Continuing	624	1 level every 24 hours Acceptance criteria are found in the method and SOP	8260B	Mid-level standard every 12 hours (after tuning) %Drift for CCCs(4) < 20.0% between RF from standard and avg RF from initial SPCCs(5): RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform
	Ending	624	N/A	8260B	N/A
	Other	624	BFB(6)tuning at the beginning of every 24 hour shift.	8260B	BFB(6)tuning at the beginning of every 12 hour shift.

Footnotes:

1 National Pollutant Discharge Elimination System.

2 Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

3 TCDD - 2,3,7,8-Tetrachlorodibenzo-p-dioxin.

4 CCC - Continuing Calibration Compounds.

5 SPCC - System Performance Check Compound.

6 BFB – Bromofluorobenzene.

7 DFTPP – Decafluorotriphenylphosphine.

8 Footnote deleted.

9 Method not listed in 40 CFR Part 136.

SECTION 21

MEASUREMENT TRACEABILITY
(NELAC 5.5.6)

21.1 OVERVIEW

Traceability of measurements must be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard must be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices (refer to Section 20.3). With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Microsyringes are verified at least semi-annually or disposed of after six months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A glassware should be routinely inspected for chips, acid etching or deformity. If the Class A glassware is suspect, the accuracy of the glassware must be assessed prior to use. Actions to correct or segregate ancillary equipment that does not meet required specifications are identified in the calibration and maintenance section of SOPs and maintenance logbooks for the specific equipment.

21.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement must be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. (Refer to Section 9 for additional

information on purchasing). The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor-certified different lot is acceptable for use as a second source. For unique situations, where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g., calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to The Corporate Environmental Health & Safety Manual (CW-E-M-001) or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

21.4 DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to TestAmerica's Corporate SOP CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor-supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in each group. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96%, a correction must be made to concentrations applied to solutions prepared from the stock commercial material.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into standard or reagent logbooks, and are assigned a unique identification number. The following information is typically recorded in the Standards Logbook.

- Standard ID

- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation date
- Expiration date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent standard analyte concentration (if applicable)
- Parent standard amount used (if applicable)
- Component analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained in each group for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date, and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration date (include prep date for reagents)
- Standard ID (from the Standards Logbook)
- Special health/safety warnings, if applicable

21.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date, and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority:

1) With the manufacturer's recommendations
2) With requirements in the specific analytical methods as specified in the laboratory SOP

SECTION 22

SAMPLING
(NELAC 5.5.7)

22.1 OVERVIEW

The laboratory provides sampling services. Sampling procedures are described in SOP NC-SC-006, Sample Procurement Protocol.

22.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are available from the vendor electronically, or stored at the laboratory.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the Chain-of-Custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in “days” (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in “hours” (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends 24 hours after sampling. Holding times for analysis include any necessary re-analysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of holding time length.

Company Confidential & Proprietary

22.4 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method-required holding times (refer to Tables 23-1 to 23-7 and in SOPs) or preservation requirements are not met, the reports must be qualified using a flag, footnote, or case narrative. As soon as possible, or "ASAP", is an EPA designation for tests for which rapid analysis is advised; but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample needs consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative sub-sample or aliquot of the sample provided for analysis. In that regard the following guidelines apply to analysts:

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots and sub-sampling are located in each analytical SOP.

Tables 23-1 to 23-7 detail holding times, preservation and container requirements, and sample volumes for SDWA and NPDES methods. The sample volumes are intended to be a minimal amount to perform the method. The containers used may be of larger size.

Please note: The holding times are program specific and different programs may have different holding times for equivalent methods, e.g., there are differences in holding times for many organic analytes between SDWA and NPDES. RCRA methods may also be different.

Table 22-1. Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Acidity	Water	100 mL	305.1 SM2310B	250 mL plastic or glass. Cool to 4°C, 14 days	---	N/A
Alkalinity, Bicarbonate, Carbonate	Water	100 mL	310.1 2320B	250 mL plastic or glass. Cool to 4°C, 14 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Ammonia	Water	100 mL	350.1 350.2 SM4500NH_3-E SM4500NH_3-F	500 mL plastic or glass. Cool to 4°CH_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Arsenic (ASV) Anodic Stripping Voltammetry	Water	100 mL	---	N/A	7063	250 mL plastic. Cool to 4°C. HCl to pH <2, 28 days
Biochemical Oxygen Demand (BOD), Carbonaceous	Water	1000 mL	405.1 5210B	1000 mL plastic or glass. Cool to 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Bromide	Water	50 mL	300.0A[7]	250 mL plastic or glass. No preservative required, 28 days	9056A	Cool to 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Cation Exchange	Solid	8 oz	---	N/A	9081	8 or 16 oz glass. Cool to 4°C, 6 months
Chemical Oxygen Demand (COD)	Water	100 mL	410.4 5220D	250 mL glass or plastic. Cool to 4°C, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Chloride	Water	50 mL	300.0A [7] 325.2	250 mL plastic or glass. No preservative required, 28 days	9056A 9251	Method 9056: Cool to 4°C. Analyze ASAP after collection. Method 9251: 250ml plastic or glass, no preservative required, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Chlorine, Residual	Water	100 mL	330.5	250 mL glass or plastic. Cool to 4°C, analyze immediately	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Chromium (Cr^{+6})	Water	100 mL	3500 Cr-D	Method 218.4: 200 mL plastic or glass. Cool to 4°C, 24 hours. Method 3500 Cr-D: 200 mL quartz, TFE, or polypropylene HNO_3 to pH <2. Cool to 4°C. Analyze ASAP after collection	7196A	200 mL plastic or glass. Cool to 4°C, 24 hours
	Solid	20 g	---	N/A	7196A 3060A	250 mL plastic or glass, 30 days to digestion, 96 hours after digestion
	Waste	N/A	---	N/A	---	N/A
Conductivity	Water	100 mL	120.1 2510B	200 mL glass or plastic. Cool to 4°C, 28 days	9050A	200 mL glass or plastic. Cool to 4°C, 24 hours
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Cyanide (Amenable)	Water	250 mL	335.1 SM4500CN-G	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6]. Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours.	9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6]. Cool to 4°C, 14 days
	Solid	50g	---	N/A	9012A	Not Specified
	Waste	50g	---	N/A	9012A	Not Specified
Cyanide (Total)	Water	1L	335.2 335.3 335.4 (7) SM4500CN-E 335.2-CLP-M	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6]. Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours.	9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6]. Cool to 4°C, 14 days.
	Solid	50g	--	N/A	9012A	8 or 16 oz glass Teflon-lined lids, Cool to 4°C, 14 days
	Waste	50g	--	N/A	9012A	8 or 16 oz glass Teflon-lined lids, Cool to 4°C
Flashpoint (Ignitability)	Liquid	100 mL	---	N/A	1010 ASTM D93-9	No requirements, 250 mL amber glass. Cool to 4°C recommended
	Solid	100 g	--	N/A	---	N/A
	Waste	100 mL	--	N/A	---	N/A
Fluoride	Water	300 mL	300.0 [7] 340.2	500 mL plastic. No preservation required, 28 days.	9056A	Cool to 4°C. Analyze ASAP after collection.
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Hardness (Total)	Water	50 mL	130.2 2340B	250 mL glass or plastic, HNO_3 to pH < 2, 6 months	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Iron (Ferrous)	Water	100 mL	3500-Fe D	1 liter glass or polyethylene container, 6 months. **This test should be performed in the field.**	-	N/A
	Solid	N/A	-	N/A	-	N/A
	Waste	N/A	-	N/A	-	N/A
Nitrate	Water	50 mL	300.0A [7] SM4500NO_3-E	Method 300.0: 250 mL plastic or glass. Cool to 4°C, 48 hours.	9056A	Method 9056: Cool to 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	Not Specified
Nitrite	Water	50 mL	300.0A [7] 354.1	250 mL plastic or glass. Cool, 4°C, 48 hours	9056A	Cool, 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Nitrate-Nitrite	Water	50 mL	353.2	250 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Ortho-phosphate	Water	50 mL	300.0A [7] 365.1 SM4500P-E	100 mL plastic or glass. Filter on site. Cool to 4°C, 48 hours	9056A	Cool to 4°C. Analyze ASAP collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
pH	Water	50 mL	150.1 SM4500H-B	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.**	9040B	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.(8)**
	Solid	N/A	---	N/A	9045C	4 oz glass or plastic. Cool to 4°C. Analyze as soon as possible.[8]
	Waste	N/A	---	N/A	9045C	4 oz glass or plastic, Cool to 4°C. Analyze as soon as possible.[8]
Phenolics	Water	100 mL	420.1	500 mL glass, Cool to 4°C, H_2SO_4 to pH < 2, 28 days	9065	1 liter glass recommended, Cool to 4°C, H_2SO_4 to pH < 4, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	9065	Not Specified
Phosphate	Water	50 mL	---	N/A	9056A	Cool to 4°C, analyze ASAP collection
	Solid	N/A	---	N/A	9056A	N/A
	Waste	N/A	---	N/A	9056A	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Phosphorus (Total)	Water	100 mL	365.2 365.1 SM4500P-E	100 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Settleable Solids	Water	1000 mL	160.5	1000 mL plastic or glass. Cool to 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Specific Conductance	Water	50 mL	120.1 2510B	250 mL plastic or glass. Cool to 4°C, 24 hours	9050A	250 mL plastic or glass. Cool to 4°C, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Sulfate (SO_4)	Water	50 mL	300.0A [7] 375.4	100 mL plastic or glass. Cool to 4°C, 28 days	9056A 9038	Method 9056: Cool to 4°C. Analyze ASAP collection. Method 9038: 200 mL plastic or glass, Cool to 4°C, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	100 mL	---	N/A	9038	200 mL plastic or glass. Cool to 4°C, 28 days

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Sulfide	Water	250 mL	376.1	500 mL plastic or glass. Cool to 4°C, Add 2 mL zinc acetate plus NaOH to pH > 9, 7 days	9030A 9030B/ 9034	500 mL plastic, No headspace. Cool to 4°C. Add 4 drops of 2N zinc acetate per 100 mL of sample, adjust the pH to > 9 with 6 N NaOH solution, 7 days
	Solid	50 g	---	N/A	9030A 9030B/ 9034	Cool to 4°C. Fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free
	Waste	50 g	---	N/A	9030A 9030B/ 9034	Cool to 4°C. Fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free
Total Dissolved Solids (Filterable)	Water	100 mL	160.1 2540C	250 mL plastic or glass. Cool to 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Total Kjeldahl Nitrogen (TKN)	Water	100 mL	351.3	500 mL plastic or glass. Cool to 4°C, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Organic Carbon (TOC)	Water	100 mL	415.1 SM5310D	100 mL plastic or glass. Cool to 4°C, H_2SO_4 to pH < 2, 28 days	9060 Walkley-Black	100 mL glass or 40 mL VOA vials,Cool to 4°C, H_2SO_4 or HCl to pH < 2, 28 days
	Solid	N/A	---	N/A	9060 Walkley-Black	Not Specified
	Waste	N/A	---	N/A	9060 Walkley-Black	Not Specified
Total Organic Halides (TOX) (EOX)	Water	100 mL	450.1 (7)	500 mL amber glass, Teflon®-lined lid. Cool to 4°C, HNO_3 to pH <2, no headspace, 28 days	9020B 9023 (EOX)	500 mL amber glass, Teflon®-lined lid. Cool to 4°C, H_2SO_4 to pH < 2, no headspace, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Solids	Water	100 mL	160.3	250 mL plastic or glass. Cool to 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Suspended Solids (Nonfilterable)	Water	100 mL	160.2	250 mL plastic or glass. Cool, 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Turbidity	Water	50 mL	180.1	250 mL plastic or glass. Cool, 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Volatile Solids	Water	100 mL	160.4	250 mL plastic or glass. Cool, 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Metals (excludes Hg)	Water	100 mL	200 series	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 6 months	6010B 6020	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 6 months
	Solid	200 g	200 series	2, 8, or 16 oz glass or polyethylene container storage at 4 °C	6010B 6020	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
	Waste	200 g	200 series	N/A	6010B 6020	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
Mercury (CVAA) (CVAFS)	Water	100 mL	245.1 1631E	250 mL glass or polyethylene container, HNO_3 to pH $\leq$ 2, 28 days	7470A	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 28 days
	Solid	200 g	--	2, 8, or 16 oz glass or polyethylene container. Cool to 4°C, 28 days. Not applicable for Method 1631E.	7471A	8 or 16 oz glass or polyethylene container. Cool to 4°C, 28 days (CORP-MT-0007)
	Waste	200 g	--	N/A	7471A	8 or 16 oz glass or polyethylene container. Cool, 4°C, 28 days (CORP-MT-0007)

Footnotes

[1] Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.

[2] National Pollutant Discharge Elimination System - MCAWW, March 1983.

[3] Holding times are calculated from date of collection. Holding Times are determined based on date of collection to preparation/analysis.

[4] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA, (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

[5] Solid matrix type includes soil, sediment, sludge and other solid materials not classified as waste.

[6] Samples to be analyzed for cyanide should be field-tested for residual chlorine. If residual chlorine is detected, ascorbic acid should be added.

[7] Method not listed in 40 CFR Part 136.

[8] If not done in the field (ASAP) per the method and requested by client, analyze in lab within 48 hours.

[9] EPA issued memo recommending not to use reactive cyanide and sulfide methods.

Table 22-2. Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Aromatic Volatiles	Water	40 mL	602	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 7 days with pH > 2,14 days with pH ≤ 2	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 1:1 HCl to pH ≤2, 14 days with pH ≤ 2
	Solid [5]	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool, 4°C [12]
	Waste	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Max holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol). Cool to 4°C [12]

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Halogenated Volatiles by GC	Water	40 mL	--	N/A	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 1:1 HCl to pH ≤ 2, 14 days
	Solid [5]	5 g or 25 g	--		8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool to 4°C [12]
	Waste	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool, 4°C [12].

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Herbicides	Water	1L	615 [(10)]	1 liter amber glass with Teflon®-lined lid, Sodium thiosulfate or ascorbic acid if residual chlorine present. Cool to 4°C, Extraction, 7 days. Analysis, 40 days after extraction	8151A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool to 4°C. Extraction, 7 days. Analysis, 40 days of the start of extraction.
	Solid	50 g	--	N/A	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.
	Waste	50 g	--	N/A	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Pesticides/ PCBs	Water	1L	608	1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present and aldrin is being determined. Cool, 4°C. Extraction, 7 days. Analysis, 40 days after extraction.	8081A 8082	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL 10% sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days. Analysis, 40 days of the start of the extraction.
	Solid	50 g	---	N/A	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.
	Waste	50 g	---	N/A	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Petroleum Hydrocarbons /Oil and Grease	Water	1 L	1664A[(7)]	1 liter glass, Cool, 4°C HCl or H_2SO_4 to pH <2 28 days	9071B	1 liter glass, Cool, 0-4°C HCl or H_2SO_4 to pH <2 28 days
	Solid	30 g	1664A[(7)]	8 or 16 oz. Wide mouth glass jar, Cool, 4°C, 28 days	9071B	8 or 16 oz. wide mouth glass jar, Cool, 0-4°C, 28 days
	Waste	---	---	N/A	9071B	N/A
Purgeable Halocarbons by GC	Water	40 mL	601	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, 14 days.	8021B	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, 1:1 HCl to pH $\leq$ 2, 14 days.
	Solid	5 g or 25 g	---	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol). Cool, 4°C[12].

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Purgeable Halocarbons by GC (cont'd)	Waste	5 g or 25 g	---	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore™ sampler is 48 hours (before the sample is added to methanol). Cool, 4°C[12]
Semivolatiles	Water	1L	625	1 liter amber glass with Teflon®-lined lid. Cool, 4°C. Extraction, 7 days. Analysis, 40 days.	8270C	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days. Analysis, within 40 days of extraction.
	Solid	50 g	---	N/A	8270C	8 or 16 oz glass wide mouth with Teflon-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction.
	Waste	50 g	---	N/A	8270C	8 or 16 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction.

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Volatile Organics	Water	40 mL	624	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH ≤ 2[8].	8260B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2[9].
	Solid[5]	5 g or 25 g	--	N/A	8260B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Maximum holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol or sodium bisulfate). Cool to 4°C[(12)]
	Waste	5 g or 25 g	--	N/A	8260B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Maximum holding time for Encore™ sampler is 48 hrs (before sample is added to methanol or sodium bisulfate). Cool to 4°C[12]

Footnotes

[1] Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.

[2] National Pollutant Discharge Elimination System - 40 CFR Part 136, Appendix A.

[3] Holding times are calculated from the date of collection.

[4] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

[5] Solid matrix type includes soil, sediment, sludge or other solids not classified as waste.

[6] Only one determination method is listed when separate methods are required for preparation and analysis.

[7] Method 1664 was promulgated by the EPA with an effective date of June 14, 1999.

[8] For acrolein and acrylonitrile the pH should be adjusted to 4-5. This pH adjustment is not required if acrolein is not measured. Samples requiring analysis of acrolein that received no pH adjustment must be analyzed within three days of sampling.

[9] For acrolein and acrylonitrile the pH should be adjusted to 4-5.

[10] Method not listed in 40 CFR Part 136.

[11] Should only be used in the presence of residual chlorine.

[12] Depending on regulatory programs, EnCore™ samplers may be preserved for up to 14 days from sampling by freezing at -5 to-12°C until analysis. Alternatively the EnCore™ sample may be transferred to a 40-ml VOA vial and preserved by freezing at -5 to -12°C until analysis. Some regulatory agencies may require 4 or 8 oz glass with Teflon®-lined lid, Cool 4°C, 14 days. This technique is not recommended, but will be supported where required. (Preservation and holding times are subject to client specifications.)

Company Confidential & Proprietary

Table 22-3. Sample Containers, Preservatives, and Holding Times for TCLP[1] and SPLP[2]

Analytical Parameters	Matrix	Minimum Sample Size	TCLP Method 1311 and SPLP Method 1312 Requirements	
			From Field Collection to TCLP/SPLP Extraction	From TCLP/SPLP Extraction to Analysis
Mercury	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 28 days	Glass or polyethylene 28 days
Metals (except mercury)	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 180 days	Glass or polyethylene 180 days
Semivolatiles	Liquid Solid Waste	1L	1L glass, Cool 4°C, 14 days	1L glass Extraction of leachate within 7 days of TCLP extraction, Analyze extract within 40 days
Volatiles	Liquid Solid Waste	6 oz	4 oz glass, Cool 4°C, 14 days	40 mL glass, 14 days

Footnotes

[1] TCLP = Toxicity Characteristic Leaching Procedure
[2] SPLP = Synthetic Precipitation Leaching Procedure

SECTION 23

HANDLING OF SAMPLES
(NELAC 5.5.8)

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the Sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the Sample Control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The COC is stored with project information and the report.

23.1.2 Legal / Evidentiary Chain-of-Custody

The lab does not accept samples that require legal Chain-of-Custody.

23.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated Sample Receiving personnel, and a unique laboratory project identification number is assigned. Each sample container must be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking, and storage procedures are summarized in the following sections. SOP NC-SC-005, Sample Receiving and Sample Control, describes the laboratory's sample receipt procedure.

23.2.1 Laboratory Receipt

Samples must be received and logged in at TestAmerica by a designated sample custodian or other properly trained associate. Upon sample receipt, the sample custodian shall, as appropriate:

- Wear appropriate personal protective equipment. At a minimum, this consists of cut-resistant gloves, a lab coat, and safety glasses
- Examine the shipping containers to verify that the custody tape is intact
- Examine all sample containers for damage
- Open shipping containers in adequately ventilated areas to assure worker safety
- Determine if the temperature required by the requested testing program has been maintained during shipment. Document the shipping container temperature on the Cooler Receipt Form
- Compare samples received against those listed on the COC
- Verify that sample holding times have not been exceeded
- Examine all shipping records for accuracy and completeness
- Determine sample pH (if required for the scheduled analysis) (except VOA and TOX samples) and record on the Cooler Receipt Form (CRF)
- Sign and date the COC immediately (only after shipment is accepted) and attach the waybill
- Note any problems associated with the coolers and samples on the cooler receipt form and

notify the PM who in turn notifies the client

- Attach durable (water-resistant) laboratory sample container labels with unique laboratory identification number and test
- Place the samples in proper laboratory storage.

A Cooler Receipt Form (CRF) or an equivalent form/system is generated by sample control during the sample log-in process to document anomalies identified upon the receipt of samples in the laboratory. These anomalies are outside of laboratory control and do not require corrective actions to be taken within the laboratory. The affected client must be notified by the PM or designee of all CRFs generated for their samples. The PM is responsible for resolving with the client how to proceed with the samples and documenting the decision to proceed with the analysis of compromised samples. CRFs must be resolved prior to sample preparation and analysis. The completed CRF must be stored in the project file. An example CRF is shown in Figure 24-4. The report narrative must include an explanation of sample receiving related anomalies.

23.2.1.1 Unique Sample Identification

A unique lot number is generated for each project that is logged. The lot number is nine characters in length, and is based on the date of receipt. For example, Lot **A9C010121** is described as follows:

A - TestAmerica location where the samples were received (A = North Canton, C = Pittsburgh, etc.)

9 - Last digit of the year (i.e., 2009)

C - Month (i.e., A = January, B = February, C = March)

01 - These two numeric characters identify the day of the month--in this case, the first day of the month.

0121 - These four numeric characters are the sequential assignment of numbers specific to each lot received. Each day, the first lot logged receives the number "0101"; the second lot receives the number "0102", etc.

As each sample is logged, individual *sample numbers* are created in sequential order, noted as the three-digit suffix following the lot number. For example, four samples were received on May 10, 2008 for PCB analysis with the following client IDs: AB100, AB101, AB102, AB103.

Client ID - Lot No. - Sample No.	Analysis
AB100A8E100101-001	PCB
AB101A8E100101-002	PCB
AB102A8E100101-003	PCB
AB103A8E100101-004	PCB

If a client requests an MS/MSD for a sample(s), it is denoted in LIMS by an "S" for the MS and a "D" for the MSD.

Work Order Numbers: As each parameter is logged, a random unique 8-digit Work Order number is generated by LIMS. For example, Work Order **A5WE1-1-1C** is described as follows:

A5WE1 - The first five characters of the Work Order number identifies each unique sample.

1 - The "modifier" indicates the run number. This number will always be a "1" at log-in. However, additional modifier numbers may be generated by the analytical lab groups when necessary (example: for a re-extract).

1C - The "suffix" identifies a specific test for each sample, but is unique to the test being performed on each sample.

Each sample container representing the same sample (as indicated by the client ID) will have the same 5-digit Work Order number, along with a 3-digit suffix that corresponds to the bottle count (example: -001, -002, -003, etc.). For example, if five containers are submitted for the same sample and the LIMS-generated Work Order number is CREE4, LIMS will generate five labels--CREE4-001, CREE4-002, CREE4-003, CREE4-004, and CREE4-005. These numbers are used to track sample containers through the analytical process.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy outlined in SOP NC-SC-005, Sample Receiving and Sample Control, that clearly outlines the circumstances under which samples must be accepted or rejected. These include:

- A COC filled out completely
- Samples must be properly labeled
- Proper sample containers with adequate volume for the analysis and necessary QC
- Samples must be preserved according to the requirements of the requested analytical method
- Sample holding times must be adhered to
- All samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time
- The Project Manager must be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

Once sample acceptance is verified, the samples are logged into LIMS according to SOP NC-SC-005.

23.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators suitable for the sample matrix. Metals samples may be unrefrigerated. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards, or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every week.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

All samples per SOP are treated as hazardous. If any extra or known hazards are present in the sample, the sample is flagged and precautions / instructions are put in the comments. Hazardous samples are segregated out, and go into the waste stream profile for the nature of the hazard. All soils--foreign and domestic--go to a USDA approved incinerator.

23.6 SAMPLE SHIPPING

In the event the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The Chain-of-Custody form is signed by the Sample Control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper Chain-of-Custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent; however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client

requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist--the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP NC-SC-005, Sample Receiving and Sample Control). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work. Waste disposal complies with all federal and state laws and regulations.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), and names of individuals who conducted the arrangements and physically completed the task. Sample labels are destroyed through the disposal method, e.g., samples are incinerated. A Waste Manifest is completed.

Figure 23-1. Example: Chain of Custody (COC)

TestAmerica North Canton
4101 Shuffle Drive N.W.
North Canton, OH 44720
phone 330-497-9396 fax 330-497-0772

Chain of Custody Record

Client Contact	**Project Manager:**	**Site Contact:**	**Date:**
Your Company Name here	**Tel/Fax:**	**Lab Contact:**	**Carrier:**
Address	**Analysis Turnaround Time**		
City/State/Zip	Calendar (C) or Work Days (W)		
(xxx) xxx-xxxx Phone	TAT if different from Below _____		
(xxx) xxx-xxxx FAX	☐ 2 weeks		
Project Name:	☐ 1 week		
Site:	☐ 2 days		
P O #	☐ 1 day		

Sample Identification	**Sample Date**	**Sample Time**	**Sample Type**	**Matrix**	**# of Cont.**	**Filtered Sample**

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification

☐ *Non-Hazard* ☐ *Flammable* ☐ *Skin Irritant* ☐ *Poison B* ☐ *Unknown*

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)

☐ *Return To Client* ☐ *Disposal By Lab* ☐ *Archive For _____ Months*

Special Instructions/QC Requirements & Comments:

Relinquished by:	Company:	Date/Time:	Received by:	Company:
Relinquished by:	Company:	Date/Time:	Received by:	Company:
Relinquished by:	Company:	Date/Time:	Received by:	Company:

Figure 23-2.

Example: Custody Seal

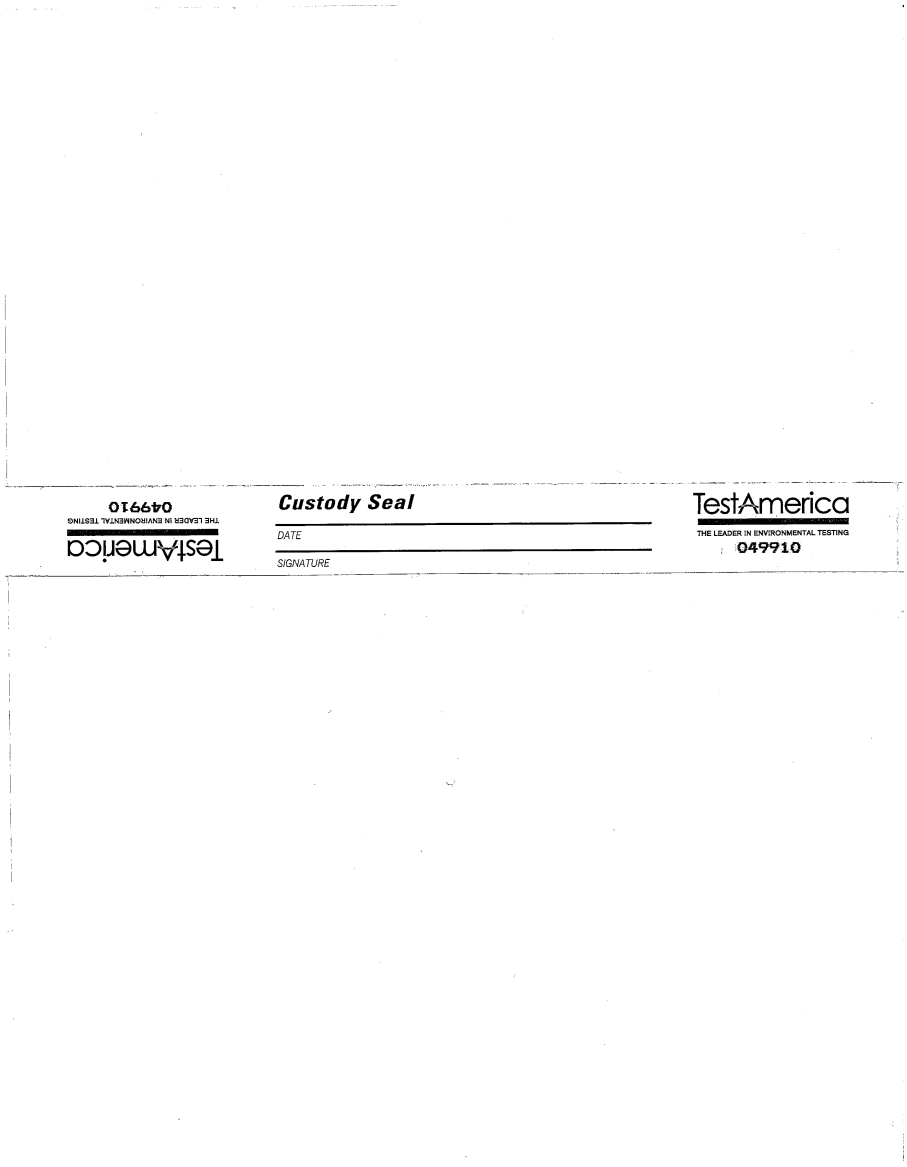


Figure 23-3. Example: Internal Chain of Custody (COC)

TestAmerica Laboratories, Inc.
Sample Control Record

Client:

Lot Number:

Case Number/SDG:

Storage Location:

Laboratory Sample ID	Transferred By	Date	Entered	Removed	Reason	Date Returned

Figure 23-4. Example: Cooler Receipt Form (Page 1)

TestAmerica Cooler Receipt Form/Narrative **Lot Number:**_____
North Canton Facility

Client _____ Project _____ By _____

Cooler Received on _____ Opened on _____ (Signature)
FedEx ☐ UPS ☐ DHL ☐ FAS ☐ Stetson ☐ Client Drop Off ☐ TestAmerica Courier ☐ Other _____

TestAmerica Cooler # _____ Multiple Coolers ☐ Foam Box ☐ Client Cooler ☐ Other_____

1. Were custody seals on the outside of the cooler(s)? Yes ☐ No ☐ Intact? Yes ☐ No ☐ NA ☐
   If YES, Quantity_____ Quantity Unsalvageable _____
   Were custody seals on the outside of cooler(s) signed and dated? Yes ☐ No ☐ NA ☐
   Were custody seals on the bottle(s)? Yes ☐ No ☐
   If YES, are there any exceptions? _____
2. Shipper's packing slip attached to the cooler(s)? Yes ☐ No ☐
3. Did custody papers accompany the sample(s)? Yes ☐ No ☐ Relinquished by client? Yes ☐ No ☐
4. Were the custody papers in the appropriate place? Yes ☐ No ☐
5. Packing material used: Bubble Wrap ☐ Foam ☐ None ☐ Other _____
6. Cooler temperature upon receipt _____ °C See back of form for multiple coolers/temps ☐

METHOD: IR ☐ Other ☐ _____
COOLANT: Wet Ice ☐ Blue Ice ☐ Dry Ice ☐ Water ☐ None ☐

7. Did all bottles arrive in good condition (Unbroken)? Yes ☐ No ☐
8. Could all bottle labels be reconciled with the COC? Yes ☐ No ☐
9. Were sample(s) at the correct pH upon receipt? Yes ☐ No ☐ NA ☐
10. Were correct bottle(s) used for the test(s) indicated? Yes ☐ No ☐
11. Were air bubbles >6 mm in any VOA vials? Yes ☐ No ☐ NA ☐
12. Sufficient quantity received to perform indicated analyses? Yes ☐ No ☐
13. Was a Trip Blank present in the cooler(s)? Yes ☐ No ☐ Were VOAs on the COC? Yes ☐ No ☐

Contacted PM _____ Date _____ by _____ via Verbal ☐ Voice Mail ☐ Other ☐
Concerning_____

14. CHAIN OF CUSTODY

The following discrepancies occurred:
_____
_____
_____
_____
_____
_____

15. SAMPLE CONDITION

Sample(s)_____ were received after the recommended holding time had expired.
Sample(s)_____ were received in a broken container.
Sample(s)_____were received with bubble >6 mm in diameter. (Notify PM).

16. SAMPLE PRESERVATION

Sample(s) _____ were further preserved in Sample Receiving to meet recommended pH level(s). *Nitric Acid Lot* # 100108-HNO_3 - *Sulfuric Acid Lot* # 100108-H_2SO_4; *Sodium Hydroxide Lot* # 073007-*NaOH; Hydrochloric Acid Lot* # 092006-*HCl; Sodium Hydroxide and Zinc Acetate Lot* # 050205-$(CH_3COO)_2ZN$/*NaOH.* What time was preservative added to sample(s)? _____

Client ID	pH	Date	Initials

Example: Cooler Receipt Form (Page 2)

TestAmerica Cooler Receipt Form/Narrative
North Canton Facility

Client ID	pH	Date	Initials

Cooler	Temp °C	Method	Coolant

Discrepancies Cont'd:

SECTION 24

ASSURING THE QUALITY OF TEST RESULTS
(NELAC 5.5.9)

24.1 OVERVIEW

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g., Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 NEGATIVE CONTROLS

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blanks (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable or injected at specifed frequencies throughout an analytical sequence. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve. These blanks may be termed Initial Calibration Blanks (ICB) or Continuing Calibration Blanks (CCB),

Table 24-1. Example – Negative Controls

Control Type	Details
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blanks [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 POSITIVE CONTROLS

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon:

1) Method Performance [Laboratory Control Sample (LCS) or Blank Spike (BS)], which entails both the preparation and measurement steps

2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed.

Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch.

Note that frequency of control samples vary with specific regulatory, methodology, and project-specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

24.4.1.1 The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

24.4.1.2 The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

24.4.1.3 Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g., solid matrix LCS for metals, TDS, etc.).

24.4.1.4 The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally one for each batch of sample--not to exceed 20 environmental samples.

24.4.1.5 If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable, e.g., no spike of pH. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

24.4.1.5.1 For methods that have 1-10 target analytes, spike all components.

24.4.1.5.2 For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

24.4.1.5.3 For methods with more than 20 target analytes, spike at least 16 components.

24.4.1.5.4 Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

24.4.1.5.5 Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project-specific basis.

24.5 SAMPLE MATRIX CONTROLS

Table 24-2 Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	To assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially, a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.
[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

24.6.1 As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project-specific control

limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes, and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Note: For Ohio VAP the laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit.

24.6.2 Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

24.6.3 Laboratory-generated Percent Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

24.6.3.1 Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV), (unless the analytical method specifies a tighter limit).

24.6.3.2 In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evlauted against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) an be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

24.6.3.3 The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5%, and the analyte must be detectable and identifiable.

24.6.3.4 The maximum acceptable recovery limit will be 200%.

24.6.3.5 The maximum acceptable RPD limit will be 30% for organic methods and 20% for inorganic methods. The minimum RPD limit is 10%.

24.6.3.6 If either the high or low end of the control limit changes by $\leq$ 10% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.4 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical

control limits. Refer to NC-QA-018, Statistical Evaluation of Data and Development of Control Charts, for details.

24.6.4.1 One example: The QA Department generates a Quality Control Limit Summary that contains tables that summarize the precision and accuracy acceptability limits for analyses. Unless otherwise noted, limits within these tables are laboratory generated. Once reviewed and approved, the limits are entered into LIMS and are effective immediately. The Quality Assurance department maintains an archive of all limits used within the laboratory.

24.6.5 An LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be re-analyzed if possible. If re-analysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal Corrective Action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without re-analysis if:

24.6.5.1 The analyte results are below the reporting limit and the LCS is above the upper control limit.

24.6.5.2 If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Note: For Ohio VAP the laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as ‘not VAP certified’ on the affidavit.

24.6.5.3 Or, for NELAC and Department Of Defense (DOD) work, there are an allowable number of Marginal Exceedances (ME):

- <11 analytes 0 marginal exceedances are allowed.
- 11 – 30 Analytes 1 marginal exceedance is allowed
- 31-50 Analytes 2 marginal exceedances are allowed
- 51-70 Analytes 3 marginal exceedances are allowed
- 71-90 Analytes 4 marginal exceedances are allowed
- > 90 Analytes 5 marginal exceedances are allowed

24.6.5.3.1 Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).

Note: Use of Marginal Exceedances is not permitted for Ohio VAP.

24.6.5.3.2 Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

24.6.5.3.3 Though marginal excedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.6 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and re-analyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.7 If a surrogate standard falls outside the acceptance limits, and if there is not obvious chromatographic matrix interference, re-analyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the re-analysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client).

Note: A more detailed discussion of acceptance criteria and corrective action can be found in the laboratory's method SOPs and in Section 12.

24.7 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

24.7.1 The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21), and use of PT samples (see Section 15).

24.7.2 A discussion regarding MDLs, Limit of Detection (LOD), and Limit of Quantitation (LOQ) can be found in Section 19.

24.7.3 Use of formulae to reduce data is discussed in the method SOPs and in Section 20.

24.7.4 Selection of appropriate reagents and standards is included in Sections 9 and 21.

24.7.5 A discussion on selectivity of the test is included in Section 5.

24.7.6 Constant and consistent test conditions are discussed in Section 18.

24.7.7 The laboratory sample acceptance policy is included in Section 23.

SECTION 25

REPORTING RESULTS
(NELAC 5.5.10)

25.1 OVERVIEW

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is a conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory must work with the client during project setup to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 19.

25.2 TEST REPORTS

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed, reviewed, and signed by the appropriate Project Manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title with a "Sample Result" header.

25.2.2 Each report cover page printed, which includes the laboratory name, address, and telephone number.

25.2.3 A unique identification of the report (e.g., Work Order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented at the bottom of each page. The report is sequentially paginated. The final page of the report is labeled as "End of Report".

25.2.4 A copy of the Chain-of-Custody (COC).

- Any COCs involved with subcontracting are included.

- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g., Sampling information).

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis.

25.2.9 Date reported or date of revision, if applicable

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc)

25.2.11 Reporting limit

25.2.12 Method detection limits (if requested)

25.2.13 Definition of data qualifiers and reporting acronyms, e.g., ND

25.2.14 Sample results

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (refer to Section 25.2.4 – Item 3, regarding additional addenda).

25.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.18 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

25.2.19 When NELAC accreditation is required, the lab must certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

25.2.20 The laboratory includes a cover page.

25.2.21 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.22 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.23 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.24 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report, e.g., partial report, or how your lab identifies it. A complete report must be sent once all of the work has been completed.

25.2.25 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy CA-L-P-002 for details on internally applying electronic signatures of approval.

25.2.26 Reports for Ohio VAP work require a VAP affidavit be completed and included with the report.

Note: For additional information on Ohio VAP affidavits refer to OAC Rule 3745-300-04 and OAC Rule 3745-300-13(N), effective March 1, 2009.

25.3 REPORTING LEVEL OR REPORT TYPE

The laboratory offers two levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Standard report – all features in Section 25.2
- Expanded deliverable – standard report presented on CLP-like forms and relevant calibration information. All supporting raw data is supplied.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Procedures used to ensure client confidentiality are outlined in Section 25.7.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica services. TestAmerica North Canton offers a variety of EDD formats including (but not limited to) ADR, EQuIS, GISKey, Region 5, NJHAZsite, and a wide variety of client specific multi-file, Excel and flat file formats.

EDD specifications are submitted to the IT Department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs must be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

25.4.1 Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

25.4.2 Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

25.4.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

25.4.4 Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response must be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP CA-L-S-002, Subcontracting.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of the TestAmerica network are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 CLIENT CONFIDENTIALITY

In situations involving the transmission of environmental test results by telephone, facsimile, or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the client or any other person designated by the client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

"Confidentiality Notice: The information contained in this message is intended only for the use of the addressee, and may be confidential and/or privileged. If the reader of this message is not the intended recipient, or the employee or agent responsible to deliver it to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify the sender immediately."

25.7 FORMAT OF REPORTS

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 AMENDMENTS TO TEST REPORTS

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

When the report is re-issued, a notation of "report reissue" is placed on the cover/signature page of the report or at the top of the narrative page with a brief explanation of reason for the reissue and a reference back to the lst final report generated.

25.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

25.9.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

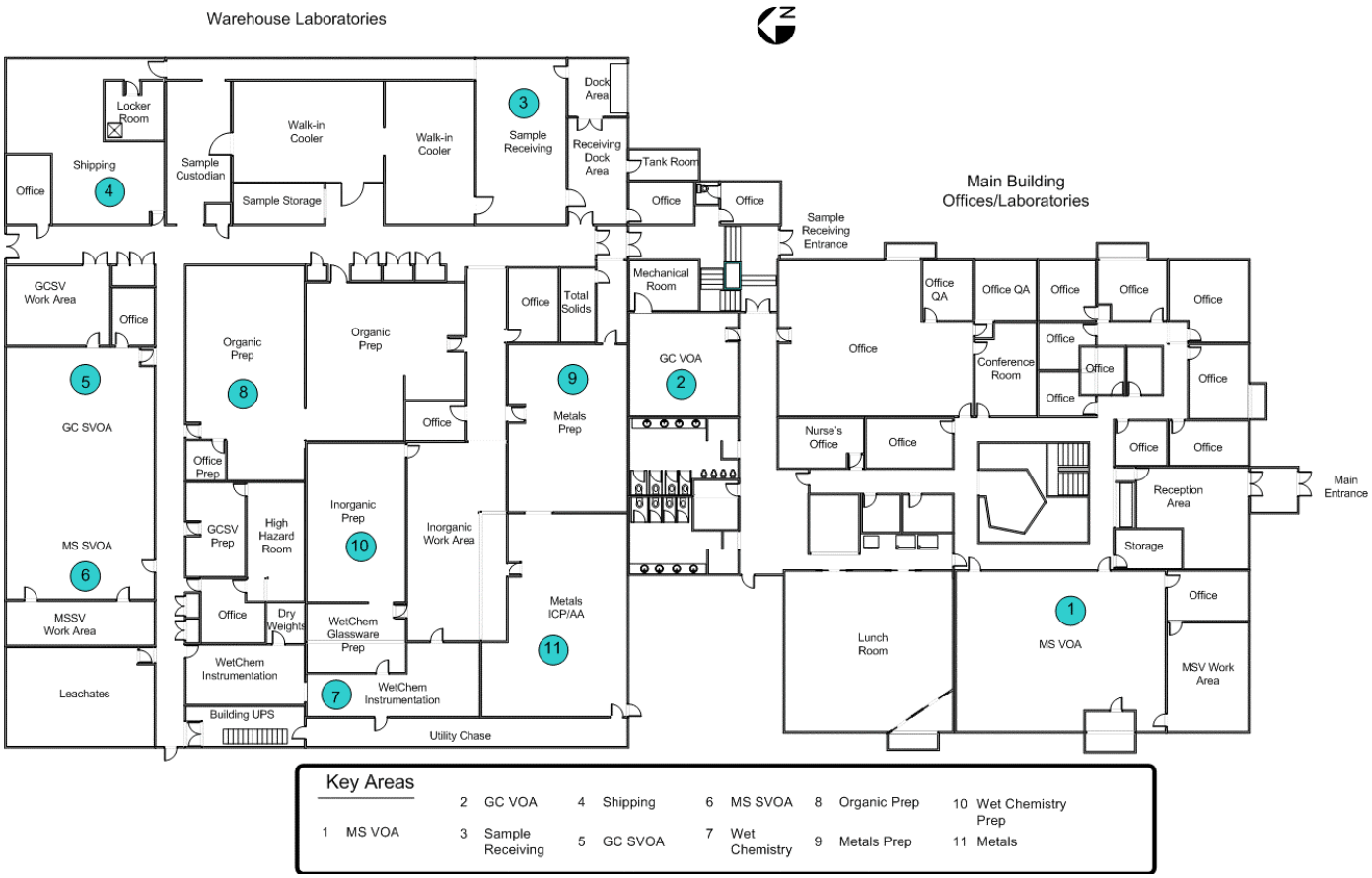
Appendix 1.

Laboratory Floor Plan

TestAmerica – North Canton

4101 Shuffel Dr NW
North Canton, OH 44720

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING



Appendix 2. Laboratory Method Listing

Wet Chemistry Methods [1]

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Acidity	Water		305. [2] SM 2310 B	--	--
Alkalinity, Bicarbonate, Carbonate	Water		305. [2] SM 2320 B		
	Solid		EPA 310.1[2] (M)	--	--
Arsenic (ASV) Anodic Stripping Voltammetry	Water		--	EPA 7063	--
Ash Content	Solid		--	--	ASTM D29-74
Biochemical Oxygen Demand, Carbonaceous	Water		EPA 405.1 SM 5210 B	--	--
Bromide	Water		EPA 300.0A	EPA 9056A	--
	Waste		EPA 300.0A	EPA 9056A	--
	Solid		EPA 300.0A (M)	EPA 9056A	--
Cation-Exchange Capacity	Solid		--	EPA 9081	--
Chemical Oxygen Demand	Water		EPA 410.4 SM 5220D	--	--
	Waste		EPA 410.4	--	--
Chloride	Water		EPA 300.0A EPA 325.2[2]	EPA 9056A EPA 9251	EPA 325.2[2]
	Waste		EPA 300.0A	EPA 9056A	--
	Solid		EPA 300.0A (M)	EPA 9056A EPA 9251(M)	--
Chromium, Hexavalent	Water		EPA 3500-Cr-D	EPA 7196A	--
	Waste		EPA 3500-Cr-D	EPA 7196A	--
	Solid		--	EPA 3060A EPA 7196A	--

[1] Any matrix not listed is not applicable for the associated method
[2] Removed from 40CFR

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Specific Conductance	Water	EPA 120.1 SM 2510B	EPA 9050A	--
	Waste	EPA 120.1	EPA 9050A	--
	Solid	--	EPA 9050A	--
Chlorine, Residual	Water	EPA 330.5[2] SM 3500 CL-G	--	--
Cyanide (Amenable)	Water	EPA 335.1[2] SM 4500 CN-G	EPA 9012A	--
	Solid	--	EPA 9012A	--
Cyanide (Total)	Water	SM 4500-CN E EPA 335.4 335.2-CLP-M	EPA 9012A	---
	Waste	--	EPA 9012A	--
	Solid	--	EPA 9012A	--
Cyanide (Weak and Dissociable) (Free)	Water	SM 4500-CN I	--	--
Dissolved Oxygen	Water	360.1[2] SM 4500 O-G	--	--
Flash Point	Waste	--	EPA 1010	ASTM D93-9
	Solid	--	EPA 1010	ASTM D93-9
Fluoride	Water	EPA 300.0A EPA 340.2[2]	EPA 9056A	SM 4500 F-C, ISE
	Waste	EPA 340.2 (M)[2] EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 340.2 (M)[2] EPA 300.0A (M)	EPA 9056A	--
Iron, Ferrous & Ferric	Water	SM 3500 FE D	--	--
Hardness	Water	EPA 130.2[2]	--	SM 2340B
Moisture	Solid	---	EPA 160.3 (M) ASTM D2216-90	---
Nitrogen, Ammonia	Water	EPA 350.1	--	EPA 350.2[2]
	Waste	EPA 350.1	--	EPA 350.2[2]
	Solid	EPA 350.1	--	EPA 350.2[2]
	Water	SM 4500 NH_3-B (Distillation)	--	--
	Water	SM 4500 NH_3-E (Titration)	--	--
	Water	SM 4500 NH_3-F (ISE)	--	--

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Nitrite (NO_2)	Water		EPA 300.0A	EPA 9056A	--
	Waste		EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 300.0A (M)	EPA 9056A	--
Nitrate (NO_3)	Water		EPA 300.0A	EPA 9056A	SM 4500 NO_3-E
	Waste		EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 300.0A (M)	--	--
Nitrate plus Nitrite NO_2/NO_3	Water		EPA 353.2	--	--
	Waste		EPA 353.2	--	--
Total Kjeldahl Nitrogen (TKN)	Water		EPA 351.3	--	SM 4500 NO_3
	Waste		EPA 351.3	--	--
	Solid		EPA 351.3	--	--
Oil and Grease (Hexane Extractable Material)	Water		EPA 1664A	EPA 9071B	--
	Waste		EPA 1664A	EPA 9071B	--
	Solid		--	EPA 9071B	--
Ortho-phosphate o-PO_4	Water		EPA 300.0A EPA 365.1	EPA 9056A	SM 4500 P-E
	Waste		EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 300.0A (M) EPA 365.1	EPA 9056A	--
pH	Water		EPA 150.1[2]	EPA 9040B	EPA 9041
	Waste		SM 4500 H-B	EPA 9045C	--
	Solid		---	EPA 9045C	--
Paint Filter	Water		--	EPA 9095A	--
Phenolics	Water		EPA 420.1	--	--
	Waste		--	EPA 9065	--
	Solid		--	EPA 9065	--
Phosphorus (Total)	Water		EPA 365.1	--	SM 4500 P-E
	Waste		EPA 365.1	--	--
	Solid		EPA 365.1	--	--
Sulfate (SO_4)	Water		EPA 300.0A EPA 375.4[2]	EPA 9056A EPA 9038	--
	Waste		EPA 300.0A (M) EPA 375.4[2]	EPA 9056A EPA 9038	--
	Solid		EPA 300.0A (M)	EPA 9056A EPA 9038 (M)	--

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA	Other
Sulfide	Water		EPA 376.1[2]	EPA 9030A SM 4500	9030B/9034
Total Organic Carbon (TOC)	Water		EPA 415.1[2]	EPA 9060	SM 5310 D
	Waste		--	EPA 9060	--
	Solid		EPA 415.1 (M)	EPA 9060 (M)	Walkley-Black
Total Organic Halides (TOX)	Water		--	EPA 9020B EPA 9023(EOX)	EPA 450.1
	Waste		--	--	--
	Solid		--	EPA 9020B	--
Total Petroleum Hydrocarbons	Water		EPA 1664A (SGT-HEM)	EPA 9071B	--
	Waste		EPA 1664A (SGT-HEM)	EPA 9071B	--
	Solid		--	EPA 9071B	--
Total Solids	Water		EPA 160.3	--	--
	Waste		EPA 160.3	--	--
	Solid		EPA 160.3 (M)	--	--
Total Dissolved Solids	Water		EPA 160.1	--	2540E
Total Suspended Solids	Water		EPA 160.2	---	2540E
Volatile and Volatile Suspended Solids	Water		EPA 160.4	--	--
Settleable Solids	Water		EPA 160.5	--	--
Turbidity	Water		EPA 180.1	--	--

Methods for Mercury by Cold Vapor Atomic Absorption

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Mercury (CVAA)	Water		EPA 245.1	EPA 7470A	--
	TCLP Leachate		--	EPA 7470A	--
	Waste		--	EPA 7471A	--
	Solid		EPA 254.5	EPA 7471A	--

Methods for Mercury by Cold Vapor Atomic Fluororescence

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Mercury, Low Level (CVAFS)	Water		--	--	EPA 1631E

Methods for Metals by ICP and ICPMS

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Metals by ICP analysis	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		EPA 200.7	EPA 6010B	---
Metals by ICPMS analysis	Water		EPA 200.8	EPA 6020	---
	Waste		---	EPA 6020	---
	Solid		EPA 200.8	EPA 6020	---

Metals Sample Preparation Methods

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Toxicity Characteristic Leaching Procedure (TCLP)	Water		---	EPA 1311	---
	Waste		---	EPA 1311	---
	Solid		---	EPA 1311	---
ICP Metals	Water		EPA 200.7	EPA 3005A EPA 3010A	---
	TCLP Leachate		---	EPA 3010A	---
	Waste		---	EPA 3050B	---
	Solid		---	EPA 3050B	---
ICPMS Metals	Water		EPA 200.8	EPA 3010A	---
	TCLP		---	EPA 3010A	---
	Waste		---	EPA 3050B	---
	Solid		---	EPA 3050B	---
CVAA Mercury	Water		EPA 245.1	EPA 7470A	---
	TCLP Leachate		---	EPA 7470A	---
	Waste		---	EPA 7471A	---
	Solid		---	EPA 7471A	
CVAFS Mercury Low Level	Water		---	---	EPA 1631E

Organic Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	EPA 624	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Halogenated Volatiles by GC	Water	EPA 601	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Aromatic Volatiles by GC	Water	EPA 602	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Semivolatiles by GC/MS	Water	EPA 625	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---
Pesticides/PCBs by GC	Water	EPA 608	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Herbicides by GC	Water	EPA 615	EPA 8151A	---
	Waste	---	EPA 8151A	---
	Solid	---	EPA 8151A	---
Total Petroleum Hydrocarbons (Gasoline Range) by GC	Water	---	EPA 5030B	WI GRO
	Waste	---	EPA 5030B EPA 5035	WI GRO
	Solid	---	EPA 5035 EPA 5035	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC	Water	---	EPA 3510C EPA 3520C	WI DRO
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3580A	WI DRO
	Solid	---	EPA 3550B	WI DRO

Organic Methods of Analysis

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water		EPA 624	EPA 8260B	---
	Waste		---	EPA 8260B	---
	Solid		---	EPA 8260B	---
Halogenated Volatiles by GC	Water		EPA 601	EPA 8021B	---
	Waste		---	EPA 8021B	---
	Solid		---	EPA 8021B	---
Aromatic Volatiles by GC	Water		EPA 602	EPA 8021B	---
	Waste		---	EPA 8021B	---
	Solid		---	EPA 8021B	---
Semivolatiles by GC/MS	Water		EPA 625	EPA 8270C	
	Waste		---	EPA 8270C	---
	Solid		---	EPA 8270C	---
Pesticides/PCBs by GC	Water		EPA 608	Pesticides 8081A PCBs 8082	---
	TCLP Leachate		---	Pesticides 8081A PCBs 8082	---
	Waste		---	Pesticides 8081A PCBs 8082	---
	Solid		---	Pesticides 8081A PCBs 8082	---

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Phenoxyacid Herbicides by GC	Water		---	EPA 8151A	---
	TCLP Leachate		---	EPA 8151A	---
	Waste		---	EPA 8151A	---
	Solid		---	EPA 8151A	---
Gasoline Range Organics by GC	Water		---	EPA 8015B (M)	WI GRO
	Waste		---	EPA 8015B (M)	---
	Solid		---	EPA 8015B (M)	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC/FID	Water		---	EPA 8015B (M)	WI DRO
	Waste		---	EPA 8015B (M)	---
Dissolved Gases RSK-175	Water		---	---	SOP
Formaldehyde Carbonyl Compounds	Water		---	EPA 8315	---

Appendix 3. Glossary/Acronyms

Glossary

Acceptance Criteria:
Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation:
The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority:
The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

Accuracy:
The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst:
The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Assessment:
The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of NELAC). (NELAC)

Assessment Criteria:
The measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)

Assessment Team:
The group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (NELAC)

Assessor:
One who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (NELAC)

Audit:
A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.

Batch:
Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Blank:
A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample:
A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration:
To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve:
The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method:
A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard:
A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM):
A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

Chain-of-Custody:
An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

Clean Air Act:
The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):
The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

Compromised Samples:
Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

Confidential Business Information (CBI):
Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation:
Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional Cleanup procedures
(NELAC)

Conformance:
An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction*:*
Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action:
The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit:
A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction:
The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency:
An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Detection Limit:
The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

Document Control:
The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses:
The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank:
Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

External Standard Calibration:
Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA):
The enabling legislation under 7 U.S.C. 135 et seq., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (NELAC)

Federal Water Pollution Control Act (Clean Water Act, CWA):
The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

Field Blank:
Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Testing:
NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

Finding:
An assessment conclusion that identifies a condition having a significant effect on an item or activity. As assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (NELAC)

Holding Times (Maximum Allowable Holding Times):
The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Inspection:
An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Internal Standard:
A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

Internal Standard Calibration:
Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank:
A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Response:
Instrument response is normally expressed as either peak area or peak height however it may also reflect a numerical representation of some type of count on a detector (e.g. Photomultiplier tube, or Diode array detector) and is used in this document to represent all types.

Laboratory:
A defined facility performing environmental analyses in a controlled and scientific manner. (NELAC)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):
A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS must be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples must be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

Laboratory Duplicate:
Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Least Squares Regression (1st Order Curve):
The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit of Detection (LOD):
An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Matrix:
The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions must be used:

> Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.
>
> Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.
>
> Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with ,15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples must be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with .15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample):
Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. Matrix spikes must be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) must be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and must be reported to the client whose sample was used for the spike. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):
A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. Matrix spike duplicates or laboratory duplicates must be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory must document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) must be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and must be reported to the client whose sample was used for the duplicate. (QAMS)

Method Blank:
A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

Method Detection Limit:
The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

National Environmental Laboratory Accreditation Conference (NELAC):
A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

National Environmental Laboratory Accreditation Program (NELAP):
The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Negative Control:
Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

NELAC Standards:
The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (NELAC)

Performance Audit:
The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS):
A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control:
Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision:
The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation:
Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Testing:
A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

Proficiency Testing Program:
The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Test Sample (PT):
A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Quality Assurance:
An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance [Project] Plan (QAPP):
A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control:
The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample:
An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

Quality Manual:
A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System:
A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

Quantitation Limits:
The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range:
The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Reagent Blank (method reagent blank):
A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Reference Material:
A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method:
A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard:
A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

Replicate Analyses:
The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Report Limit (RL):
The laboratory nominal Quantitation Limit (QL) or the level of sensitivity required by the client but not lower than the LOD. Reporting limits are easily accessible via LIMS (QC Browser program).

Requirement:
Denotes a mandatory specification; often designated by the term “shall”. (NELAC)

Resource Conservation and Recovery Act (RCRA):
The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the “cradle-to-grave”, including its generation, transportation, treatment, storage, and disposal. (NELAC)

Safe Drinking Water Act (SDWA):
The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

Sample Duplicate:
Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Second Order Polynomial Curve (Quadratic): The 2^{nd} order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2^{nd} order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00

indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity:
(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity:
The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Spike:
A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period. (NELAC)

Standard:
The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs):
A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM):
A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor (however named):
The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

Surrogate:
A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and must be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit):
A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Director:
Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Test:
A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method:
An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Toxic Substances Control Act (TSCA):
The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability:
The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

Uncertainty:
A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

United States Environmental Protection Agency (EPA):
The Federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation:
The process of substantiating specified performance criteria. (EPA-QAD)

Verification:
Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

Note: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed must be kept on the measuring instrument's individual record.

Acronyms

BS	Blank Spike
BSD	Blank Spike Duplicate
CAR	Corrective Action Report
CCV	Continuing Calibration Verification
CF	Calibration Factor
CFR	Code of Federal Regulations
COC	Chain of Custody
CRS	Change Request Form
DOC	Demonstration of Capability
DQO	Data Quality Objectives
DU	Duplicate
DUP	Duplicate
EHS	Environment, Health and Safety
EPA	Environmental Protection Agency
GC	Gas Chromatography
GC/MS	GasChromatography/Mass Spectrometry
HPLC	High Performance Liquid Chromatography
ICP	Inductively Coupled Plasma Atomic Emission Spectroscopy
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IH	Industrial Hygiene
IS	Internal Standard
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
PT	Performance Testing
QAM	Quality Assurance Manual
QA/QC	Quality Assurance / Quality Control
QAPP	Quality Assurance Project Plan
RF	Response Factor
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SD	Standard Deviation
SOP	Standard Operating Procedure
TAT	Turn-Around Time
VOA	Volatiles
VOC	Volatile organic Compound

Appendix 4. Laboratory Certifications, Accreditations, Validations

TestAmerica North Canton maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Organization	Certificate Number	Organization	Certificate Number
California	01144CA	Nevada	OH-00048208A
Connecticut	PH-0590	New Jersey	OH001
Florida	E87225	New York	10975
Georgia	---	OVAP	CL0024
Illinois	001298	Pennsylvania	68-00340
Kansas	E-10336	US ACE (Army)	---
Kentucky Underground Storage Tank Program	0058	USDA (Dept.of Agriculture)	P330-08-00123
Minnesota	039-999-348	West Virginia	210
DoD - ACLASS	ADE-1437---	Wisconsin	999518190

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, Marketing, and Project Management.

ATTACHMENT B

CRS TCL 4.2 VOCs
Matrix: Soil

Method: SW846 8260B

#	Compound	RL	Units	MDL	Units	T	A	Check LCL	Check UCL	Check RPD	T	A	Spike LCL	Spike UCL	Spike RPD
11	Acetone	20	ug/kg	6.3	ug/kg		Y	58	130	30		Y	10	200	66
196	Benzene	5	ug/kg	0.23	ug/kg	C	Y	75	129	20	C	Y	55	138	20
323	Bromodichloromethane	5	ug/kg	0.28	ug/kg		Y	72	125	30		Y	47	131	51
340	Bromoform	5	ug/kg	0.33	ug/kg		Y	43	149	30		Y	26	141	64
343	Bromomethane	5	ug/kg	0.54	ug/kg		Y	24	152	30		Y	15	152	72
372	2-Butanone	20	ug/kg	1.4	ug/kg		Y	27	200	46		Y	21	195	60
459	Carbon disulfide	5	ug/kg	0.44	ug/kg		Y	50	137	30		Y	27	149	73
463	Carbon tetrachloride	5	ug/kg	0.37	ug/kg		Y	57	137	30		Y	32	143	68
521	Chlorobenzene	5	ug/kg	0.33	ug/kg	C	Y	75	127	22	C	Y	49	139	22
535	Dibromochloromethane	5	ug/kg	0.55	ug/kg		Y	49	135	30		Y	44	135	61
550	Chloroethane	5	ug/kg	0.86	ug/kg		Y	31	144	30		Y	32	140	66
569	Chloroform	5	ug/kg	0.29	ug/kg		Y	73	115	30		Y	59	128	46
574	Chloromethane	5	ug/kg	0.41	ug/kg		Y	15	136	30		Y	28	130	81
669	Cyclohexane	10	ug/kg	0.33	ug/kg		Y	50	150	20		Y	50	150	20
539	1,2-Dibromo-3-chloropropane	10	ug/kg	1.3	ug/kg		Y	50	150	30		Y	50	150	20
870	1,2-Dibromoethane	5	ug/kg	0.5	ug/kg		Y	50	150	30		Y	50	150	20
904	1,2-Dichlorobenzene	5	ug/kg	0.36	ug/kg		Y	50	150	30		Y	50	150	20
907	1,3-Dichlorobenzene	5	ug/kg	0.35	ug/kg		Y	50	150	20		Y	50	150	20
910	1,4-Dichlorobenzene	5	ug/kg	0.66	ug/kg		Y	50	150	20		Y	50	150	20
924	Dichlorodifluoromethane	5	ug/kg	0.5	ug/kg		Y	50	150	20		Y	50	150	20
933	1,1-Dichloroethane	5	ug/kg	0.36	ug/kg		Y	77	119	30		Y	56	130	54
936	1,2-Dichloroethane	5	ug/kg	0.34	ug/kg		Y	78	121	30		Y	56	126	38
948	cis-1,2-Dichloroethene	5	ug/kg	0.36	ug/kg		Y	77	114	30		Y	48	127	52
950	trans-1,2-Dichloroethene	5	ug/kg	0.41	ug/kg		Y	68	117	30		Y	47	127	58
943	1,1-Dichloroethene	5	ug/kg	0.52	ug/kg	C	Y	55	142	27	C	Y	43	147	27
986	1,2-Dichloropropane	5	ug/kg	0.69	ug/kg		Y	78	116	30		Y	54	125	43
998	cis-1,3-Dichloropropene	5	ug/kg	0.34	ug/kg		Y	71	125	30		Y	30	138	49
1000	trans-1,3-Dichloropropene	5	ug/kg	0.54	ug/kg		Y	67	125	30		Y	34	134	57
1332	Ethylbenzene	5	ug/kg	0.26	ug/kg		Y	79	114	30		Y	36	133	72
1515	2-Hexanone	20	ug/kg	0.63	ug/kg		Y	29	200	41		Y	20	190	70
1578	Isopropylbenzene	5	ug/kg	0.16	ug/kg		Y	50	150	20		Y	50	150	20
1774	Methyl acetate	10	ug/kg	1.4	ug/kg		Y	50	150	20		Y	50	150	20
1799	Methylcyclohexane	10	ug/kg	0.31	ug/kg		Y	50	150	20		Y	50	150	20
1811	Methylene chloride	5	ug/kg	0.67	ug/kg		Y	58	130	30		Y	45	129	49
1845	4-Methyl-2-pentanone	20	ug/kg	0.54	ug/kg		Y	68	142	60		Y	42	143	60
2772	Methyl tert-butyl ether	20	ug/kg	0.43	ug/kg		Y	70	130	30		Y	70	130	30
2355	Styrene	5	ug/kg	0.15	ug/kg		Y	80	114	30		Y	23	136	65
2439	1,1,2,2-Tetrachloroethane	5	ug/kg	0.34	ug/kg		Y	70	133	30		Y	33	162	90
2445	Tetrachloroethene	5	ug/kg	0.52	ug/kg		Y	72	120	30		Y	31	137	81
2489	Toluene	5	ug/kg	0.27	ug/kg	C	Y	71	130	24	C	Y	46	147	24
2515	1,2,4-Trichlorobenzene	5	ug/kg	0.27	ug/kg		Y	50	150	20		Y	50	150	20
2518	1,1,1-Trichloroethane	5	ug/kg	0.56	ug/kg		Y	67	123	30		Y	48	132	57
2522	1,1,2-Trichloroethane	5	ug/kg	0.39	ug/kg		Y	82	116	30		Y	58	128	52
2525	Trichloroethene	5	ug/kg	0.42	ug/kg	C	Y	70	131	23	C	Y	46	143	23
1428	Trichlorofluoromethane	5	ug/kg	0.34	ug/kg		Y	50	150	20		Y	50	150	20
2566	1,1,2-Trichloro-1,2,2-trifluoroethane	5	ug/kg	1.3	ug/kg		Y	50	150	30		Y	70	130	30
2613	Vinyl chloride	5	ug/kg	0.39	ug/kg		Y	24	152	30		Y	30	136	80
2627	Xylenes (total)	10	ug/kg	0.67	ug/kg		Y	80	114	30		Y	33	135	78
337	4-Bromofluorobenzene					X	Y	47	158		X	Y	47	158	
2735	1,2-Dichloroethane-d4					X	Y	61	130		X	Y	61	130	
2740	Toluene-d8					X	Y	60	143		X	Y	60	143	
2863	Dibromofluoromethane					X	Y	59	138		X	Y	59	138	

CRS TCL 4.2 VOCs
Matrix: Water

#	Method: Compound	RL	Units	MDL	Units	T	A	Check LCL	Check UCL	Check RPD	T	A	Spike LCL	Spike UCL	Spike RPD
11	Acetone	10	ug/L	1.1	ug/L		Y	22	200	95		Y	45	128	30
196	Benzene	1	ug/L	0.13	ug/L	C	Y	80	116	20	C	Y	78	118	20
323	Bromodichloromethane	1	ug/L	0.15	ug/L		Y	87	130	30		Y	80	146	30
340	Bromoform	1	ug/L	0.64	ug/L		Y	76	150	30		Y	58	176	30
343	Bromomethane	1	ug/L	0.41	ug/L		Y	64	129	30		Y	55	145	30
372	2-Butanone	10	ug/L	0.57	ug/L		Y	28	237	65		Y	71	123	30
459	Carbon disulfide	1	ug/L	0.13	ug/L		Y	73	139	30		Y	69	138	41
463	Carbon tetrachloride	1	ug/L	0.13	ug/L		Y	75	149	30		Y	63	176	30
521	Chlorobenzene	1	ug/L	0.15	ug/L	C	Y	76	117	20	C	Y	76	117	20
535	Dibromochloromethane	1	ug/L	0.18	ug/L		Y	81	138	30		Y	71	158	30
550	Chloroethane	1	ug/L	0.29	ug/L		Y	66	126	30		Y	59	142	30
569	Chloroform	1	ug/L	0.16	ug/L		Y	84	128	30		Y	83	141	30
574	Chloromethane	1	ug/L	0.3	ug/L		Y	48	123	30		Y	40	137	39
669	Cyclohexane	1	ug/L	0.12	ug/L		Y	70	130	30		Y	70	130	30
539	1,2-Dibromo-3-chloropropane	2	ug/L	0.67	ug/L		Y	70	130	30		Y	70	130	30
870	1,2-Dibromoethane	1	ug/L	0.24	ug/L		Y	70	130	30		Y	70	130	30
904	1,2-Dichlorobenzene	1	ug/L	0.13	ug/L		Y	70	130	30		Y	70	130	30
907	1,3-Dichlorobenzene	1	ug/L	0.14	ug/L		Y	70	130	30		Y	70	130	30
910	1,4-Dichlorobenzene	1	ug/L	0.13	ug/L		Y	70	130	30		Y	70	130	30
924	Dichlorodifluoromethane	1	ug/L	0.31	ug/L		Y	70	130	30		Y	70	130	30
933	1,1-Dichloroethane	1	ug/L	0.15	ug/L		Y	86	123	30		Y	88	127	30
936	1,2-Dichloroethane	1	ug/L	0.22	ug/L		Y	79	136	30		Y	71	160	30
948	cis-1,2-Dichloroethene	1	ug/L	0.17	ug/L		Y	85	113	30		Y	87	114	30
950	trans-1,2-Dichloroethene	1	ug/L	0.19	ug/L		Y	80	120	30		Y	85	116	30
943	1,1-Dichloroethene	1	ug/L	0.19	ug/L	C	Y	63	130	20	C	Y	62	130	20
986	1,2-Dichloropropane	1	ug/L	0.18	ug/L		Y	82	115	30		Y	87	114	30
998	cis-1,3-Dichloropropene	1	ug/L	0.14	ug/L		Y	84	130	30		Y	82	130	30
1000	trans-1,3-Dichloropropene	1	ug/L	0.19	ug/L		Y	84	130	30		Y	73	147	30
1332	Ethylbenzene	1	ug/L	0.17	ug/L		Y	86	116	30		Y	86	132	30
1515	2-Hexanone	10	ug/L	0.41	ug/L		Y	35	200	52		Y	81	128	30
1578	Isopropylbenzene	1	ug/L	0.13	ug/L		Y	70	130	30		Y	70	130	30
1774	Methyl acetate	10	ug/L	0.38	ug/L		Y	70	130	30		Y	70	130	30
1799	Methylcyclohexane	1	ug/L	0.13	ug/L		Y	70	130	30		Y	70	130	30
1811	Methylene chloride	1	ug/L	0.33	ug/L		Y	78	118	30		Y	82	115	30
1845	4-Methyl-2-pentanone	10	ug/L	0.32	ug/L		Y	78	141	32		Y	82	135	30
2772	Methyl tert-butyl ether	5	ug/L	0.17	ug/L		Y	70	130	30		Y	70	130	30
2355	Styrene	1	ug/L	0.11	ug/L		Y	85	117	30		Y	83	120	30
2439	1,1,2,2-Tetrachloroethane	1	ug/L	0.18	ug/L		Y	85	118	30		Y	88	116	30
2445	Tetrachloroethene	1	ug/L	0.29	ug/L		Y	88	113	30		Y	85	121	30
2489	Toluene	1	ug/L	0.13	ug/L	C	Y	74	119	20	C	Y	70	119	20
2515	1,2,4-Trichlorobenzene	1	ug/L	0.15	ug/L		Y	70	130	30		Y	70	130	30
2518	1,1,1-Trichloroethane	1	ug/L	0.22	ug/L		Y	78	140	30		Y	71	162	30
2522	1,1,2-Trichloroethane	1	ug/L	0.27	ug/L		Y	83	122	30		Y	86	129	30
2525	Trichloroethene	1	ug/L	0.17	ug/L	C	Y	75	122	20	C	Y	62	130	20
1428	Trichlorofluoromethane	1	ug/L	0.21	ug/L		Y	70	130	30		Y	70	130	30
2566	1,1,2-Trichloro-1,2,2-trifluoroethane	1	ug/L	0.28	ug/L		Y	70	130	30		Y	70	130	30
2613	Vinyl chloride	1	ug/L	0.22	ug/L		Y	61	120	30		Y	88	126	30
2627	Xylenes (total)	2	ug/L	0.28	ug/L		Y	87	116	30		Y	89	121	30
337	4-Bromofluorobenzene					X	Y	74	116		X	Y	74	116	
2735	1,2-Dichloroethane-d4					X	Y	61	128		X	Y	61	128	
2740	Toluene-d8					X	Y	76	110		X	Y	76	110	
2863	Dibromofluoromethane					X	Y	73	122		X	Y	73	122	

TCL 4.2 + DCBs & 124-TCB
Matrix: Water
CRS SVOCs

#	Method: Compound	RL	Units	MDL	Units	T	A	Check LCL	Check UCL	Check RPD	T	A	Spike LCL	Spike UCL	Spike RPD
1	Acenaphthene	0.2	ug/L	0.1	ug/L	C	Y	40	110	30	C	Y	36	110	30
5	Acenaphthylene	0.2	ug/L	0.1	ug/L		Y	43	110	30		Y	39	110	30
24	Acetophenone	1	ug/L	0.34	ug/L		Y	50	130	30		Y	50	130	30
122	Anthracene	0.2	ug/L	0.1	ug/L		Y	54	114	30		Y	46	110	30
158	Atrazine	1	ug/L	0.34	ug/L		Y	50	130	30		Y	50	130	30
3398	Benzaldehyde	1	ug/L	0.39	ug/L		Y	10	130	30		Y	10	130	30
202	Benzo(a)anthracene	0.2	ug/L	0.1	ug/L		Y	55	115	30		Y	52	110	30
205	Benzo(b)fluoranthene	0.2	ug/L	0.1	ug/L		Y	43	122	30		Y	33	114	30
208	Benzo(k)fluoranthene	0.2	ug/L	0.1	ug/L		Y	43	124	30		Y	32	121	30
210	Benzo(ghi)perylene	0.2	ug/L	0.1	ug/L		Y	45	120	30		Y	34	116	30
211	Benzo(a)pyrene	0.2	ug/L	0.1	ug/L		Y	43	116	30		Y	33	110	30
3474	1,1'-Biphenyl	1	ug/L	0.8	ug/L		Y	50	130	30		Y	50	130	30
289	bis(2-Chloroethoxy)methane	1	ug/L	0.32	ug/L		Y	39	110	30		Y	35	110	30
293	bis(2-Chloroethyl) ether	1	ug/L	0.1	ug/L		Y	34	113	30		Y	27	110	30
302	bis(2-Ethylhexyl) phthalate	2	ug/L	0.8	ug/L		Y	36	163	30		Y	40	140	30
348	4-Bromophenyl phenyl ether	2	ug/L	0.8	ug/L		Y	51	114	30		Y	42	113	30
403	Butyl benzyl phthalate	1	ug/L	0.8	ug/L		Y	53	126	30		Y	51	121	30
5101	Caprolactam	5	ug/L	0.8	ug/L		Y	50	130	30		Y	50	130	30
2751	Carbazole	1	ug/L	0.28	ug/L		Y	53	120	30		Y	49	114	30
518	4-Chloroaniline	2	ug/L	0.8	ug/L		Y	10	110	30		Y	10	110	30
578	4-Chloro-3-methylphenol	2	ug/L	0.8	ug/L	C	Y	39	110	30	C	Y	33	110	30
589	2-Chloronaphthalene	1	ug/L	0.1	ug/L		Y	39	110	30		Y	34	110	30
600	2-Chlorophenol	1	ug/L	0.29	ug/L	C	Y	27	110	30	C	Y	26	110	30
602	4-Chlorophenyl phenyl ether	2	ug/L	0.3	ug/L		Y	50	115	30		Y	43	113	30
633	Chrysene	0.2	ug/L	0.1	ug/L		Y	55	115	30		Y	52	111	30
860	Dibenz(a,h)anthracene	0.2	ug/L	0.1	ug/L		Y	46	122	30		Y	35	118	30
863	Dibenzofuran	1	ug/L	0.1	ug/L		Y	46	111	30		Y	41	110	30
891	Di-n-butyl phthalate	1	ug/L	0.67	ug/L		Y	55	122	30		Y	50	117	30
904	1,2-Dichlorobenzene	1	ug/L	0.29	ug/L		Y	23	110	30		Y	22	110	30
907	1,3-Dichlorobenzene	1	ug/L	0.8	ug/L		Y	19	110	30		Y	19	110	30
910	1,4-Dichlorobenzene	1	ug/L	0.34	ug/L	C	Y	19	110	30	C	Y	17	110	30
918	3,3'-Dichlorobenzidine	5	ug/L	0.37	ug/L		Y	19	110	30		Y	10	110	30
971	2,4-Dichlorophenol	2	ug/L	0.8	ug/L		Y	33	110	30		Y	30	110	30
1082	Diethyl phthalate	1	ug/L	0.6	ug/L		Y	33	134	30		Y	33	130	30
1145	2,4-Dimethylphenol	2	ug/L	0.8	ug/L		Y	12	110	30		Y	11	110	30
1149	Dimethyl phthalate	1	ug/L	0.29	ug/L		Y	15	143	30		Y	36	124	30
1167	4,6-Dinitro-2-methylphenol	5	ug/L	2.4	ug/L		Y	28	112	30		Y	25	110	30
1187	2,4-Dinitrophenol	5	ug/L	2.4	ug/L		Y	17	112	30		Y	11	119	30
1191	2,4-Dinitrotoluene	5	ug/L	0.27	ug/L	C	Y	52	123	30	C	Y	46	119	30
1193	2,6-Dinitrotoluene	5	ug/L	0.8	ug/L		Y	52	119	30		Y	48	115	30
1162	Di-n-octyl phthalate	1	ug/L	0.8	ug/L		Y	44	128	30		Y	36	124	30
1414	Fluoranthene	0.2	ug/L	0.1	ug/L		Y	54	122	30		Y	53	111	30
1417	Fluorene	0.2	ug/L	0.1	ug/L		Y	47	112	30		Y	43	110	30
1482	Hexachlorobenzene	0.2	ug/L	0.1	ug/L		Y	51	112	30		Y	40	113	30
1489	Hexachlorobutadiene	1	ug/L	0.27	ug/L		Y	13	110	30		Y	14	110	30
1492	Hexachlorocyclopentadiene	10	ug/L	0.8	ug/L		Y	10	110	30		Y	10	110	30
1497	Hexachloroethane	1	ug/L	0.8	ug/L		Y	12	110	30		Y	10	110	30
1535	Indeno(1,2,3-cd)pyrene	0.2	ug/L	0.1	ug/L		Y	46	121	30		Y	36	116	30
1566	Isophorone	1	ug/L	0.27	ug/L		Y	44	128	30		Y	34	125	30
1829	2-Methylnaphthalene	0.2	ug/L	0.1	ug/L		Y	35	110	30		Y	35	110	30
1851	2-Methylphenol	1	ug/L	0.8	ug/L		Y	30	110	30		Y	26	110	30
1857	4-Methylphenol	1	ug/L	0.8	ug/L		Y	32	110	30		Y	25	110	30
1932	Naphthalene	0.2	ug/L	0.1	ug/L		Y	31	110	30		Y	32	110	30
1960	2-Nitroaniline	2	ug/L	0.8	ug/L		Y	43	130	30		Y	31	129	30
1964	3-Nitroaniline	2	ug/L	0.28	ug/L		Y	45	116	30		Y	23	112	30
1968	4-Nitroaniline	2	ug/L	0.8	ug/L		Y	45	120	30		Y	26	115	30
1972	Nitrobenzene	1	ug/L	0.04	ug/L		Y	37	115	30		Y	26	118	30
1998	2-Nitrophenol	2	ug/L	0.28	ug/L		Y	29	110	30		Y	30	110	30
2001	4-Nitrophenol	5	ug/L	2.4	ug/L	C	Y	12	130	30	C	Y	13	127	30
2028	N-Nitrosodiphenylamine	1	ug/L	0.31	ug/L		Y	53	113	30		Y	28	118	30
2024	N-Nitrosodi-n-propylamine	1	ug/L	0.8	ug/L	C	Y	37	121	30	C	Y	25	119	30
3597	2,2'-oxybis(1-Chloropropane)	1	ug/L	0.4	ug/L		Y	25	128						
2118	Pentachlorophenol	5	ug/L	2.4	ug/L	C	Y	26	110	30	C	Y	23	110	30
2154	Phenanthrene	0.2	ug/L	0.1	ug/L		Y	52	114	30		Y	47	110	30
2155	Phenol	1	ug/L	0.6	ug/L	C	Y	14	112	30	C	Y	16	110	30
2252	Pyrene	0.2	ug/L	0.1	ug/L	C	Y	55	120	30	C	Y	54	115	30
##	1,2,4-Trichlorobenzene	1	ug/L	0.24	ug/L	C	Y	25	110	30	C	Y	25	110	30
2555	2,4,5-Trichlorophenol	5	ug/L	0.3	ug/L		Y	39	110	30		Y	36	110	30
2559	2,4,6-Trichlorophenol	5	ug/L	0.8	ug/L		Y	35	110	30		Y	34	110	30
1425	2-Fluorobiphenyl					X	Y	28	110		X	Y	28	110	
1426	2-Fluorophenol					X	Y	10	110		X	Y	10	110	
2512	2,4,6-Tribromophenol					X	Y	22	120		X	Y	22	120	
2736	Nitrobenzene-d5					X	Y	27	111		X	Y	27	111	
2737	Phenol-d5					X	Y	10	110		X	Y	10	110	
2738	Terphenyl-d14					X	Y	37	119		X	Y	37	119	

CRS SVOC
Matrix: Soil
TCL 4.2 + DCBs and 124-TCB

Method: SW846 8270C

#	Compound	RL	Units	MDL	Units	T	A	Check LCL	Check UCL	Check RPD	T	A	Spike LCL	Spike UCL	Spike RPD
1	Acenaphthene	6.67	ug/kg	3.3	ug/kg	C	Y	46	110	30	C	Y	10	200	30
5	Acenaphthylene	6.67	ug/kg	3.3	ug/kg		Y	47	110	30		Y	10	200	30
24	Acetophenone	100	ug/kg	9.2	ug/kg		Y	50	130	30		Y	50	130	30
122	Anthracene	6.67	ug/kg	3.3	ug/kg		Y	56	111	30		Y	10	200	30
158	Atrazine	200	ug/kg	9.1	ug/kg		Y	50	130	30		Y	50	130	30
3398	Benzaldehyde	100	ug/kg	12	ug/kg		Y	10	130	30		Y	10	130	30
202	Benzo(a)anthracene	6.67	ug/kg	3.3	ug/kg		Y	58	111	30		Y	10	200	30
205	Benzo(b)fluoranthene	6.67	ug/kg	3.3	ug/kg		Y	43	124	30		Y	10	200	30
208	Benzo(k)fluoranthene	6.67	ug/kg	3.3	ug/kg		Y	38	122	30		Y	10	200	30
210	Benzo(ghi)perylene	6.67	ug/kg	3.3	ug/kg		Y	44	120	30		Y	10	200	30
211	Benzo(a)pyrene	6.67	ug/kg	3.3	ug/kg		Y	44	115	30		Y	10	200	30
3474	1,1'-Biphenyl	50	ug/kg	27	ug/kg		Y	50	130	30		Y	50	130	30
289	bis(2-Chloroethoxy)methane	100	ug/kg	22	ug/kg		Y	42	110	30		Y	36	110	30
293	bis(2-Chloroethyl) ether	100	ug/kg	2	ug/kg		Y	41	110	30		Y	32	118	30
302	bis(2-Ethylhexyl) phthalate	50	ug/kg	19	ug/kg		Y	56	123	30		Y	10	200	30
348	4-Bromophenyl phenyl ether	50	ug/kg	13	ug/kg		Y	53	112	30		Y	44	120	30
403	Butyl benzyl phthalate	50	ug/kg	10	ug/kg		Y	57	121	30		Y	43	138	30
5101	Caprolactam	330	ug/kg	37	ug/kg		Y	50	130	30		Y	50	130	30
2751	Carbazole	50	ug/kg	27	ug/kg		Y	56	115	30		Y	10	162	30
518	4-Chloroaniline	150	ug/kg	17	ug/kg		Y	25	110	30		Y	11	110	30
578	4-Chloro-3-methylphenol	150	ug/kg	21	ug/kg	C	Y	42	110	30	C	Y	32	117	30
589	2-Chloronaphthalene	50	ug/kg	3.3	ug/kg		Y	46	110	30		Y	40	110	30
600	2-Chlorophenol	50	ug/kg	27	ug/kg	C	Y	39	110	30	C	Y	32	110	30
602	4-Chlorophenyl phenyl ether	50	ug/kg	13	ug/kg		Y	53	110	30		Y	47	116	30
633	Chrysene	6.67	ug/kg	1.1	ug/kg		Y	56	111	30		Y	10	200	30
860	Dibenz(a,h)anthracene	6.67	ug/kg	3.3	ug/kg		Y	45	122	30		Y	10	200	30
863	Dibenzofuran	50	ug/kg	3.3	ug/kg		Y	50	110	30		Y	10	200	30
891	Di-n-butyl phthalate	50	ug/kg	15	ug/kg		Y	57	119	30		Y	31	145	30
918	3,3'-Dichlorobenzidine	100	ug/kg	18	ug/kg		Y	31	110	30		Y	10	110	30
##	1,2-Dichlorobenzene	50	ug/kg	9.7	ug/kg		Y	42	110	30		Y	29	110	30
907	1,3-Dichlorobenzene	50	ug/kg	11	ug/kg		Y	40	110	30		Y	29	110	30
910	1,4-Dichlorobenzene	50	ug/kg	20	ug/kg	C	Y	38	110	30	C	Y	26	110	30
971	2,4-Dichlorophenol	150	ug/kg	20	ug/kg		Y	40	110	30		Y	33	110	30
1082	Diethyl phthalate	50	ug/kg	16	ug/kg		Y	55	114	30		Y	48	118	30
1145	2,4-Dimethylphenol	150	ug/kg	20	ug/kg		Y	28	110	30		Y	19	114	30
1149	Dimethyl phthalate	50	ug/kg	17	ug/kg		Y	54	112	30		Y	47	116	30
1167	4,6-Dinitro-2-methylphenol	150	ug/kg	80	ug/kg		Y	21	110	30		Y	10	110	30
1187	2,4-Dinitrophenol	330	ug/kg	80	ug/kg		Y	10	110	30		Y	10	110	30
1191	2,4-Dinitrotoluene	200	ug/kg	27	ug/kg	C	Y	55	116	30	C	Y	42	118	30
1193	2,6-Dinitrotoluene	200	ug/kg	21	ug/kg		Y	54	115	30		Y	28	137	30
1162	Di-n-octyl phthalate	50	ug/kg	27	ug/kg		Y	45	123	30		Y	10	182	30
1414	Fluoranthene	6.67	ug/kg	3.3	ug/kg		Y	55	118	30		Y	10	200	30
1417	Fluorene	6.67	ug/kg	3.3	ug/kg		Y	51	110	30		Y	10	187	30
1482	Hexachlorobenzene	6.67	ug/kg	2.1	ug/kg		Y	51	110	30		Y	37	122	30
1489	Hexachlorobutadiene	50	ug/kg	27	ug/kg		Y	39	110	30		Y	30	110	30
1492	Hexachlorocyclopentadiene	330	ug/kg	27	ug/kg		Y	10	110	30		Y	10	110	30
1497	Hexachloroethane	50	ug/kg	9	ug/kg		Y	38	110	30		Y	13	110	30
1535	Indeno(1,2,3-cd)pyrene	6.67	ug/kg	3.3	ug/kg		Y	45	121	30		Y	10	200	30
1566	Isophorone	50	ug/kg	13	ug/kg		Y	46	117	30		Y	32	129	30
1829	2-Methylnaphthalene	6.67	ug/kg	3.3	ug/kg		Y	46	110	30		Y	10	200	30
1851	2-Methylphenol	200	ug/kg	80	ug/kg		Y	36	110	30		Y	19	124	30
1857	4-Methylphenol	200	ug/kg	80	ug/kg		Y	40	110	30		Y	27	116	30
1932	Naphthalene	6.67	ug/kg	3.3	ug/kg		Y	42	110	30		Y	10	200	30
1960	2-Nitroaniline	200	ug/kg	9.1	ug/kg		Y	47	124	30		Y	31	141	30
1964	3-Nitroaniline	200	ug/kg	16	ug/kg		Y	44	110	30		Y	24	110	30
1968	4-Nitroaniline	200	ug/kg	26	ug/kg		Y	50	110	30		Y	23	124	30
1972	Nitrobenzene	100	ug/kg	2.2	ug/kg		Y	40	110	30		Y	33	111	30
1998	2-Nitrophenol	50	ug/kg	27	ug/kg		Y	35	110	30		Y	17	110	30
2001	4-Nitrophenol	330	ug/kg	80	ug/kg	C	Y	24	117	30	C	Y	10	125	30
2028	N-Nitrosodiphenylamine	50	ug/kg	21	ug/kg		Y	54	112	30		Y	10	169	30
2024	N-Nitrosodi-n-propylamine	50	ug/kg	27	ug/kg	C	Y	40	114	30	C	Y	30	121	30
3597	2,2'-oxybis(1-Chloropropane)	100	ug/kg	9.5	ug/kg		Y	36	116	30					
2118	Pentachlorophenol	150	ug/kg	80	ug/kg	C	Y	10	110	30	C	Y	10	182	30
2154	Phenanthrene	6.67	ug/kg	3.3	ug/kg		Y	54	110	30		Y	10	200	30
2155	Phenol	50	ug/kg	27	ug/kg	C	Y	39	110	30	C	Y	10	144	30
2252	Pyrene	6.67	ug/kg	3.3	ug/kg	C	Y	58	113	30	C	Y	10	200	30
##	1,2,4-Trichlorobenzene	50	ug/kg	27	ug/kg	C	Y	43	110	30	C	Y	33	110	30
2555	2,4,5-Trichlorophenol	150	ug/kg	25	ug/kg		Y	42	110	30		Y	32	112	30
2559	2,4,6-Trichlorophenol	150	ug/kg	80	ug/kg		Y	37	110	30		Y	22	110	30
1425	2-Fluorobiphenyl					X	Y	34	110		X	Y	34	110	
1426	2-Fluorophenol					X	Y	26	110		X	Y	26	110	
2512	2,4,6-Tribromophenol					X	Y	10	118		X	Y	10	118	
2736	Nitrobenzene-d5					X	Y	24	112		X	Y	24	112	
2737	Phenol-d5					X	Y	28	110		X	Y	28	110	
2738	Terphenyl-d14					X	Y	41	119		X	Y	41	119	

CRS Metals List
Matrix: Soil

Method: SW846 6010B

#	Compound		RL	Units	MDL	Units	T	A	Check LCL	Check UCL	Check RPD	T	A	Spike LCL	Spike UCL	Spike RPD
128		Antimony	1	mg/kg	0.39	mg/kg		Y	80	120	20		Y	75	125	20
140		Arsenic	1	mg/kg	0.3	mg/kg		Y	80	120	20		Y	75	125	20
1605		Lead	0.3	mg/kg	0.19	mg/kg		Y	80	120	20		Y	75	125	20
1539		Iron	10	mg/kg	4.9	mg/kg		Y	73	137	20		Y	75	125	20

Matrix: Water
Method: SW846 6010B

#	Compound		RL	Units	MDL	Units	T	A	Check LCL	Check UCL	Check RPD	T	A	Spike LCL	Spike UCL	Spike RPD
128		Antimony	6	ug/L	1.8	ug/L		Y	80	120	20		Y	75	125	20
140		Arsenic	10	ug/L	3.2	ug/L		Y	80	120	20		Y	75	125	20
411		Cadmium	2	ug/L	0.66	ug/L		Y	80	120	20		Y	75	125	20
1605		Lead	3	ug/L	1.9	ug/L		Y	80	120	20		Y	75	125	20
1539		Iron	100	ug/L	81	ug/L		Y	80	120	20		Y	75	125	20
1659		Manganese	15	ug/L	0.41	ug/L		Y	80	120	20		Y	75	125	20

PCBs - CRS
Matrix: Water

Method: SW846 8082

#	Compound		RL	Units	MDL	Units	T	A	Check LCL	Check UCL	Check RPD	T	A	Spike LCL	Spike UCL	Spike RPD
2082		Aroclor 1016	33	ug/kg	16	ug/kg	C	Y	34	127	30	C	Y	10	199	30
2085		Aroclor 1221	33	ug/kg	16	ug/kg										
2088		Aroclor 1232	33	ug/kg	14	ug/kg										
2091		Aroclor 1242	33	ug/kg	13	ug/kg										
2094		Aroclor 1248	33	ug/kg	17	ug/kg										
2097		Aroclor 1254	33	ug/kg	17	ug/kg										
2100		Aroclor 1260	33	ug/kg	17	ug/kg	C	Y	32	141	30	C	Y	10	199	30
2732		Decachlorobiphenyl					X	Y	10	199		X	Y	10	199	
2739		Tetrachloro-m-xylene					X	Y	10	196		X	Y	10	196	

Matrix: Soil
Method: SW846 8082

#	Compound		RL	Units	MDL	Units	T	A	Check LCL	Check UCL	Check RPD	T	A	Spike LCL	Spike UCL	Spike RPD
2082		Aroclor 1016	0.5	ug/L	0.17	ug/L	C	Y	44	119	30	C	Y	10	166	30
2085		Aroclor 1221	0.5	ug/L	0.13	ug/L										
2088		Aroclor 1232	0.5	ug/L	0.16	ug/L										
2091		Aroclor 1242	0.5	ug/L	0.22	ug/L										
2094		Aroclor 1248	0.5	ug/L	0.1	ug/L										
2097		Aroclor 1254	0.5	ug/L	0.16	ug/L										
2100		Aroclor 1260	0.5	ug/L	0.17	ug/L	C	Y	41	118	30	C	Y	21	140	30
2732		Decachlorobiphenyl					X	Y	10	127		X	Y	10	127	
2739		Tetrachloro-m-xylene					X	Y	27	130		X	Y	27	130	

STEERMAN ENVIRONMENTAL

via electronic transfer and U.S. Mail

May 23, 2012

Michelle Kerr
Remedial Project Manager
U.S EPA – Region 5
77 W. Jackson Blvd.
Mail Code: S-6J
Chicago, IL 60604-3590

Re: Chemical Recovery Systems, Inc.
United States of America v. AK Steel Corporation et. al.,
Case No. 1:10-cv-00996-KMO
Notification of Sampling Activities

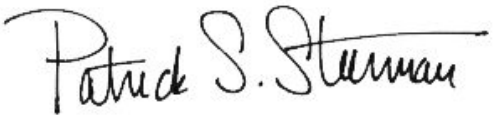
Dear Ms. Kerr:

In accordance with Paragraph 27 of the RD/RA Consent Decree for the Chemical Recovery Systems, Inc. ("CRS") Site entered July 16, 2010 in the above-captioned case ("Consent Decree"), the CRS Site RD/RA Group Settling Performing Defendants ("Group") are required to notify U.S. EPA and the State of Ohio not less than thirty (30) days in advance of any sample collection activity unless shorter notice is agreed to by U.S. EPA. Section 27 also states that U.S. EPA or the State shall have the right to take any additional samples that U.S. EPA or the State deem necessary. In accordance with the schedule included in the Supplemental Additional Groundwater Studies Work Plan ("the Plan") that was approved by U.S. EPA on May 14, 2012, implementation of field activities outlined in the plan will begin on May 28, 2012. As required by the Plan, certain environmental samples will be collected during the field activities and analyzed. The exact schedule for collection of these samples cannot be precisely determined thirty (30) days in advance, and will depend on the availability and scheduling of personnel. Therefore, the Group requests that U.S. EPA agree that this notice serves as the required sample collection notification under the Consent Decree for the field activities under the Plan and the Group will endeavor to provide further notification when specific sampling events are scheduled to allow at least seven days for EPA or the State of Ohio to arrange for samples as necessary.

If you have any questions or wish to discuss this further, please contact me at (770) 992-2836, or by electronic message at psteerman@charter.net.

Steerman Environmental
Management & Consulting, LLC
422 Creek View Lane
Roswell, Georgia 30075
(404) 421-3275 - Voice
psteerman@charter.net

Best Regards,



Patrick S. Steerman
CRS Site Project Coordinator

ec: Larry Antonelli, Ohio EPA
Richard Karl, Director, Superfund Division EPA Region 5
Doug McWilliams, CRS Site RD/RA Group Chair and Common Counsel
Tom Nash, Esq., U.S. EPA
Jim Peeples, Brown & Caldwell
CRS Site RD/RA Group Performing Parties

CRS Site AGWS-S Schedule of Field Activities

Field Task	Week of 6/4					Week of 6/11					Week of 6/18					Week of 6/25					Week of 7/2					Week of 7/9					Week of 7/16					Week of 7/23					Week of 7/30				
Packer Testing																																												
Bedrock Coring																																												
Transducer Installation																																											
Download Data																																											
Remove Transducers																																											
Seep Inspection																																											
Surface Water Sampling*																																											
NAPL Sampling/Drawdown																																											
NAPL Measurement																																											
River Bed Survey																																											
Location Survey																																											

* Actual surface water sample dates will be dependent on weather and river stage.

STEERMAN ENVIRONMENTAL

via electronic transfer and U.S. Mail

September 6, 2012

Michelle Kerr
Remedial Project Manager
U.S EPA – Region 5
77 W. Jackson Blvd.
Mail Code: S-6J
Chicago, IL 60604-3590

Re: Chemical Recovery Systems, Inc.
United States of America v. AK Steel Corporation et. al.,
Case No. 1:10-cv-00996-KMO
Proposed Revision to AGWS-S Surface Water Sampling

Dear Ms. Kerr:

Enclosed is a Brown and Caldwell letter proposing a modification to surface water sampling specified in the Additional Groundwater Studies Supplemental ("AGWS-S") Work Plan. As we have discussed, and as included in the June and July 2012 Monthly Progress Reports, surface water sampling cannot be performed as originally proposed in the AGWS-S work plan. Transducers installed in the river confirm that river level elevations are controlled by a dam located just downstream of the Site and we do not see fluctuations in the river level that were anticipated during development of the work plan. Since the purpose of surface water sampling is to confirm the results for samples analyzed as part of the Remedial Investigation/Feasibility Study, Brown and Caldwell proposes to collect and analyze one additional round of samples to supplement and confirm current data. To ensure that the surface water sampling results can be available in time for preparation of the Additional Groundwater Study report, currently scheduled for submittal on October 8, 2012, the CRS Site RD/RA Group is requesting that U.S. EPA approve this modification to the AGWS-S work plan by electronic mail as soon as practicable. Intermittent rain has been forecast this week, which may present an opportunity to sample under optimal conditions on or before September 10th.

If you have any questions or wish to discuss this further, please contact me at (770) 992-2836, or by electronic message at psteerman@charter.net.

Best Regards,

Patrick S. Steerman

Patrick S. Steerman
CRS Site Project Coordinator

Steerman Environmental
Management & Consulting, LLC
422 Creek View Lane
Roswell, Georgia 30075
(404) 421-3275 - Voice
psteerman@charter.net

ec: Larry Antonelli, Ohio EPA
Richard Karl, Director, Superfund Division EPA Region 5
Doug McWilliams, CRS Site RD/RA Group Chair and Common Counsel
Tom Nash, Esq., U.S. EPA
Mike Watkins, Brown & Caldwell
CRS Site RD/RA Group Performing Parties

6055 Rockside Woods Blvd., Suite 350
Independence, Ohio 44131
Tel: 216-606-1300
Fax: 216-606-1350
www.brownandcaldwell.com

September 06, 2012

Brown AND Caldwell

142766

Patrick S. Steerman
CRS Site Project Coordinator
422 Creek View Lane
Roswell, GA 30075

Subject: AGWS-S Surface Water Sampling Scope Modification
United States of America v. AK Steel Corporation et. al.
Case No. 1:10-cv-00996-KMO
Chemical Recovery Systems Superfund Site, Elyria, Ohio

Dear Mr. Steerman:

The Chemical Recovery Systems, Inc. (CRS) Site RD/RA Group Settling Performing Defendants (the Performing Parties) have completed one of the three surface water sampling events scoped for the CRS Site in the approved Additional Groundwater Studies-Supplemental (AGWS-S) Work Plan. The approved scope includes sampling the East Branch of the Black River during base flow conditions, then again at river stages that are approximately 0.5 ft. and 1.0 ft. above base flow conditions, respectively, with sampling events separated by at least one week. The first round of samples were collected on June 11, 2012 during base flow conditions, corresponding to a river stage elevation of approximately 683 ft, msl. The purpose of the surface water sampling program is to verify the results obtained during the Remedial Investigation. The remaining two scoped sampling events have not been completed because of the river stage's failure to meet the sampling criteria of 0.5 ft. and 1.0 ft. above base flow river stage. These circumstances were discussed in the June and July 2012 Monthly Progress Reports.

The Site received two significant rain events (approximately one inch or more of rain received over a 24-hour period) in June (June 17 and 18) and July (July 3). Brown and Caldwell visited the Site shortly after each of those rain events in an attempt collect the additional rounds of surface water samples corresponding to the 0.5' and/or 1.0' above base flow river stage. However, in both cases the river stage was found to be within 0.2' of base flow river stage. Review of the transducer data indicated that the river rose one to two feet during and immediately following the rain events, but quickly returned to near base flow conditions, typically within about an hour. The immediate increase was apparently in response to surface runoff "flushing" via overland flow and storm sewer discharge. The lack of a sustained increase in river stage following significant rain events suggests that the fixed dam across the East Branch of the Black River, located less than ½ mile downstream from the Site, serves to regulate the river stage at the Site and prohibits a sustained elevated river stage. Based on review of FEMA maps made available by the City of Elyria Engineering Department, the elevation of the top of the dam is approximately 381 ft, msl. The elevation of the dam, coupled with Brown and Caldwell's observation of continuous flow over the dam during multiple inspection dates, supports the dam serving to regulate the river stage at the Site.

Although the sampling criteria identified in the approved Work Plan for the second and third rounds of samples have not been met, there is still benefit in collecting an additional round of samples to verify the first round results, and Brown and Caldwell proposes that an additional round of samples be collected. This second round will target a date approximately two days after a significant rain event, which generally corresponds to elevated groundwater table levels in response to the event; however, in any event the additional round will be collected by September 10, 2012 in order to not delay work on the AGWS-S Summary Report.

If you or USEPA has any comments or concerns regarding the information and proposed sampling approach discussed above, please contact me at (216)-606-1309.

Sincerely,

Brown and Caldwell

Michael L. Watkins

Michael L. Watkins
Project Manager

ec: CRS Site RD/RA Group Performing Parties
Doug McWilliams, CRS Site RD/RA Group Chair and Common Counsel

{In Archive} Re: CRS Site - AGWS-S Work Plan Modfication

Michelle Kerr to: Patrick Steerman 09/06/2012 11:48 AM

Cc: "McWilliams, Douglas A.", Larry Antonelli, Mike Watkins, Thomas Nash

Archive: This message is being viewed in an archive.

Pat, this modification is acceptable.

Thank you,

Michelle Kerr
US EPA Region 5 Superfund Division
Remedial Project Manager
77 W. Jackson Blvd. SRF 6J
Chicago, IL 60604
Fx: 312.697.2658
T: 312.886.8961

Patrick Steerman | Michelle: Attached is a letter proposing a modific... | 09/06/2012 10:07:24 AM

From: Patrick Steerman <psteerman@charter.net>
To: Michelle Kerr/R5/USEPA/US@EPA, Thomas Nash/R5/USEPA/US@EPA, Richard Karl/R5/USEPA/US@EPA, Larry Antonelli <larry.antonelli@epa.state.oh.us>,
Cc: "McWilliams, Douglas A." <Douglas.McWilliams@squiresanders.com>, Mike Watkins <MWatkins@Brwncald.com>
Date: 09/06/2012 10:07 AM
Subject: CRS Site - AGWS-S Work Plan Modfication

Michelle:

Attached is a letter proposing a modification of the Additional Groundwater Studies Supplemental Work Plan. As we have discussed, we are proposing to reduce the number of surface sampling events to two. The plan originally proposed three rounds but the expected changes in the river level elevation have not been observed. The presence of a dam just downstream of the site maintains the river level at a base flow elevation.

Please let me know if U.S. EPA approves this change so that we may schedule the next round of samples.

If you have any questions or comments, please give me a call or respond to this message.

Best Regards,

Pat

Patrick S. Steerman

Steerman Environmental Management and Consulting

422 Creek View Lane
Roswell, Georgia 30075
(770) 992-2836 – Office

(404) 421-3275 – Mobile
psteerman@charter.net[attachment "09-06-12 CRS Site AGWS-S Proposed Surface Water Sampling Modification.pdf" deleted by Michelle Kerr/R5/USEPA/US]