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August 8, 2013

Mr. Michael Mikulka
United States Environmental Protection Agency
77 West Jackson Boulevard
Mail Code: LU-9J
Chicago, IL 60604

Re: RCRA Facility Investigation Work Plan, Solid Waste Management Unit 23 and Additional Areas of Potential Concern AA 02 through AA 09, Revision 3, July 2013; AK Steel Middletown Site, Middletown, Ohio

Dear Mr. Mikulka:

Please find enclosed two (2) paper copies and one electronic copy of the *RCRA Facility Investigation Work Plan, Solid Waste Management Unit 23 and Additional Areas of Potential Concern AA 02 through AA 09*, Revision 3, July 2013. The enclosed document is considered final based upon the USEPA et al. approval letter received on July 29, 2013 (dated July 23, 2013). All modifications in response to USEPA et al. comments are incorporated into the enclosed version of the RFI Work Plan. Please note that a technical correction was made to the last sentence of the third paragraph on page 35 of the Workplan. The word "surveyed" was replaced with "GPS-located" to be consistent with the intent and the Data Management Plan.

Please contact me at (740) 373-1266 or mrochotte@kemron.com, or Pat Gallo at (513) 425-3476 or Pat_Gallo@aksteel.com with any comments or questions regarding this submittal.

Sincerely,
KEMRON Environmental Services, Inc.

Mary Lou Rochotte, CPG, PMP
Senior Project Manager

Enclosure: Two paper RFI WP copies and one CD-ROM

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REPONSES TO USEPA et al. February 28, 2013 CONDITIONAL APPROVAL LETTER: SWMU 23 and 8 Additional Areas RCRA Facility Investigation Work Plan (dated November 2012)

On February 28, 2013, AK Steel and KEMRON received a conditional approval of the above referenced RCRA Facility Investigation (RFI) Work Plan. Each condition is repeated below in italic font, with responses for the conditions presented afterward. The responses to the conditions have been incorporated into the attached April 26, 2013 Revision 1 of the RFI Work Plan. Change pages are provided for those figures, tables and portions of attachments that were edited in response to the conditions. A full revision of the text is provided due to the number of comments received.

Please note that one page of the Public Involvement Plan (PIP) is included as a change page. This page was revised based on final Quality Control review, which identified the need for future updates and changes in the mailing list for Consent Decree work as the location of the work changes through time compared to the location of Interim Measures implemented to date.

Where text and table change pages are provided, both a redline markup and an unmarked version of the pages are provided. All revised figures have an April 2013 date. The entire primary RFI Work Plan text is labeled as Revision 1, April, 2013.

USEPA General Comments:

1. Page 1 of Attachment 2 to the Consent Decree identifies six steps in the RCRA Corrective Actions Program that AK Steel is to complete during the conduct of the RFI/CMS. Step 5 requires that AK Steel "Evaluate the overall integrity of existing containment structures and activities at the Facility and Additional Areas intended for long-term containment." All three lagoons are said to have been closed. Two of the lagoons have clay caps intended for long-term containment. The drying bed lagoon is said to have closed earlier but it isn't clear if that closure involved engineering controls intended for long-term containment. Revise the work plan to include tasks for evaluating the overall integrity of the existing long- term containment at SWMU 23.

Response: The data quality objectives (DQOs) and text have been updated to present a phased approach to address this comment. If Constituents of Potential Concern (COPCs) concentrations in soil samples indicate one or more COPC(s) is/are present at leachable levels, based upon comparison to relevant USEPA Soil Screening Levels (SSLs), USEPA RSLs, or other relevant standards, permeability testing will be performed.

2. *The work plan appears to recognize the need to determine the nature and extent of contamination associated with SWMU 23 and the Additional Areas (AAs) as it repeatedly states that this is an objective of the investigation. However, the included investigations of the AAs inappropriately invoke risk assessment scenarios to limit the extent of the investigations. Examples include limiting horizontal delineation in the AAs to the upper three feet of soil as 'This 3 foot upper horizon is the most accessible portion of the soil for potential human or ecological exposure to potential PCB concentrations, and will be critical data for use in the*

risk assessments to be conducted under this RFI" and terminating the initial borings in the AA's 12 feet based on "a depth that correlates to residential foundation or basement construction on each parcel" (work plan pages 26, 29, 37, and elsewhere). Similarly, borings in the SWMU 23 area are inappropriately limited to 15 feet as "A review of the well logs in the area indicates that a maximum soil boring depth of 15 feet will ensure that groundwater contamination will not inadvertently occur, should RCRA metals and/or VOC constituents be present in subsurface soils at elevated levels" (workplan page 28 and elsewhere). Remove references to risk assessment scenarios and concern about causing groundwater contamination at SWMU 23 as limiting factors in the investigation throughout the work plan (including all attachments and figures). Revise to the work plan and the DQOs in Table 4 to include investigations that will determine the actual nature and extent of contamination independent of such considerations and in accordance with the comments below.

Response: Multiple changes to text were made to the workplan to address this comment. All references to 12 and 15 foot depth limitations or references to potential basement construction depths were removed and clarifications made in the text concerning evaluating nature and extent. For purposes of clarification regarding how soil samples will be screened, please note that Region 5 ecological screening levels (ESLs) will be used in conjunction with USEPA Regional Screening Levels (RSLs) to screen soil samples collected in the top three (3) feet of soil. For depths greater than 3 feet, the soil RSLs will be applied for screening purposes. The 3 foot horizon is consistent with the potential exposure for ecologic receptors of interest. This clarification is provided in the Data Quality Objectives (DQOs) and in Section 2.2.5 of the text. Specifically, the following language has been added at the end of Section 2.2.5:

> "Data screening for all parcels will include screening of soil samples to a depth of three (3) feet against ecological screening levels. This depth is consistent with the depths in relevant ecological risk assessment guidance, based upon the potential ecological receptors of interest. Additionally, all samples analyzed from soil borings will be screened against relevant USEPA Regional Screening Levels (RSLs). For any property not owned by AK Steel, residential (e.g., unrestricted) levels will be used for RFI screening purposes. Leachability to groundwater will be assessed based upon soil concentrations detected, with standard regulatory methods such as USEPA Soil Screening Levels, protection of groundwater RSLs and/or other relevant screening values applied for evaluation. This screening approach will assure that the above identified data gaps are addressed in performance of the RFI."

All subsequent text references to screening of soil samples will be conducted in accordance with this methodology. The DQOs also have been reviewed and edited as necessary to assure clarity in the screening process that will be applied to the analytical data from soil samples.

3. Revise the work plan to clarify what the term "site" encompasses as used in the phrase "site activities" on pages 18 and 19 and in the phrase "site-specific data" on page 36.

Response: The text has been revised to clarify the intent.

USEPA Specific Comments

4. Page 4, 2.1.1, SWMU 23, first paragraph on page, last sentence. Thank you for providing these recently identified files. Revise the work plan to include this information in an appendix or as an attachment to the revised work plan.
Response: This information has been included as Attachment H.

5. Page 6, 2.1.4, last paragraph, first and second sentences. Delete the first sentence. Significant portions of many of the Additional Areas (AAs) are within the 100 year Dick's Creek floodplain (see CCR, Figure 6). The floodplain is by definition ecologically significant habitat. Note also that records indicate that the dredge spoils were placed primarily on the southern portion of each parcel. It is the southern portions of the parcels that are in the 100 year flood plain. Revise second sentence, this discussion, and the remainder of the work plan accordingly.
Response: The referenced portion of the text has been edited to indicate the 8 AAs are within the Dicks Creek floodplain. Subsequent text of the RFI Work Plan has been edited for consistency. Please also refer to edits incorporated to the Preliminary Site Conceptual Model for the 8 AAs.

Numerous other changes have been made in the work plan text that are relevant to similar comments.

6. Page 9, 2.1.5.2, first complete sentence and first sentence of first complete paragraph. The first complete sentence states "The ODNR has no records of endangered species or species of concern in the area." The first sentence of the next paragraph states "The ODNR has one record of a state threatened species-the Peregrin Falcon (Falco peregrinus) in the project area." Revise the work plan to correct the first complete sentence on page 9.
Response: The text has been edited to state: "The ODNR has one record of a state threatened species-the Peregrin Falcon (*Falco peregrinus*) in the project area."

7. Page 10, 2.2.1.1, SWMU 23, third paragraph. Revise the work plan to discuss the influent to the treatment system, the treatment provided, and the effluent to the lagoons to the extent this information is available.
Response: Additional information has been added to this section to describe the treatment process, based on historic knowledge of AK Steel personnel. No facility-specific records detailing the process were able to be located.

8. Starting on page 10, 2.2.1.1, SWMU 23, groundwater discussion. Please revise the work plan to provide potentiometric surface maps for each unit being monitored as part of the investigation, or better, superimpose the potentiometric surface on figures depicting existing and proposed monitoring well locations.
Response: Figures 5 and 12 have been revised to include general groundwater flow direction arrows. Currently available potentiometric maps only indicate one or two potentiometric lines in the vicinity of SWMU 23, based upon a limited monitoring network in this area. The directional arrows are based on a review of multiple groundwater flow maps. As part of the RFI, additional

groundwater monitoring wells will be installed, and potentiometric maps will be updated in the RFI Report.

9. Page 11, 2.2.1.1, SWMU 23: (a) on page 11, there is a discussion of existing ground water modeling information and a reference to production well 34 at AK Steel. Yet, the location of production well 34 is not shown on any of the supporting Figures. Add the location of this production well and any other relevant production wells to the Figures. (b) At page 11, there is a discussion of existing wells installed near SWMU 23. The discussion includes GM-3S, and the boring and well logs for that well are included in Appendix A, but the location of the well is not shown on the Figures. Add the location of this monitoring well and any other relevant monitoring wells to the Figures. (c) The RFI should evaluate if SWMU 23 is a source of TCE, and, if so, delineate the nature and extent (d) existing monitoring wells GM-lS, GM-lD and GM-2S appear to be upgradient monitoring wells, based on the groundwater modeling information and the existing AK Steel production well pumping scheme. That may not have been the case in the past, as the direction of groundwater flow may currently be the opposite of past conditions, and may revert to that condition in the future if the production well pumping scheme changes significantly. Revise the work plan to reflect the above considerations.

Response: a) Figure 5 has been revised to show proximity of production wells 34 and35 to SWMU 23; b) GM-03S has been added to Figure 5; c) analysis of soil and groundwater samples from SWMU 23 will include TCE as an analyte. Please note that the full target compound list (TCL) for VOCs is included in the analyses proposed for SWMU 23 groundwater samples (please refer to RFI WP QAPP, Attachment B for complete analytical list). The analytical results of groundwater samples will be evaluated and if deemed necessary additional delineation will be proposed; d) the production well pumping scheme historic records will be reviewed in conjunction with RFI groundwater data. However, KEMRON notes that groundwater monitoring wells sampled as part of the RFI will provide current analytical results from groundwater for all COPCs, as well as samples of in-situ soil conditions. If neither in-situ soils nor groundwater contain COPCs at unacceptable risk levels, further evaluation of historic pumping conditions would not be necessary. If RFI analytical results indicate further evaluation is necessary, significant historic records regarding the sitewide groundwater monitoring, and both current and past modeling evaluations (conducted by ARCADIS, formerly Geraghty and Miller) will be evaluated as part of the site groundwater records. The evaluation of the available history regarding relevant well installation(s), pumping and effects on groundwater flow will be incorporated as determined necessary based on RFI data results.

10. Page 10, 2.2.1.1, SWMU 23, fourth paragraph. Revise the work plan to clarify where the solids were taken that were removed from the drying bed, or more specifically, were any of the solids disposed of in the vicinity of SWMU 23?

Response: Based upon AK Steel personnel's knowledge of the former operations at SWMU 23, solids removed from the drying bed were sent to onsite landfills at Middletown Works for disposal. No Coil Paint Plant records remain available for evaluation regarding the specific landfill(s) that would have received the materials. However, AK Steel confirms that none of the solids were stockpiled onsite or otherwise placed in waste piles. The text has been edited to incorporate the information.

11. Page 11, 2.2.1.1, SWMU 23, third paragraph, fourth sentence. This sentence states "It is noted that these measured water table depths are somewhat less than the top of well screens within the respective wells, therefore, the individual well screens are submerged below the water table interface." If the water levels are below the top of the screen, the screens cannot be submerged. Please reconcile.
Response: Text has been updated to clarify water table interface measurements are shallower in depth than the depth of the top of well screen for existing "shallow" aquifer wells GM-1S and GM-2S. A statement has also been added that well GM-1S was identified as an intermediate depth well in a historical document. On the basis of well screen depth versus water table measurement depth, it is assumed that wells GM-1S and GM-2S more accurately represent "intermediate" depth aquifer monitoring wells. Please note that the designations used in the RFI Work Plan are consistent with the potentiometric maps presented in the sitewide groundwater monitoring report dated March 14, 2013.

12. Page 12, Site-Wide Groundwater Monitoring, third paragraph. The semi-annual reports discussed in this paragraph are submitted to both Ohio EPA and U.S. EPA, and the monitoring program is now being conducted in support of the work required by the Consent Decree. Please revise accordingly.
Response: This text has been revised accordingly**.**

13. Page 14, 2.2.2, second paragraph. This data should have been evaluated for usability when the CCR was prepared. At this point, it needs to be evaluated in this work plan as it potentially impacts the identification of data gaps.
Response: This text has been revised to clarify what data is considered usable and what data gaps exist. To clarify, the referenced data was included in the CCR and determined to be usable. The specific sections and findings of the CCR are now referenced.

14. Page 15, 2.2.3, first paragraph, first sentence. Revise sentence to read "RCRA metals or VOC constituents are not known to <u>currently</u> be present above regulatory standards in environmental media at SWMU 23 from historic Coil Paint Plant operations."
Response: This text has been revised accordingly.

15. Page 15, 2.2.3, first paragraph, revise the work plan to delete the last sentence: "The lagoons have been closed for over thirty (30) years, and no on-going generation of release of the constituents of potential concern exists at SWMU 23." See page 10, last paragraph, first sentence "The potential exists for release of RCRA metals and volatile organic compound (VOC) constituents to the subsurface environment due to the unlined nature of the lagoons" and page 11, 2.2.1.1, second sentence of paragraph above Investigation of Groundwater Flow Conditions heading, "Available information indicates that there is release potential for metal constituents and VOCs into the environment from historical operation of the wastewater lagoons."
Response: The last sentence has been edited to delete the phrase "and no on-going generation of release of the constituents of potential concern exists at SWMU 23". As noted in the comment, the work plan also discusses the potential for a release, consistent with the CCR.

16. Page 15, 2.2.3, second paragraph, first sentence. Delete the phrase "from potentially multiple stream" so the sentence reads "The Additional Areas 02 through 09 have potential PCB-containing material sourced from the Dicks Creek stream bed and banks, due to reported spoils materials being placed on the properties at the time of stream dredging in the late 1960's and potentially at other times as well."
Response: This text has been revised to read "The AAs 02 through 09 have potential PCB-containing material sourced from the Dicks Creek stream bed and banks, due to reported spoils materials being placed on the properties at the time of stream dredging in the late 1960's and/or 1980's".

17. Page 15, 2.2.4.1, first sentence. Please revise the work plan to qualify as historic data, detected concentrations of VOCs above MCLs. Also, include a description of groundwater data gaps in this paragraph as was done for soils.
Response: Text has been modified in sections 2.2.4.1 and 2.2.1.1 to indicate what VOCs have been detected and which were above MCLs for SWMU 23 area monitoring wells for sampling events completed between 1989-2010. Also, in Sec 2.2.5, a sentence has been added indicating that existing shallow wells GM-1S and GM-2S are considered to be intermediate depth wells, and that monitoring of water table interface, or shallow aquifer groundwater quality conditions, is considered to be a data gap.

18. Page 17, 2.3, Preliminary Site Conceptual Model. The first incomplete paragraph on this page states "related to the Additional Areas would be those associated with the habitats of Dicks Creek. Potential ecological impacts of Dicks Creek and the associated riparian habitat are being addressed within the Dicks Creek interim measures effort." As noted in comment 5 above, significant portions of many of the AAs are within the 100 year floodplain. These areas are part of the riparian habitat for Dicks Creek and are outside of the area addressed by the Dicks Creek remediation. They are also the areas of the AAs most likely to have been used for sediment disposal. If PCBs are detected in these areas, they will need to be evaluated for risks to both human health and to the environment consistent with Figure 42 of the CCR. In addition, invertebrates will need to be evaluated for these areas. Revise the work plan accordingly.
Response: As indicated on Figure 45 of the CCR (Preliminary Site Conceptual Model for Additional Areas), "If site activities are shown to have resulted in chemical contamination in the AAs, ecological impacts from surface soil will be evaluated for these areas consistent with the exposure pathway for Dicks Creek floodplain soil as shown in Figure 42". Human receptors will be evaluated as indicated in Figure 45 of the CCR (surface soil for AAs).

Receptors of interest were determined as part of the CCR and soil invertebrates were not included as an ecological ROI for floodplain soil (Fig 42). Figure 9 of the RFI Work Plan has been revised to include soil invertebrates as potential ecological ROIs for direct contact with soil and soil ingestion pathways. The text has been edited consistent with the revised figure in response to this comment. The text has been edited as indicated below to address the comment.

2.3 Preliminary Site Conceptual Model

A preliminary site conceptual model (SCM) was prepared and presented in the CCR

(ARCADIS, 2012b). This section summarizes key points from the CCR related to the SCM. The CCR SCM Figures 48 and 49 are reproduced (and revised as necessary) in this RFI WP as Figures 8 and 9.

For purposes of this RFI WP, the existing preliminary SCMs for SWMU 23 and the AAs have been incorporated. The SCMs for SWMU 23 and for the AAs will be updated and refined following RFI data collection. A Risk Assessment Assumptions Report (RAAR) will be prepared, and the risks to human health and the environment will be assessed as part of the RFI.

It is currently anticipated that SWMU 23 will include an evaluation of risk to both potential human and ecological receptors. The need for ecological risk assessment of the AAs will be dependent upon the RFI data findings. The primary potential ecological receptors related to the AAs would be those associated with the habitats of Dicks Creek. Potential ecological impacts within Dicks Creek are being addressed as part of the Dicks Creek Interim Measures effort. Therefore, pending additional data acquisition and consistent with the Preliminary SCM for the AAs as included in the approved CCR (Fig 45 of the CCR), ecological receptors will not be evaluated for the AAs unless site soil data indicate chemical (PCB) constituents in surface soil (based on the 0-3' analytical results) that would be available to ecological receptors. The SCM (Fig 9) includes a pathway for ecological evaluation should the presence of PCBs above relevant ecological screening levels in surface soil of the AAs be confirmed. Similarly, SWMU 23 (summarized in Figure 8) will have an ecological risk assessment conducted based upon a comparison of surface soil concentrations of VOC and RCRA metals to relevant ecological screening levels.

19. Page 18, 2.3.2.3, last sentence. This sentence states that, among other things, chemicals of potential concern will be evaluated based on "…essential nutrient status." Please clarify how essential nutrient status enters into the evaluation of chemicals of potential concern, and explain how essential nutrient status is determined as there is no related sampling included in this work plan.

Response: The reference to essential nutrient status has been deleted, since only RCRA metals and VOCs are being analyzed at SWMU 23.

20. Page 18, 2.3.3, Preliminary Ecological Conceptual Site Model. Revise this discussion consistent with comment 18 above.

Response: Figure 9 of the RFI Work Plan has been revised to include soil invertebrates as potential ecological ROIs for direct contact with soil and soil ingestion pathways. The text has been edited as indicated below to address the comment. Please also see response to comment 18.

2.3.3 Preliminary Ecological Site Conceptual Model

Based on information regarding land and groundwater use at and around the site a preliminary identification of potentially exposed ecological populations under current and reasonably expected future land use was completed in the CCR. Figure 8 provides the preliminary SCM for SWMU 23. An ecological risk assessment will be performed for SWMU 23 consistent with this SCM.

As stated above, the potential ecological receptors within Dicks Creek are being addressed through the Dicks Creek interim measures effort. Therefore, pending additional data acquisition and consistent with the preliminary SCM originally developed in the approved CCR (see CCR Figure 45), ecological receptors will not be evaluated for the AAs and the ecological SCM for this effort is limited to SWMU 23 unless analytical data indicate chemical (PCB) contamination in surface soil of the AAs above USEPA Region 5 ecological screening levels (ESLs). The SCM (Figure 9) includes a pathway for ecological evaluation should the presence of PCBs above ecological screening levels in surface soil of the AAs be confirmed. Figure 9 identifies currently anticipated ecological ROIs for the 8 AAs should analytical results indicate the need for conduct of an ecological risk assessment. Ecological receptors for the AAs, as applicable, will be refined in the Risk Assessment Assumptions Report (RAAR).

The potential ecological ROIs identified in the CCR for SWMU 23 and the adjacent woodland habitat include:

- Soil Invertebrates;
- Plants; and,
- Terrestrial Wildlife.

The objectives of the ecological risk evaluation are to identify potentially significant risks of adverse effects on ecological resources at the site (if any), and to develop risk-based clean-up levels where potentially significant ecological risks exist. Potential ecological risks will be evaluated for terrestrial areas of the SWMU 23 site where (1) ecologically relevant habitat is present, and (2) historic site activities (e.g., wastewater discharges at SWMU 23) could potentially have resulted in chemical consituents exceeding acceptable levels.

Ecological components of the preliminary site conceptual model for the woodland habitat adjacent to SWMU 23 are described below, as summarized from the CCR. The ecological site conceptual model includes the identification of exposure pathways, receptors of interest, and assessment endpoints, as well as a discussion of the approach for identifying chemicals of potential ecological concern (COPECs).

21. Page 19, 2.3.3, Preliminary Ecological Site Conceptual Model. The first sentence at the top of the page states "The potential ecological ROIs identified in the CCR for the woodland habitat adjacent to SWMU 23 include:" The Site Conceptual Model in CCR Figure 8 is not limited to the adjacent woodland habitat. Revise to read "The potential ecological ROIs identified in the CCR for SWMU 23 and the adjacent woodland habitat include …"
Response: Text has been revised accordingly. Please also see responses to comments 18 and 20.

22. Pages 20 and 21, 2.3.3.3, Assessment Endpoints. Consistent with the third bullet, the first and second bullets should also include trophic structure
Response: Trophic structure was introduced in the third bullet with all bullets being additive (e.g., the second bullet ending in "and"), and with intent being implied consideration of more

than one trophic level in an ecosystem. Text has been revised to include "trophic structure" in each bullet and the ecosystem trophic structure will be assessed qualitatively for the woodland habitat.

23. Page 21, 2.4.1. Is any information available regarding how the third lagoon (drying bed) was closed? If so, please discuss in the revised work plan.
Response: No closure documentation was determined to be available, based upon review of files and discussion with AK Steel personnel. The text has been revised to state this.

24. Pages 21 and 22, 2.5, sentence immediately above DQO problem statements. The sentence states there are four problems to be addressed. Five problem statements then follow. In a possibly related matter, note that in Table 4, 5 is combined with 4. Please reconcile text within 2.5 and with Table 4.
Response: Table and text have been reconciled.

25. Pages 21 and 22, 2.5, DQO problem statements. Statements 3 and 4 include the phrase "Assessing potential, and evaluating actual, ecological and human health exposures…" Replace this phrase with "Evaluating potential current and future ecological and human health exposures…"
Response: This text and the DQO Table 4 have been revised to address the comment.

26. Pages 21 and 22, 2.5, DQO problem statements. Consistent with the requirements of the RFI/CMS SOW, add the following problem statement: "6. Evaluate the overall integrity of existing containment structures at SWMU 23." Revise the work plan and the DQOs (Table 4) to include this problem statement and the tasks necessary to evaluate the existing containment
Response: This problem statement has been added to section 2.5 and Table 4. The necessary tasks have been added to Table 4.

27. Page 23, 3.0, first paragraph, last sentence. Groundwater samples will be collected from both existing and new monitoring wells. Please revise accordingly.
Response: This text has been revised accordingly.

28. Page 25, 3.7. TSCA deed restrictions will be required for any areas left with PCB concentrations of 1 ppm or higher. Please include deed restrictions in the milestones.
Response: This comment has been withdrawn by USEPA; therefore, no response is provided.

29. Pages 26-33, 4.0. Please revise this section to reference specific SOPs by number.
Response: The SOP references have been inserted.

30. Page 26, 4.0 paragraph 4. Delete the last sentence "Based on potential exposure pathways that require evaluation, borings will be advanced to a maximum of 12 feet below grade (e.g., depth of basement construction)." See comment 2 above.

Also, this discussion would apply only to borings conducted in the AAs as borings conducted at SWMU 23 will need to penetrate further into native material than two feet.

Response: The text has been revised accordingly.

31. Page 26, 4.1, first paragraph, first sentence. The sampling plan proposes only adding two soil borings outside of SWMU 23. The lagoons cover an area that is approximately 200ft. x 300ft. At least one soil boring per side of the lagoon area should be included in this investigation.
Response: One additional boring location has been added to the south and the monitoring well planned for the northeast portion will also serve as a soil boring to provide one per side of the lagoon area. This text and Figure 12 have been revised accordingly.

32. Page 26, 4.1, SWMU 23 Sampling: It is proposed to install 2 additional monitoring wells to be screened in the shallow aquifer. Revise the work plan to state that the depth of the wells will be sufficient to determine the nature and extent of groundwater contamination, considering the target constituents.
Response: Text has been added in Sec 4.1 indicating that four new monitoring wells will be installed at SWMU 23. Three monitoring wells will be installed to monitor the shallow aquifer/water table interface. Well screen intervals will be set at an approximate depth range from 20-30 feet bgs (10-foot well screen lengths). One additional intermediate aquifer monitoring well has also been added with screen interval to be set at approximately 40-50 feet bgs. These monitoring well installations will complement existing intermediate and deep aquifer monitoring wells at the SWMU 23 location. The text of Section 4.1 also has been edited to include a statement that "Groundwater monitoring will be completed to determine the nature and extent of COPCs." Figure 12 has been revised to show the four well locations.

33. Page 27, 4.1, Existing Monitoring Well Inspection, first sentence. Revise the work plan to specify what information indicates GM-lD is downgradient of and monitoring SWMU 23? Include a potentiometric map in the figures for this unit. See comment 8 above.
Response: The second paragraph within the "Existing Monitoring Well Inspection" subsection indicates GM-1S and GM-1D monitoring wells to be upgradient of SWMU 23. KEMRON has reviewed numerous potentiometric surface maps for annual/periodic water level surveys completed from 1995 through 2012. The general groundwater gradient direction for both "intermediate" and "lower" aquifer groundwater has been added to Figure 5 and Figure 12 of the Work Plan. Historical potentiometric surface maps may be found in Appendix E-5 of the CCR and within the *2011 Site-Wide Groundwater Pumping and Groundwater Flow Monitoring Report* (Arcadis, 2011). A text edit was added to the "Monitoring Well Installation and Sampling" subsection indicating the addition of gradient flow direction to Figure 12.

34. Page 27, 4.1, Existing Monitoring Well Inspection. The text states that sampling data from "25 other site-wide monitoring wells……which were analyzed for trichloroethene (TCE), indicated no detectable TCE concentrations in deeper aquifer material groundwater at the SWMU 23 area." How many, if any, of the 25 other wells are in the vicinity of and down gradient of SWMU 23? If not monitoring SWMU 23, remove those wells from this discussion.
Response: There are no other wells in the vicinity of SWMU 23 currently being monitored for TCE via the sitewide monitoring program. The text has been edited accordingly.

35. Pages 27 carrying over onto page 28, 4.1, vapor intrusion pathway evaluation. Include evaluation of soil as well as groundwater sample results in the determination of whether soil vapor intrusion sampling and/or evaluation should be conducted.
Response: This text and DQOs have been revised accordingly.

36. Page 28, 4.1, Soil Boring Completions, first paragraph, last sentence. Revise consistent with comment 31 above.
Response: This text has been revised accordingly.

37. Page 28, 4.1, Soil Boring Completions, second paragraph. The work plan limits soil borings in the SWMU 23 area to 15 feet in order to "…ensure that groundwater contamination will not inadvertently occur, should RCRA metals and/or VOC constituents be present in subsurface soils at elevated levels." Remove this limitation from the work plan (see comment 2 above). Revise this section to indicate that all soil borings in the SWMU 23 area will be advanced until the water table is reached. With the exception of geotechnical samples collected from the cap, the focus of soil sampling should be the native material starting immediately beneath the backfill, and continuing down to the surface of the water table.
Response: This text has been revised accordingly. Geotechnical sampling has been addressed as a potential future RFI phase, with decision criteria included in the text and DQOs.

38. Page 28, 4.1, Soil Boring Completions, third paragraph. Describe how VOC soil samples will be collected in light of splitting every core longitudinally. Reference specific SOPs for collecting all samples, and identify the sequence of events from when the core is first removed from the borehole until VOC soil samples are collected.
Response: Soil samples collected for VOC analysis will be obtained in accordance with KEMRON SOP 12-SSP-01. This SOP requires immediate collection of the VOC sample fraction, filling the provided laboratory container completely, avoiding direct contact with the core liner. Soil samples for VOC analysis will therefore be collected and containerized at 2-foot intervals upon collection and cutting open of the core liner. An additional small volume of sample will be collected for headspace VOC determination (using a PID), placed in a small zip-lock bag and set aside for ambient temperature stabilization. Following collection of the VOC sample and headspace fractions, the remainder of the recovered core sample will be measured, photographed, and descriptively logged by the field geologist.

39. Page 28, 4.1, Soil Boring Completions, fourth and fifth paragraphs. The fourth paragraph states that "Cap material thickness will be described, but is not anticipated to be sampled for laboratory analysis." The fifth paragraph states that "… borings SB-2, SB-6 and SB-9 will have samples collected within the cap material. The cap material samples will be analyzed for RCRA metals, VOCs and grain size." Delete "but is not anticipated to be submitted for laboratory analysis" from the sentence in the fourth paragraph and revise the fifth paragraph to include permeability testing of the cap materials sufficient to adequately estimate the permeability of the cap(s). Revise the DQOs (Table 4) and this discussion to state that if contamination is detected beneath the cap, U.S. EPA's HELP model (or other tool) will be used to estimate leakage through the cap. It is assumed the cap is being analyzed for RCRA metals and VOCs as it was obtained on-site and its composition was not documented

during closure. It is also assumed that the sample results will be used to evaluate direct contact pathways during the risk assessments. Please clarify in the work plan if this is the case.

Response: The text and DQOs have been revised accordingly. Also please see sections 2.3.2.2 and 2.3.3.1 which specify exposure pathways to be included in the SWMU 23 risk assessment.

40. Page 28, 4.1, Soil Boring Completions, fifth paragraph. The work plan states "In general, a lagoon soil sample will be collected immediately below the surface cap material (estimated to be at a depth of approximately 2-3 feet below grade) for analysis." A second sample is proposed to be collected based on "the field- recorded PID reading between the interval of 3' below grade and the maximum boring depth above the water table interface." Samples of the backfill placed during closure are of limited use in terms of evaluating whether residual contamination in the SWMU 23 area has the potential to contaminate surrounding environmental media, particularly with respect to groundwater. Given the work plan proposes to submit only two samples per borehole to the lab for analysis, samples of lagoon backfill should only be submitted if field screening indicates contamination. The two samples selected from fixed locations for laboratory analysis should be collected from native material immediately beneath the lagoon backfill and native material immediately above the water table. As presently discussed in the work plan, additional samples should be selected for laboratory analysis depending on field screening results, including samples of the backfill or samples of native material collected between the two fixed location samples discussed above. Please reference a specific SOP for the field screening and describe how the decision will be made to submit additional samples based on field screening. Revise the DQOs (Table 4) to indicate that if significant contamination is detected in samples beneath the cap, the HELP model results will be used in conjunction with a leaching model to determine if contaminants are capable of migrating to the groundwater in concentrations that would elevate the groundwater above screening levels.

Response: Samples of lagoon backfill will only be submitted if field screening indicates contamination. The two samples selected from fixed locations for laboratory analysis will be collected from native material immediately beneath the lagoon backfill and native material immediately above the water table. Field headspace screening of soil samples will be conducted in accordance with SOP 12-PID-AKMW. In accordance with other comments, all SOPs are referenced by number and review has been conducted to assure all applicable SOPs are incorporated into the RFI Work Plan.

Headspace screening results will be recorded onto the field soil boring log. Anomalous headspace results, if detected, will be used to determine if a sample is submitted for laboratory analysis. If no anomalous headspace results are indicated, then the soil sample obtained from immediately above the water table interface will be submitted for analysis.

Section 4.1, “Soil Boring Completions” is revised as follows:

> **Soil Boring Completions**
> Figure 12 indicates the planned locations for soil boring completions. Three (3) soil borings will be completed within each of the areas once occupied by Lagoons 1 and 2

(northern and central smaller lagoons). Six (6) soil borings will be completed within the larger southern Lagoon 3. Boring locations will be staked in the field using measurements from existing key features (building structures, monitoring wells, etc.) as the lagoons are now buried with no visible surface expression of lagoon boundaries. Four (4) additional soil borings will be completed on each side of the lagoons within the AK Steel owned property to sample soil at locations exterior of the former lagoons (Figure 12), with the northeast ,east and west side borings being completed as water table monitoring wells. (Note that in addition to providing soil samples, boring locations SB-4 and SB-15 will be completed with shallow monitoring well installations, and SB-16 will be completed with an intermediate depth well installation. The shallow monitoring well location paired with SB-16 will not be sampled for soil.) Four additional surface soil samples (0 to 1 ft below ground surface) will be collected as indicated on Figure 12 for use in evaluating direct contact exposure pathways.

Continuous soil sampling will be advanced (via push tube or rotosonic drilling) until the water table is reached. Available data from monitoring wells indicates the water table to be within 25 feet of the ground surface. Existing borings logs for the SWMU 23 area wells indicate essentially medium to coarse sand and gravel as the predominant geologic formation at SWMU 23.

The field geologist will log individual soil cores for recovery percentage, soil type, and any notable environmental characteristics (discolorations, moisture, odors, non-native material, etc.). The field geologist will also be equipped with a photoionization detector (PID) to field screen individual soil samples for the presence of VOC in soil headspace samples. Soil samples will be retained at 2 foot sample intervals for headspace testing and for potential analytical sample selection. Individually collected samples will be kept in a cooler with ice, with separate headspace portions retained under ambient conditions. Photographs will be taken of individual open soil cores for documentation purposes.

Soil samples collected for VOC analysis will be obtained in accordance with KEMRON SOP # 12-SSP-01. This SOP specifies EPA Method 5035 will be followed for VOC soil analysis at RCRA sites. Soil samples for VOC analysis will be collected and containerized at 2-foot intervals upon collection and cutting open of the core liner. An additional small volume of sample will be collected for in-field headspace VOC determination (using a PID), placed in a small zip-lock bag and set aside for ambient temperature stabilization. Following collection of the VOC sample and headspace fractions, the remainder of the recovered core sample will be measured, photographed, and descriptively logged by the field geologist. Following this, another sample aliquot from each 2-foot interval will be removed from the core liner, placed into a stainless steel bowl, homogenized, then placed into a sample container for potential RCRA metals analysis.

Soil samples will be analyzed for VOC (EPA Method 8260) and RCRA metals (EPA Method 6010 and/or 6020). Two (2) soil samples per soil boring will be retained and submitted for laboratory analysis. Additional soil samples may be submitted based on field observation of soil samples, and/or field screening results.

Borings SB-2, SB-6 and SB-9 will have samples collected within the cap material. Cap material thickness will be described. The cap material samples will be analyzed for RCRA metals, VOCs and grain size (ASTM Method D-422). A decision on the need for permeability testing of the cap material will be made after laboratory analytical results of the soil borings are received and evaluated. If there COPC concentrations beneath the lagoons do not exceed relevant leaching values, no permeability testing will be performed. If COPC concentrations beneath the lagoons exceed the relevant leaching values, additional samples of the cap material will be collected for permeability testing (ASTM Method D-5084) and leakage through the cap(s) will be estimated using USEPA's HELP model (or other model with prior USEPA et al. approval). Please see the RFI QAPP (Attachment B) for further information regarding permeability testing methodology.

For lagoon soil borings, a minimum of two soil samples will be collected from 1) native material immediately below the lagoon backfill and 2) native material immediately above the water table. Additional samples beneath the surface cap material may be submitted for analysis based on the field-recorded PID reading. Field headspace screening of soil samples will be conducted in accordance with SOP# 12-PID-AKMW. Headspace screening results will be recorded on the field soil boring log. Anomalous headspace results, if detected, will be used to determine if one or more additional samples are submitted for laboratory analysis.

For soil borings outside of the lagoons, a minimum of two soil samples will be collected. One sample will be the native material immediately above the water table while the second sample will be selected based on the field-recorded PID reading.

Following completion of field headspace testing and final selection of samples for laboratory analysis, remaining soil sample material will be properly containerized, managed and disposed off-site as investigation derived waste (IDW). IDW will be temporarily staged on AK Steel secure property (location to be determined) designated for IDW temporary storage prior to disposal.

Open soil borings will be backfilled with granular bentonite to the ground surface to seal the boreholes. The ground surface will be patched with soil. Individual soil boring GPS locations will be measured with portable GPS for mapping location determination. Existing monitoring wells will also be GPS-located for mapping reference.

At the present time, no vapor intrusion pathway sampling and analysis is planned. Should VOCs be detected in soil or groundwater samples associated with SWMU 23 during the RFI, the concentrations of the detections will be evaluated and a determination will be made as to whether sampling and analysis of groundwater and/or soil for a potential vapor intrusion pathway is appropriate.

41. Page 28, 4.1, Soil Boring Completions, fifth paragraph. Revise the work plan to specify that surface soil samples will be collected at the four boring locations outside of the lagoons, and at four additional locations around the lagoons where borings are not conducted, in

support of evaluating direct contact exposure pathways.
Response: This text has been revised accordingly.

42. Page 29, 4.2, AA sampling: This section is confusing as to what is actually being proposed. Sampling at 3 foot intervals is discussed, but past downstream Reach 2 floodplain sampling was focused on the 0-8" zone, and past initial Reach 1 floodplain sampling was based on 0-1' and 1-2' floodplain soil layers. Please clarify specifically what you are proposing in the sampling scheme; perhaps a figure would be useful to illustrate the proposed sampling.
Response: The sampling strategy text has been revised to clarify and address the comment. The revised text and DQOs are generally consistent with the approach implemented in prior delineation investigation of floodplain soil. The approach will assure full delineation of PCB impacts. The dredge spoil sampling has been revised consistent with this and other comments to assure vertical and horizontal delineation, and to provide decision making criteria regarding which sampled intervals will be subject to immediate laboratory analyses and which will held for analyses pending the laboratory results of other samples. While the screening criteria (ESLs and/or RSLs) for the sample results are different from past delineation sampling and analysis, the approach is consistent with methodologies approved previously. Revisions include the specific text change in Section 4.2 below:

> The primary drilling and sampling objective on the AAs will be to determine the presence and thickness of potential dredged Dicks Creek sediment material used as fill for site grading or filling. Soil boring completion depths will be terminated when original soil horizons/root zones are encountered. Soil samples will be logged on a continuous basis with photographs taken of individual open soil cores for documentation purposes. PID headspace screening will be conducted on collected samples as described in SOP# 12-PID-AKMW. Based on the COPCs being limited to PCBs, and the absence of any PCB source that could result in non-aqueous phase liquids at the sites, no monitoring well installations are currently planned for completion for this portion of the RFI.
>
> Soil samples will be retained at continuous 1-foot sample intervals for logging purposes and for potential analytical sample selection. Detailed soil boring logs will be generated by the field geologist at each location. Field observations of recovered soil samples will note in detail changing soil characteristics with depth. The intent is to be able to differentiate surficial fill (non-dredged fill materials), from potential dredged fill material, from original native materials. Individual soil boring conditions and observations will dictate the depth of samples submitted for analysis from each boring, with a minimum of 4 samples submitted for analysis per boring (or fewer if sediment deposit depth is less than 3 feet below ground surface).
>
> The number of samples submitted for analysis from each borehole will be based on the thickness of the sediment in the borehole, visual observations, PID readings and analytical results. For this assessment, it is anticipated that (at a minimum) the following soil samples will be submitted from each boring for PCB analysis:
>
> - Soil intervals 0-1’ bgs, 1-2’ bgs and 2-3’ bgs; and

- Subsurface 1 ft. interval at base of sediment deposit (above native material horizon, if definable in field and if not included in 0-3' intervals).

Additional samples will be collected and analysis will proceed if necessary based on the following decision criteria:

- Top of native material sample (below sediment deposit interface, and if not included in 0-3' interval) will be collected. Laboratory analysis will be conducted if analytical results from the base of sediment sample (see above) exceed the relevant soil leaching to groundwater screening level(s);
- Subsurface soil below 3' depth and base of sediment sampling interval will be biased to any visual observations and field measurements potentially indicating presence of PCBs (such as oily materials/sheen, discoloration, elevated PID readings, etc.);
- For borings that meet or exceed ten (10) feet in depth, if analytical results indicate that either the 2-3' bgs interval or base of sediment deposit interval exceed the relevant RSL for COPCs, at least one mid-depth sample will be analyzed from that soil boring. Decisions regarding the number of mid-depth samples to be analyzed from borings less than ten (feet) in depth will be based upon the thickness of the sediment observed at each location, the PID readings and visual observations and the COPC concentrations determined to be present at the 2-3' and base of sediment intervals as compared to ESLs and/or RSLs.

43. Page 29, 4.2, third paragraph, and elsewhere in the work plan and attachments. The text states that "Soil boring completion depths will be terminated when one of the above features is determined, to the maximum depth of 12 feet." Remove 12 foot limitation on borings in the AAs. Also, remove encountering shallow groundwater as "one of the above features" that would limit vertical delineation if sediment is still present. The vertical extent of dredge materials needs to be determined regardless of depth or encountering the shallow water table. See comment 2 above.
Response: This text has been revised accordingly.

44. Page 29, 4.2, end of third paragraph. Revise to indicate that PID headspace analysis will be conducted on the soil samples. Headspace analyses proved to be relevant to the PCB releases at SWMU 39, and there is no reason to assume that they would not be potentially useful when investigating the dredged materials on the AAs.
Response: PID screening has been added.

45. Page 29, 4.2, third paragraph, last sentence. This sentence states "Individual soil boring conditions and observations will dictate the depth of samples retained for analysis from each boring, with up to 2 samples collected per boring. Revise the work plan to state the number of samples submitted for analysis from each borehole should be based on the thickness of the sediment in the borehole. Also, PID readings should be evaluated when selecting samples for analysis.
Response: Text clarification has been added to the Work Plan to provide the sampling strategy and decision making criteria to determine which samples require analyses to assure complete delineation. Both PID readings and visual observations will be used in decision making, along

with laboratory results of prioritized samples. Please also see response to Comment 42.

46. Page 29 carrying over to page 30, 4.2, discussion of sample density based on current land use. The last sentence of this discussion ties the density of the soil sampling to existing land use. AK Steel does not control land use on property that it does not own. Revise this discussion to indicate that all parcels not owned by AK Steel will be sampled assuming residential land. See comment 2 above.

Note also that the human health risk assessment will also need to assume residential land use for all properties AK Steel does not control.
Response: The same sampling density will be employed for all AAs, as illustrated on the Figures 13-20. We agree that all AAs will be assessed assuming residential and unrestricted land use. The intent of the text was to indicate that the number of samples collected on residential parcels initially versus as a contingency has been designed slightly differently to avoid protracted intrusion into these individuals' residences. All parcel sampling is planned on 75' centers as illustrated in the figures with no differentiation based on land use.

For any property not owned by AK Steel, the human health risk assessments will be based upon unrestricted land use, implementing residential screening levels. No COPCs will be screened out of the risk assessment process based on industrial/commercial screening levels for properties not owned by AK Steel. The RFI Work Plan has been reviewed and edited as necessary to resolve discrepancies, and to assure the text consistently indicates this approach. Please note, however, that the risk assessment for each AA is anticipated to also present results of a potential future industrial/commercial land use, as this information may be beneficial in future consideration of potential alternatives for these non-AK Steel owned parcels should any corrective measures need to be considered. Please also see response to General Comment 2.

47. Page 30, 4.2, first full paragraph, second sentence. Replace the phrase "on the Additional Areas" with "in bedrock."
Response: This text has been revised accordingly.

48. Page 30, 4.2, third full paragraph. The statement is made that "Soil samples which appear to define an interface between possible fill soil and underlying native soil (undisturbed soil horizon) will also be retained for analysis." It would seem necessary to submit a sample from the base of the sediment. It would also seem necessary to submit a sample of the native material beneath the sediment if the base of the sediment is contaminated. Revise the work plan.
Response: Text clarification has been added to the Work Plan indicating that samples will be collected and analyzed. Please also see response to Comment 42 for text revisions.

49. Page 30, 4.2, end of third full paragraph. Delete encountering groundwater as a condition for terminating borings. See comment 2 above. If the sediment extends below the water table, its entire depth will need to be determined and a sample will need to be submitted from the base of the sediment, groundwater notwithstanding. If sediments below the water table are contaminated, the groundwater will need to be sampled to determine if PCB

contaminated groundwater is recharging Dicks Creek.
Response: The text has been revised to delete encountering groundwater as termination. The text also has been edited to clarify that soil analytical results will be screened against relevant soil leaching screening levels. If analytical results indicate that PCB concentrations are present that pose a risk of PCBs leaching to groundwater, consistent with the DQOs, a separate phase of RFI would be executed to address groundwater PCB concentration investigation.

50. Page 30, 4.2, last paragraph, first sentence. Remove the 3 foot depth limitation on horizontal delineation. See comment 2 above.
Response: This text has been revised accordingly.

51. Page 30, 4.2, last paragraph, second sentence. Revise to state that for delineation, residential and/or ecological screening values will be used for all parcels AK Steel does not control, regardless of current land use. See comments 2 and 5 above.
Response: The delineation text has been removed from this section. Screening logic is covered by other sections. Please also see response to comment 46.

52. Page 30, 4.2, last paragraph, next to last sentence. "These locations will be sampled to the extent feasible based on the presence of buildings, utilities or other engineering limitations present on each parcel, and any limitations imposed by property owners via final access agreements to be acquired." Unless historic aerial photographs demonstrate that buildings and/or paved areas existed before the dredging and placement of the sediments occurred, PCB contaminated sediments may exist beneath the buildings and/or parking lots and should be sampled.
Response: AK Steel and KEMRON understand that the extent of PCB impacts are required to be fully delineated and believe the Work Plan text presents that commitment. The text in question is not intended to limit delineation, but rather to acknowledge that the precise locations of the borings must be confirmed in the field and may require adjustment in response to any identified limitations. Historic aerial photographs have been reviewed and will be considered and discussed in RFI results. However, please note that AK Steel's ability to install borings on private property, whether inside buildings or otherwise, may be limited due to site-specific conditions. Utilities must be avoided, and other engineering limitations may be encountered in the field that prevents installation of a boring in a particular location. Additionally, property owners may refuse to allow installation of borings through their private structures. Few structures other than residential structures on AAs that currently exist are observed to be present on aerial photographs from late 1960's when dredge spoils are reported to have initially been placed on the AAs. For structures currently present that were not in existence at the time of the earliest reported dredge spoil placement (based upon historic aerial photograph evaluation), the current figures, text and DQOs present the means by which AK Steel will assure delineation of PCBs. Should an unavoidable limitation be encountered in the field, the RFI samples will be placed outside the area of that limitation to provide coverage of the parcel such that delineation is completed. Please see revised RFI Work Plan DQOs regarding the step-outs planned to assure complete horizontal and vertical delineation of PCBs.

53. Page 31, 4.2, first paragraph. Revise this paragraph to include vertical as well as horizontal delineation. Also, note that vertical delineation should not be tied to results from

the upper three feet of soil as results in this horizon are not representative of the entire depth of the sediment. The full extent of PCB contamination, both horizontally and vertically, needs to be determined independent of any land use assumptions. See comment 2 above.
Response: AK Steel agrees that the upper three feet of soil are not representative of an entire sediment thickness at all areas of investigation. The upper zone is, however, especially critical data for conduct of risk assessment. Text changes have been made to clarify intent. Please see responses to prior comments related to sampling density and land use. As noted previously, any parcel not owned by AK Steel will be assessed assuming unrestricted land use (e.g., residential).

54. Page 31, 4.2, third paragraph, first sentence. Delete the phrases "when a depth of 12' feet bgs has been reached, or" and "or groundwater is encountered" from this sentence. See comments 2 and 43 above.
Response: This text has been revised accordingly; see responses to other similar comments.

55. Page 31, 4.2, third paragraph, last sentence. Delete this sentence. See comment 2 above.
Response: This text has been revised to delete the portion of the sentence of concern; see responses to other similar comments.

56. Page 31, 4.2, fifth paragraph. Add detection of PCBs below the water table to as a consideration in determining if groundwater sampling is appropriate.
Response: This text has been revised to include additional considerations; please also see response to comment 49.

57. Page 31 carrying over to page 32, 4.3. Delete the phrase "(SWMU 23 only)" from this sentence. See comment 42 above.
Response: This text has been revised accordingly; please also refer to response to comment 42.

58. Page 34, 5.0, first paragraph, last sentence. As discussed above, this data should have been evaluated for usability in the CCR. Provide the evaluation in the revised work plan.
Response: This text has been revised to clarify what data is considered usable; please also see response to comment number 13.

59. Page 34, 5.0, last paragraph, last sentence. This sentence states "While multiple parcels have been in industrial or commercial use for many years, AK Steel will investigate each parcel to present both an unrestricted and an industrial/commercial future use scenario for RFI purposes." Please explain the submission of an industrial/commercial future use scenario for properties that AK Steel does not control. Does AK Steel plan on submitting an environmental covenant for such properties at the time the risk assessments are submitted?
Response: Text changes have been made to clarify intent; please also see response to comment 46.

60. Page 35, 5.2.1, third paragraph. See comment 59 above.
Response: Text changes have been made to clarify intent; please also see response to comments 46 and 59.

61. Figure 4. In the northeast portion of the figure, the ground is disturbed. Historical documentation indicates that the lagoons were dredged over time. Could this be the contents of the lagoons?
Response: The dried solids were sent to the onsite landfill(s) at Middletown Works for disposal; please also see response to comment 10.

62. Figure 17: the proposal appears to be to locate contingency samples through a parking area. If that area was not paved in the past, that is acceptable. If it was paved, may be more productive to sample the side lot areas.
Response: In preparation of the RFI Work Plan, KEMRON and AK Steel reviewed the historic aerial photographs and verified the parking lot on this AA was not present in the 1960s. Assuming the property owner will allow installation of the borings as illustrated, the contingency borings will be implemented as step-out samples if the primary sample locations exceed applicable screening levels. Should the parking lot be a limiting factor beyond AK Steel's control, additional locations will be proposed as necessary for approval by USEPA et al. prior to their installation, and to ensure the concentrations of PCBs are delineated to residential screening levels and/or ecological screening levels.

63. Figure 18, AA07: This AA is the FOE facility and picnic area. The sampling scheme should be expanded to ensure the complete back lot area has sample coverage. See the attached suggested revision to Figure 18.
Response: Figure 18 has been revised to include the expanded area. Please note that the revised figure proposed sampling locations are adjusted slightly from those noted by the commentor to avoid FOE recreational features.

64. Table 2 is confusing, and it is requested that you clarify for the reader. For example, what does BDL<5 mean, as compared to BDL.
Response: The "<5" was inserted to indicate that the detection limit was "5", based on review of historic data. The footnote explaining this was inadvertently omitted from the printed area; the table has been corrected to provide the footnote explanation.

65. Table 4, Page 2, Step 4: Define Boundaries of RFI. Revise to read "The boundaries of the first phase of investigation are the parcel boundaries as shown on RFI figures."
Response: The text has been revised in accordance with the comment.

66. Table 4, Page 1 and 2, Step 7: Develop Plan of Obtaining Data, Number 7. As with groundwater, revise to include a plan responding to elevated COCs in soils.
Response: The DQO table has been revised in accordance with the comment.

67. Attachment B QAPP: (a) after revisions, submit the signature page; (b) Table 5.2a in the QAPP appears to have different reporting limits for PCB Aroclors than Table 1 in the corresponding Test America laboratory SOP DT 06-28. Please clarify. (c) Table 5.3a in the QAPP has higher reporting limits than some RSLs so would not be appropriate; it also appears that the corresponding reporting limits Table 14.2 in the Test America SOP are lower for arsenic, cadmium, chromium, lead, selenium and silver. Revise Table 5.3a in the QAPP as appropriate (assuming the detection levels in the laboratory SOP are accurate). (d)

Section 10 of the QAPP states that performance audits will be conducted at the start of the project. If those have been completed, a copy is requested. Also submit any recent laboratory audits and/or certifications for the analyses performed under the QAPP in the past 3 years. (e) for the laboratory SOPs, clarify what footnotes (2), (3) and (4) in Table 7 of SOP DT 06-28.2 mean, since they are not included. Also, there appears to be no retention times reported for Aroclors 1248 and 1254.

Response: There is not response at this time due to pending corrections of the laboratory information from the contract laboratory. These necessary corrections were identified by KEMRON on 4/26/13.

RESPONSES TO USEPA ET AL. CONDITIONAL APPROVAL OF 8 AA PORTION OF SWMU 23 AND 8 AA RFI WORK PLAN:
USEPA et al. comments dated May 31, 2013

AK Steel and KEMRON Environmental Services, Inc. received USEPA et al. conditional approval of the 8 AA portion of the April 2013 SWMU 23 and 8 AA RFI Work Plan in a letter dated May 31, 2013. The letter was received via email on June 03, 2013, with paper copy receipt on June 10, 2013. The following provides our responses to the conditions; relevant replacement pages are attached for your concurrence.

1. *Sample preparation of soil samples shall be by the EPA method previously approved for the project, pressurized fluid extraction (EPA Method 3545A). EPA Method 3550C (sonication) is not an approved method for this project and shall not be used. An alternate EPA method which can be used is Sohxlet extraction (EPA Methods 3540C or 3541).*

Response: It is anticipated that Soxhlet by method 3541 will be used. Sonication will not be used. Please note that auto-Soxhlet is available at the Test America Nashville laboratory and may be used to assist in providing expedited turnaround time on sample analytical results. The relevant SOP and information regarding the Nashville laboratory is attached for incorporation into the project QAPP. As noted in the comment, sonication will not be used and the revised QAPP pages are attached. The sonication SOP has been deleted from the QAPP.

2. *SOP #12-SBC-AKMW was referenced in Section 4.0 on page 28, but the SOP was not included in Appendix A. Submit this SOP.*

Response: The referenced SOP was included in the QAPP, which was submitted after the revised work plan. The QAPP was submitted via email on May 09, 2013 with paper and CD copies also distributed. Please refer to the field SOPs in the QAPP for the referenced item. Also, please note that SOPs with the identifier "AKMW" include a citation and cross-reference to the SOPs originally prepared by ENVIRON, which were reviewed and approved by USEPA et al.

3. *SOP #12-MWI-AKMW was referenced on page 29 for installing monitoring wells as needed at SWMW 23 (or if needed for the AAs), as is SOP #12-PID-AKMW (field head space screening), but the SOPs do not appear to have been provided. Submit copies of these SOPs.*

EPA notes that previously-approved SOPs for these tasks (sampling, MW installation, field head-space screening) were approved as part of the SWMU 38/39 RFI work plan, so it is not clear why new SOPs would be used rather than using those SOPs, for consistency. It would be acceptable to use the previously approved SOPs and reference those in lieu of new SOPs. If new SOPs are used, identify areas in the SOPs where changes from the SOPs previously approved, were made.

Response: Please see response to comment 2. Additionally, the SOPs provided are based on standard industry protocols, and have been approved by Ohio EPA and USEPA Region 5 for use in other projects. Our review of the ENVIRON SOPs for SWMUs 38 and 39 RFI/CMS did not

identify any significant differences in technical approach or methodologies from those identified in the KEMRON SOPs.

4. *DQO Step 2 states that AK will "Identify all media and areas where there exists an excess lifetime cancer risk within the range of lE-06 to lE-04 and/or a hazard index equal to or greater than 1." Please note that Attachment 2 to the Order at pages 37-38 specifies how the human health risk assessment shall be conducted and at Part 6, B.l.(ii) states: "Identify all areas and/or media where risks exceed an excess lifetime cancer risk of lE-6 and all areas and/or media with a hazard index of 1 or greater;" Later in Section B.l it states that "At AK Steel's option, the human health risk assessment may also identify all areas and/or media where excess lifetime cancer risks exceed lE-4 and lE-5." OEPA uses lE-5 cancer risk for all programs. Revise the DQO to reflect the above.*

Response: Please find a replacement page attached. The DQO Step 2 has been revised to reference the Consent Decree language and standards. We believe that Section V, paragraph 16 is consistent with the USEPA et al. condition regarding the DQO language. Therefore, the referenced DQO Step 2 has been modified to add the language in bold font below:

"Identify all media and areas where there exists an excess lifetime cancer risks within the range of 1E-06, 1E-04 and 1E-04 and /or a hazard index equal to or greater than 1 **in accordance with standards specified in the March 2006 Consent Decree."**

To clarify, we understand the OEPA cancer risk document referenced in the condition to be the August 21, 2009 Human Health Cumulative Carcinogenic Risk and Non-carcinogenic Hazard Goals for the DERR Remedial response Program guidance document. If this is not the document being referenced, please provide the citation to the appropriate OEPA guidance document.

5. *Revise Figure 9, the Preliminary Site Conceptual Model for Additional Areas, to reflect that discharge of contaminants in dredge spoils may also be possible through a surface water runoff/erosion pathway in addition to percolation.*

Response: Please find attached a revised Figure 9.

USEPA and OEPA SWMU 23 RFI WP Discussion Points, June 17, 2013

AK Steel and KEMRON received a conditional approval of the Solid Waste Management Unit (SWMU) 23 and 8 Additional Areas of Potential Concern AA) RCRA Facility Investigation Work Plan (RFI WP) from USEPA et al. via email on February 28, 2013. Following discussion of the conditions via teleconference on March 19, 2013, USEPA et al. issued a modification to the comments (also dated March 19, 2013). KEMRON submitted revisions to the RFI WP on April 26, 2013. A conditional final approval for the 8 AA portion of the RFI WP was received on June 3, 2013. This letter included a request for a teleconference or meeting regarding the SWMU 23 portion of the RFI WP; a meeting with OEPA, with USEPA participating via telephone, was conducted on June 17, 2013. USEPA submitted a list of items for discussion related to the SWMU 23 portion of the RFI WP (Revision 1, dated 04/26/13) via email on June 17. The following summary includes the original June 17, 2013 USEPA email discussion points, OEPA verbal comments, and the responses to each of the comments. Change pages have been prepared and are attached to reflect the agreements reached in the June 17, 2013 meeting.

USEPA SWMU 23 RFI WP Comments, June 17, 2013 via email:

Comment 1: *Section 2.2.1.1 SWMU 23*

1a: *Last paragraph on page 11 covers wells GM-1S, GM-2S and GM-1D, but not well GM-3S. Info for well GM-3S should be added here. Screened depths are provided for wells GM-1S, GM-2S, but not GM-1D or well GM-3S. Add screen depth info for those wells here as well.*

Response: Screen depth information has been added.

1b: *Paragraph 2 on page 12: nothing is stated regarding well GM-3S. Add that info.*

Response: Paragraph references historical groundwater monitoring data. Available historic records were reviewed, including those available at Middletown Works and in Appendix E of the Current Conditions Report (CCR), 2012. No analytical data for GM-3S has been located. Available records indicate that monitoring well GM-3S has historically been used only for groundwater elevation measurements.

Comment 2: *GW quality investigation 1989-1990 discussed on page 13 identified detections of acetone, carbon disulfide, methylene chloride, and trichloroethylene in varying concentrations at wells GM-1D, GM-2S and GM-1S. Detections of methylene chloride and trichloroethylene exceeded the MCL. However, Table 2 does not have data for acetone, carbon disulfide, or methylene chloride, and the trichloroethylene detection level is at the MCL. Revise Table 2 to present all data, and include all data for well GM-3S as well. The data contained in Appendix E-5 of the CCR appears to indicate that well GM-3S was not sampled during this period.*

Response: Data for 1989-1990 from Appendix E-4 of the CCR discussed on page 13 and also data from Sitewide Monitoring April 2012 have been added to Table 2. No analytical data for well GM-3S was presented for any of these sampling events in Appendix E-4 or E-5. Available records indicate GM-3S has historically been used only for groundwater elevation measurements.

Comment 3: *Section 2.2.4.1 Nature and Extent Related to SWMU 23*

The first paragraph of this section states in part that "No monitoring wells currently exist within the SWMU 23 area to monitor the shallow water table conditions." This appears to be inconsistent with the boring logs for wells GM-1S, 2S and 3S which appear to be screened (40-50' bgs) in the shallow zone of the first water-bearing unit. Well GM-1D is screened deeper, apparently in the same water-bearing zone. The boring log for well GM-1S does not clearly indicate the depth of water during construction, but the soil may have been saturated at a depth between 30-35' bgs. The static depth to water for well GM-1D

was identified at 42.18 ft below the MP, or about 42' bgs. This well is screened at 99.7 – 109.7 ft, and the log does not appear to identify any confining layers. The total depth to a confining layer is unknown. The boring log for well GM-2S identifies the depth to water table at 38' bgs, and the well is screened at 39.7-50' bgs. The boring log for well GM-3S identifies the depth to groundwater at 34' bgs, which is consistent with the depth during development at 37.7 ft below the MP (35.2 ft bgs). The well is screened at 36-46 ft bgs, just below the observed groundwater level. More recent data (April 2011 GW monitoring) appears to show the water table has risen in the vicinity of these wells to 24.4' (well 3S) – 27.3' (well 1D) bgs.

Response: The Work Plan proposes for installation of three (3) shallow monitoring wells to intercept the risen shallow water table. During the June 17, 2013 meeting, USEPA and OEPA both indicated that well screens in the newly installed monitoring wells should be installed such that they are submerged at least 9 feet (please also see response to comment 6b). The text has been revised to indicate that well screen intervals will be set such that the screen is fully submerged based on observations during well installation.

Comment 4: *Section 2.2.5 Data Gaps, SWMU 23*

4a: *The first bullet, add "adjacent to, below" before "within".*

Response: Text has been revised as requested.

4b: *Bullet 2: if drilling through the cap to acquire the environmental samples, it is recommended that the soil samples of the cap be collected and saved in phase 1 and analyzed if needed.*

Response: Due to hold time and storage limitations in the established analytical method, this will not be feasible. If the data are determined to be necessary, additional samples will be collected and analysis will be conducted per the applicable method.

4c: *Bullet 3 identifies the lack of shallow GW data at the current depths as a data gap. An additional data gap may be the depth of GW in the first zone since it appears a confining layer has not been reached.*

Response: Based on discussions during the June 17 meeting it was acknowledged this could be a data gap however it was agreed that the need to identify a confining layer would be evaluated after results from the current plan are received and evaluated.

Comment 5: *Section 4.0 Sampling Plan*

4th paragraph: Why are individual boring locations manually cleared to a depth of 4' prior to start of sampling using mechanized drilling methods?

Response: KEMRON's Corporate Health and Safety Plan requires manual clearing to this depth due to possible contact with unmarked underground utilities.

Comment 6: *Section 4.1 SWMU 23 sampling*

6a: *First paragraph: why will soil sampling terminate upon encountering the water table interface?*

Response: Continuous soil sampling to the depth of the water table interface, estimated at 20 ft or more, is considered sufficient in determining whether residual constituents from historical surface disposal is evident within the subsurface soils. As indicated in the Work Plan, soil sampling will terminate upon encountering the water table interface. During the June 17 meeting KEMRON noted that this was consistent with previous discussions from the March 19, 2013 teleconference regarding the original RFI

Work Plan. USEPA and OEPA indicated that no change or further discussion was necessary in response to this comment.

6b: First paragraph, page 29: Once water depth is encountered, at least 9 ft of screen should be below the water table. The observed most recent water table was at 26.1 – 27.3' bgs, not 20' bgs. The water table at the time the existing wells GM-1S, 2S and 1D were developed was deeper, at 38'-39' bgs.

Response: Water table monitoring wells will have their well screens installed to intercept the water table interface (depth measured during drilling) a minimum of 9 ft below the water table. The text on page 29 has been revised accordingly.

Comment 7: *Existing MW Inspection, page 29*

The inspection of the existing wells, and redevelopment, if needed, should occur prior to the scheduled installation of the new wells planned under this RFI.

Response: We agree that it is prudent to complete inspections and redevelopment of existing wells prior to new well installation. Text has been inserted on page 29 to indicate this will be done. KEMRON and AK Steel will verify that the existing well conditions prior to mobilizing to conduct the RFI at SWMU 23, and will notify USEPA et al. if any of the wells requires repair or replacement for the RFI to be implemented.

Comment 7: *MW Installation and Sampling, pp. 29-30*

Please ensure the target RCRA metals include Ni and Zn.

Response: Per our discussions on June 17, additional target metals Al and Zn will be added. AK Steel noted that nickel would not be a constituent of potential concern (COPC) for SWMU 23, and USEPA and OEPA agreed, based on discussion during the meeting, that nickel does not need to be added to the analytical list. Text and table revisions have been made in the workplan to include Al and Zn.

Comment 8: *Soil Boring Completions*

Paragraph 2 states continuous soil sampling will be advanced until the water table is reached. What is the basis for that depth of sampling?

Response: Continuous soil sampling to the depth of the water table interface, estimated at 20 ft or more, is considered sufficient in determining whether residual constituents from historical surface pond discharges have impacted subsurface soils. Please also see summary and response to comment 6a, above.

Comment 9: *Page 31, first full paragraph: clarify that after VOC sampling and core logging, that the full 2' core of the sample will be placed into a stainless steel bowl, homogenized, then sampled for RCRA metals, including Ni and Zn.*

Response: The VOC sampling methodology clarification has been added and metals Al and Zn have been added (not Nickel) based on discussions in our June 17 meeting; please refer to response to comment 7, above. Text revisions have been made to the work plan. Additionally, based on discussion in the June 17 meeting, the text has been revised to clarify that VOC sampling will occur in accordance with USEPA Method 5035, using EnCore® or TerraCore® (or equivalent) sampling for VOCs, while analytical method 8260 will be used.

Comment 10: *Page 31, 5th full paragraph: add at the end "and/or other evidence of contamination e.g., staining."*

Response: The requested edit has been made.

Comment 11: *Page 31, last paragraph: add "and periodically hydrated during and after filling." Is there an SOP for backfilling boreholes?*

Response: The text has been edited in accordance with discussion in the June 17 meeting. Chapter 9 the OEPA Technical Guidance Manual for Groundwater Investigations will be followed for backfilling. References to the guidance manual and rule have been added.

Comment 12: *Section 4.5 Analytical Testing*

The location of the Test America Lab is not specified, but assumed to be N. Canton, OH. It is noted that the only current lab certification submitted expires on June 30, 2013.

Response: Test America recently underwent several recertification audits. The laboratory indicated there is no reason to believe they will not be recertified. KEMRON has requested that a copy of the new certifications be provided upon receipt; this will be forwarded to USEPA et al. following receipt. During the June 17 meeting, KEMRON also clarified in response to questions and discussion from OEPA that the Florida certification is relevant, due to the State of Florida being the NELAC certifying body.

Comment 13: *Figure 12*

(a) Add the location of well GM-3S to this Figure and confirm it will be sampled as part of the RFI.
(b) Move the location of SB 15 to coincide with the arrow identifying the general groundwater flow direction (for intermediate aquifer wells)
(c) Add a deep well in the same general location of SB 16, to monitor the same depth as well GM-1D

Response:

a) and b) Figure 12 has been updated to address (a) and (b). Monitoring well is scheduled to be sampled as part of the RFI, as noted in Section 4.1.
c) During our discussions on June 17, it was agreed that the need for a deep well in the location of SB16 would be evaluated after data from the planned sampling has been evaluated. USEPA et al. agreed that geologic and analytical results from the RFI would be reviewed following completion of the RFI phase described in the current Work Plan scope. If additional deeper aquifer information was necessary, it would be determined after review of the results of this RFI phase, and an additional phase would be planned as necessary based on the SWMU 23 data, and in accordance with the Data Quality Objectives for the RFI.

Summary of OEPA verbal discussion points and resolution:

1. OEPA notes that new monitoring wells should be named to be representative of the RFI, rather than in a manner that ties them to the historic site-wide groundwater monitoring network. Significant discussion occurred regarding the presence or absence of an aquitard in the vicinity of SWMU 23 that would distinguish a shallow vs. deep aquifer. After discussion, all agreed that the existing well logs indicate that there is not a clay layer present in the vicinity of SWMU 23 that would distinguish a shallow versus deep aquifer. It was also agreed that new wells would have a nomenclature that relates the wells to the RFI rather than a "shallow" or "deep" nomenclature as has been used in the past.

2. As noted in response to USEPA Comment 9, OEPA inquired regarding language in the RFI WP for VOC sampling and analysis. The text has been revised to clarify that Method 5035 references

apply only to VOC soil sampling method, while laboratory analysis will be conducted using USEPA Method 8260.

3. OEPA inquired about the method that will be applied to determine soil leaching values as referenced in the text, and whether the values would be default or calculated values. KEMRON responded that it was anticipated the data acquired from the RFI would be reviewed with USEPA et al., and the appropriate values would be selected in conjunction with USEPA et al. It has not yet been determined whether the default or calculated values would be applied. OEPA indicated this was acceptable and that RFI WP revisions were not necessary at this time.
4. OEPA inquired what vapor intrusion guidance would be applied to determine whether soil and/or groundwater VOC concentrations from the SWMU 23 RFI indicate a potential vapor intrusion pathway and/or need for further vapor intrusion evaluation. After discussion, it was determined that the most current, effective USEPA guidance would be applied for screening purposes.

Responses to USEPA and OEPA SWMU 23 RFI WP Discussion Points dated July 17, 2013

AK Steel and KEMRON received a conditional approval of the Solid Waste Management Unit (SWMU) 23 and 8 Additional Areas of Potential Concern AA) RCRA Facility Investigation Work Plan (RFI WP) from USEPA et al. via email on February 28, 2013. Following discussion of the conditions via teleconference on March 19, 2013, USEPA et al. issued a modification to the comments (also dated March 19, 2013). KEMRON submitted revisions to the RFI WP on April 26, 2013. A conditional final approval for the 8 AA portion of the RFI WP was received on June 3, 2013. This letter included a request for a teleconference or meeting regarding the SWMU 23 portion of the RFI WP; a meeting with OEPA, with USEPA participating via telephone, was conducted on June 17, 2013. USEPA submitted a list of items for discussion related to the SWMU 23 portion of the RFI WP (Revision 1, dated 04/26/13) via email on June 17. KEMRON submitted revisions to the RFI WP on July 2, 2013 to address the June 17, 2013 comments.

At a meeting with USEPA and OEPA on July 17, 2013 additional comments were received on the July 2, 2013 Response to Comments. The following is a summary of those comments and responses.

USEPA et al. SWMU 23 RFI WP Discussion Points, provided on July 17, 2013:

Comment 1: *Response to Comment 1b. Include Response to Comment 1b in text of the document to clarify why there is no analytical data for well GM-3S.*

Response: The following text has been added on page 12. "Records reviewed to date indicate that monitoring well GM-3S has historically been used only for groundwater elevation measurements." A replacement page is attached.

Comment 2: *Response to Comment 3. During the meeting on June 17, 2013, it was determined that the ground water zones for the RFI did not have to be defined in the same way as the site wide groundwater monitoring. For the purposes of this RFI, it may be best to state that even though the wells GM-1S and 1D are labeled as shallow and deep wells, they apparently monitor the same groundwater zone.*

Response: After verbal discussions, it was agreed that the wells should not be renamed, and that this would be clarified in the RFI Report rather than additional edits being incorporated into the RFI Work Plan. KEMRON and AK Steel agreed that the current boring and monitoring well logs near SWMU 23 indicates there is not an identifiable clay layer. Therefore, currently existing geologic information indicates the monitoring network includes various screened intervals are in the same geologic unit. The RFI Report will discuss the subsurface conditions. No further edits have been made in the text and replacement pages have not been prepared based on the agreements achieved in the July 17, 2013 meeting.

Comment 3: *Response to Comment 5. Incorporate the Response, "KEMRON's Corporate Health and Safety Plan requires manual clearing to this depth due to possible contact with unmarked underground utilities." into the text of the document to clarify the reason for manually clearing boring locations to a depth of 4' prior to start of sampling using mechanized drilling methods.*

Response: The text has been revised to state "In accordance with KEMRON's Corporate Health and Safety Plan, individual boring locations are manually cleared to a depth of 4 feet for purposes of avoiding the presence of potential unmarked utilities." A replacement page is attached.

Comment 4: *Response to Comment 6. Incorporate the response, "Continuous soil sampling to the depth of the water table interface, estimated at 20 ft. or more, is considered sufficient in determining whether residual constituents from historical surface disposal is evident within the subsurface soils." into the text*

of the document to clarify the reason for terminating soil sampling upon encountering the water table interface.

Response: The text has been revised as requested and a replacement page is attached.

Comment 5: *Response to comment 8. Incorporate the Response, "Continuous soil sampling to the depth of the water table interface, estimated at 20 ft. or more, is considered sufficient in determining whether residual constituents from historical surface pond discharges have impacted subsurface soils."*

Response: The text has been revised as requested and a replacement page is attached.

Comment 6: *Summary of OEPA verbal discussion points and resolution #1. Refer to comment on the Response to Comment 3.*

Response: As stated above, USEPA and OEPA agreed that this will be addressed in the RFI Report and no further edits are necessary to the RFI Work Plan.

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

July 23, 2013

REPLY TO THE ATTENTION OF:

Via Certified Mail 7009 1680 0000 7663 7619
Return Receipt Requested

LU-9J

Pat Gallo
Environmental Affairs Manager
AK Steel Corporation
1801 Crawford Street
Middletown, OH 45043

Re: Consent Decree Case Number C-1-00530
EPA Approval of RFI Work Plan for SWMU 23 and Additional Areas, Revision 1, as modified, Pursuant to Paragraph 42 of the Consent Decree

Dear Mr. Gallo:

This letter is in regards to the SWMU 23 portion of the RCRA Facility Investigation Work Plan for SWMU 23 and Additional Areas AA02-AA09, Revision 1, prepared for AK Steel by Kemron Environmental Services, Inc. dated April 2013. The revised Work Plan was submitted to EPA by Kemron's letter dated April 26, 2013, and has since been modified to address EPA and OEPA's verbal comments provided on June 17, 2013, with your submission dated July 2, 2013, as further modified on July 10 and July 22, 2013. EPA has consulted with Ohio EPA (OEPA) and the Sierra Club regarding these documents.

EPA hereby approves your modified submission. Within 10 days, please submit a pdf of the complete revised work plan reflecting all changes made since the April 26, 2013, submission of Revision 1, including the modifications dated July 2, 10 and 22, 2013. You are hereby authorized to proceed with the approved work plan consistent with the schedule therein.

In the interim, if you have any questions, please contact me at 312-886-6760.

Sincerely,

Michael J. Mikulka
Senior Environmental Engineer
Land & Chemicals Division

cc: Mark Allen, OEPA SW District Office
Marilyn Wall, Sierra Club
Mary Lou Rochotte, Kemron
James Kemp, AK Steel, Middletown Works

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Chief, Environmental Enforcement Section, Ohio AG
Robert Guenther, Associate Regional Counsel, EPA Region 5

RCRA Facility Investigation Work Plan
SWMU 23 and Additional Areas AA02-AA09
AK Steel Corporation
Middletown, Ohio

REVISION 3

Prepared for:

AK Steel Corporation
9227 Centre Pointe Drive
West Chester, OH 45069

Prepared by:

KEMRON Environmental Services, Inc.
2343-A State Route 821
Marietta, OH 45750

July 2013

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ACRONYMS

Acronym	Definition
AA	Additional Areas
AK Steel	AK Steel Corporation
AOCs	Areas of Concern
CCR	Current Conditions Report
CMS	Corrective Measure Studies
COCs	Contaminants of Concern
COPCs	Constituents of potential concern in the subsurface
COPECs	Contaminants of Potential Ecological Concern
DI	Dionized Water
EPA	Environmental Protection Agency
ESL	Ecological Screening Level
GPS	Global Positioning System
GW	Groundwater
HWMUs	Hazardous Waste Management Units
IDW	Investigation Derived Waste
KEMRON	KEMRON Environmental Services, Inc.
NRDC	Natural Resources Defense Council
ODNR	Ohio Department of Natural Resources
OEPA	Ohio Environmental Protection Agency
PCB	Polycholorinated Biphenyls
PID	Photoionization Detector
PIP	Public Involvement Plan
PMP	Project Management Plan
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QHEI	Qualitative Habitat Evaluation Index
RAAR	Risk Assessment Assumptions Report
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
RFI/CMS	RCRA Facility Investigations and Corrective Measure Studies
ROIs	Receptors of Interest
RSL	Regional Screening Level
SB	Soil Boring
SCM	Site Conceptual Model
SERA	Screening Ecological Risk Assessment
SOP	Standard Operating Procedure
SOPs	Standard Operating Procedures
SSL	Soil Screening Level
SWMU	Solid Waste Management Unit
TCL	Target Compound List
TOC	Top of Casing
USEPA	United States Environmental Protection Agency
USFWS	United States Fish And Wildlife Service
VOC	volatile organic compound
WP	Work Plan

1.0 Introduction

On behalf of AK Steel Corporation (AK Steel), KEMRON Environmental Services, Inc. (KEMRON) has prepared this Resource Conservation and Recovery Act (RCRA) Facility Investigation Work Plan (RFI) for investigation of Additional Areas (AAs) of Potential Concern AA02 through AA09, and the Former Coil Paint Area Wastewater Treatment Plant Lagoons, which is identified as Solid Waste Management Unit (SWMU) 23, associated with the AK Steel Middletown Works, Middletown, OH facility.

The RFI WP has been prepared in accordance with the requirements of a Consent Decree signed by United States et al. (i.e., United States of America, State of Ohio, Sierra Club, and Natural Resources Defense Council [NRDC]) and AK Steel and entered on May, 15, 2006 (Civil Action Number C-1-00530). As part of the Consent Decree, AK Steel is required to conduct RCRA Facility Investigations and Corrective Measure Studies (RFI/CMS) at the Hazardous Waste Management Units (HWMUs), SWMUs, Areas of Concern (AOCs), spill areas, and other suspected source areas listed in Exhibit A to Attachment 2 of the Consent Decree. The work being performed under the Consent Decree is subject to oversight and approval by the United States Environmental Protection Agency (USEPA), the Ohio Environmental Protection Agency (OEPA), and others. The RFI described in this work plan will fully evaluate the nature and extent of contamination originating from AAs of Potential Concern AA02 through AA09, and SWMU 23, and will assess current and potential future impacts to human health and the environment at these specific locations.

The Consent Decree identifies a preliminary list of HWMUs, Solid Waste Management Units (SWMUs), AOCs, Spill areas, and AAs of Potential Concern for evaluation under the Consent Decree. Subsequent to the identification of these areas in the Consent Decree, a Project Management Plan (PMP) and Current Conditions Report (CCR) have been prepared by others on behalf of AK Steel. Both of these documents have been approved by USEPA and OEPA. AK Steel is required to develop an RFI Work Plan to define the nature and extent of contamination at SWMU 23 and other AAs of Potential Concern, identified as AA02 through AA09 associated with the AK Steel Middletown Works, Middletown, OH. One other Additional Area, AA01, was remediated under the Dicks Creek Interim Measures and therefore is not included in this work plan. This RFI Work Plan is being submitted as required, within 120 days after regulatory approval of the CCR and PMP. The CCR Version 5.0 was issued on May 11, 2012, and the PMP Version 4.0 was issued on August 15, 2012. The August 15, 2012 PMP (Rev 4) was submitted to address USEPA's comments in the Agency's conditional approval dated July 20, 2012.

The PMP Version 4.0 (ARCADIS, 2012a) discusses that SWMU 23 and AAs 02-09 have been designated as high priority for investigation. The high priority of these parcels is due to the fact that the release potential into the environment has not been assessed, the potential for PCB containing material having been managed at the AAs, and that SWMU 23 and the AAs are located off-site.

The purpose of this RFI WP is to describe and outline the process that will be followed to delineate the nature and extent of contamination at SWMU 23 and AAs 02-09, and to prepare the RFI Report.

2.0 Current Conditions

2.1 Facility Background

The CCR (Arcadis, 2012b) as approved documents current site conditions for AK Steel Middletown Works and surrounding properties. Information from this document has been excerpted and is presented in this section.

The AK Steel Middletown Works facility is located in southwestern Ohio, in Butler County, in the City of Middletown (Figure 1). Middletown Works manufactures steels, which includes carbon steel melting, casting, hot and cold rolling, and finishing operations. The entire Middletown Works facility covers an area of approximately 2,600 acres with each area involved in various aspects of iron and steel manufacturing and processing.

Several environmental investigations have taken place at various locations at the Middletown Works over the years. A CCR (ARCADIS, 2012b) has been developed which summarizes the history of environmental investigation programs at the Middletown Works. This document includes a summary review of historical aerial photographs, Sanborn fire insurance maps, and other historical data resources used for determining the industrial developmental history of AK Steel in Middletown. The CCR, PMP, and other AK Steel environmental data reports and information from personal interviews and historic information regarding the placement of materials from historic dredging of Dicks Creek all were used in development of this RFI Work Plan.

SWMU 23 is a closed, unlined set of three (3) wastewater lagoons located on a portion of the Middletown Works facility known as the Coil Paint area. The Coil Paint area is situated toward the west side of Middletown Works (Figure 2).

Land use around SWMU 23 (Figures 2 and 3) is industrial immediately adjacent to SWMU 23. Undeveloped land lies immediately west and south, public roadways and railway lines to the north, south and west, and residential use further north and south of SWMU 23.

AAs of Potential Concern AA02 through AA09 (Figure 2) consist of off-site properties (not owned by AK Steel) that are a mixture of single family residential properties and small to medium-sized commercial/industrial properties located adjacent to Dicks Creek toward the southwest side of the Middletown Works. Table 1 below provides a brief description of the individual AA parcels.

Table 1: Description of Additional Areas of Potential Concern

LOCATION	DESCRIPTION
ADDITIONAL AREAS OF POTENTIAL CONTAMINATION	
AA02	Former Glenn Cartage property (NS of DC, East of Yankee Rd). Currently designated as Garland II LLC on RFI Figures
AA03	Sturgell Property (NS of DC, W of RR bridge, E of Yankee Rd)
AA04	Back half of properties between G. Cartage and Sturgell Properties (NS of DC). Currently

LOCATION	DESCRIPTION
ADDITIONAL AREAS OF POTENTIAL CONTAMINATION	
	designated as Earls and Perkins parcels on RFI figures.
AA05	Old Armco Lot, NS DC, North of big meander
AA06	Former Burridge Machine Shop Property, NS DC. Currently designated as Schwans Consumer Brands parcel on RFI figures.
AA07	Former Cecil Osburn Lot (located between Station 12-18, NS of DC). Currently designated as Fraternal Order of Eagles on RFI figures.
AA08	Pipeline Fill, Stations 32-36, along Oxford S.R., N of Outfall 002. Currently designated as Johnson parcel on RFI figures.
AA09	Former Orman's Welding Property. Currently designated as Yankee Properties on RFI figures.

NS=North Side DC=Dicks Creek

Current zoning of each of the AAs is listed below to document current land use:
AA02: Commercial
AA03: Residential
AA04: Residential
AA05: Industrial
AA06: Commercial
AA07: Commercial
AA08: Commercial
AA09: Industrial

2.1.1 Ownership and Operational History

AK Steel Middletown Works began operation in 1901 as the American Rolling Mill Company. The company produced low carbon, high strength steel using a hot and cold flat rolling process. The company changed its name to ARMCO in 1948 and then to the Armco Steel Corporation. In 1978, the company changed its name to Armco, Inc. According to ARCADIS (2012b), in 1989, the Middletown Works became a partnership between Armco, Inc. and Kawasaki Steel Corporation. The partnership was named Armco Steel Company, L.P. In 1994, the L.P. was spun off and became AK Steel Corporation. In 1999, Armco, Inc. merged with AK Steel Corporation. The Middletown Works facility currently consists of the: (1) North Plant Area; (2) Melt Plant Area; (3) South Plant Area; (4) Former Coil Paint Area wastewater lagoons (SWMU 23); and (5) slag processing area.

Middletown Works currently is a fully integrated plant producing carbon steels, hot rolled stainless steels, hot dipped aluminized steels, hot dipped galvanized steels, and coated steels. In addition, the facility produces finished and coated steels. Hot rolled steels are used where heavy thickness, strength levels, and formability are required. The steels produced at the plant are used in a variety of applications in automotive, appliance markets, and construction markets. These include frame components, brackets, brake components, wheels, clutch plates, tubing, and compressor shells. Construction, industrial machinery, agricultural equipment, railroad, and ship building are all areas where steels are used for panels, frames, and component parts. Cold rolled steels provide excellent

press formability, surface finish, and thickness and flatness tolerances. Cold rolled steels can be used to produce parts containing simple bends to parts with extreme deep drawing requirements. Hot dipped aluminized, hot dipped galvanized, and electrogalvanized steels are best suited for applications where surface finish, corrosion resistance, or both are critical. Examples of such applications include automotive, appliance, agricultural equipment, and architectural products (AK Steel Product Data Bulletin, 2000).

SWMU 23:
As detailed in the CCR, review of historic aerial photographs through March 1961 does not show the Coil Paint Plant area as having been developed. The Coil Paint Plant can be seen as developed and active on an April 1976 aerial photograph. Drawings of this portion of the AK Steel facility include Drawing Number 697068, dated July 20, 1976. This drawing illustrates the location of the Wastewater Treatment Plant and three associated lagoons that now comprise SWMU 23. Drawing Number 697069, also dated July 20, 1976, provides additional detail of the Coil Paint Line wastewater treatment system, including two lagoons and a sand bed. The CCR indicates that the lagoons associated with the Coil Paint Plant wastewater treatment system were operated from 1971 to 1978. Final closure of two of the lagoons is documented in recently identified files as occurring in 1986 (see Attachment H).

The lagoons associated with the wastewater treatment plant were closed in the mid-1980s. Subsequently, the building formerly used as the Coil Paint Plant was sold. AK Steel has retained ownership of the parcel containing the closed lagoons.

Additional Areas:
The AAs were evaluated using aerial photographs in the CCR. Aerial photographs from 1938 through 2007 were evaluated and discussed. These properties were undeveloped land in July 1938. AA02, the Former Glenn Cartage Property, had been partially developed in March 1956. No changes were noted on aerial photographs from March 1961 and August 1962. The CCR notes that Dicks Creek channelization is evident in an October 1968 aerial photograph. This 1968 photograph also indicates the following:

- AA03, the Sturgell Property, to be in residential use at this same time, and the southern three-fourths of the parcel appear to have been disturbed;
- The structures that presently exists on AA02 AA03 and AA04 are present in the 1968 aerial photograph;
- AA09, the remainder of Ormans Welding Property appeared to have had heavy construction with a new building and most of the trees removed.

An April 1976 aerial depicts the southern portion of AA02 (Former Glenn Cartage Property) as disturbed or possibly covered with concrete. A new facility is observed south of AA02 and AA09, with one small and one large structure and an associated drive visible. A March 1980 aerial photograph shows increased commercial development, with larger structures visible. Two circular areas of disturbance can be seen on the southern portion of AA02, and a small pond is visible immediately north of these parcels and Oxford State Road.

Continued commercial development south of Oxford State Road is seen in a March 1993 aerial photograph, including a large automobile salvage yard that is still present today. No significant changes are observable in March 1999 and March 2007 aerial photographs.

The AAs are subject to investigation based on the observation of disturbances that may be related to the channelization of Dicks Creek, due to PCBs having been demonstrated to be present in the

creek sediment. In addition to the historic aerial photographs, interviews of individuals involved in stream channelization indicated that sediment may have been placed on these properties.

2.1.2 Past Project and Waste Spills

2.1.2.1 SWMU 23

No records of spills are available for the former Coil Paint Plant. The SWMU 23 lagoons were designed to receive wastewater from Coil Paint production facilities. These wastewaters contained metals. Based on painting that historically was conducted within the Coil Paint plant operations, VOCs also are to be evaluated as contaminants of potential concern (COPCs) at this site as requested by regulatory oversight personnel.

2.1.2.2 Additional Areas

AAs AA02 through AA09 are off-site properties located adjacent to Dicks Creek. Historical evidence from review of aerial photographs and interviews with local residents and workers associated with the late 1960's channelization of Dicks Creek indicates dredged spoils from the creek may have been placed on one or more of these properties. Dicks Creek sediments are documented as being impacted with PCBs, and ongoing Interim Measures (IMs) have been implemented in Reach 1 of Dicks Creek adjacent to the AAs to remove contaminated sediments and floodplain soils. The potential exists for PCB-impacted sediments, removed during historical creek channelization and/or dredging activities, to have been deposited on the AAs resulting in a potential off-site release of PCBs to the environment.

2.1.3 Past Permits and Enforcement Actions

There are no records available regarding environmental or other permits for the former Coil Paint Plant.

AK Steel has not operated any units on the AAs; therefore, no permits or enforcement actions for these sites are known to exist outside of the 2006 Consent Decree.

AK Steel currently holds, and historically has held, many permits. However, all of the currently available information regarding permits relates to the units within the plant. Sections 2.7 and 2.8 of the CCR present information on environmental permits and enforcement actions related to the AK Steel on-site facilities.

2.1.4 Major Habitat Types

Middletown, Ohio lies within the Eastern Corn Belt Plains ecoregion, as identified by Omernik (1987). The Eastern Corn Belt Plains is primarily a rolling till plain with local end moraines. Originally, beech forests were common on Wisconsinan soils (glacial deposits), while beech forests and elm-ash swamp forests dominated the wetter pre- Wisconsinan soils. Today, extensive corn, soybean, and livestock production occurs and has affected stream chemistry and turbidity (U.S. EPA, 2002). The Ohio EPA uses Eco region designations to determine aquatic biological criteria for fish and invertebrate community quality, as well as reference values for metals in sediment.

While no surface water features are present on the AAs or SWMU 23, the major habitat associated with the AAs includes Dicks Creek, and its associated floodplain. A portion of each of

the 8 AAs is located in the Dicks Creek 100 year floodplain and the creek is immediately adjacent to each of the AAs.

Each of these habitats is discussed below. No wetlands are identified outside the Dicks Creek and Great Miami River riparian corridors, and therefore are not discussed further in this RFI WP. A portion of Dicks Creek flows between the AK Steel plant westward toward the Great Miami River. The Dicks Creek watershed extends approximately 12 miles east of the Great Miami River and encompasses an area of approximately 50 square miles. Major tributaries include the North Branch of Dicks Creek (which flows from north to south adjacent to the AK Steel facility before joining the main branch of Dicks Creek at approximate river mile 5.3), Shaker Creek (which enters Dicks Creek at river mile 4.6), and Millers Creek (which flows into Shaker Creek a short distance upstream of its confluence with Dicks Creek). A small tributary, known locally as Monroe Ditch, traverses the southwestern portion of the Slag Processing Area and drains into Dicks Creek.

In the 1960s much of Dicks Creek was channelized as part of the Miami Conservancy District flood control program. The channelization process involved widening, deepening, and straightening the natural channel of Dicks Creek and rerouting the North Branch of Dicks Creek to its current course. As a result of channelization, sediment deposition rapidly filled the excavated trapezoidal channel of Dicks Creek, creating an abbreviated floodplain, through which a narrower stream channel became incised (i.e., the existing low-flow channel). Additional stream dredging is reported to have occurred in the 1980's. A more detailed description of Dicks Creek, Monroe Ditch, and associated streamside (riparian) habitat is provided in the *Interim Measures Remediation Work Plan* (ENVIRON, 2008a). Additionally, the *Focused CCR* (Appendix F) contains a description of habitat conditions in Dicks Creek upstream of Reach 1.

The Great Miami River drainage basin covers 5,385 square miles in southwestern Ohio and Indiana. The main stem of the river is 170 miles in length and drains to the Ohio River. Land use in the Great Miami watershed is primarily row-crop production of corn, soybeans, and wheat. Major urban areas in the watershed include Dayton and Cincinnati, Ohio (Debrewer et al., 2000; Ohio EPA, 1997). Use designations applicable to the Great Miami River in the Middletown area include Warmwater Habitat, Agricultural and Industrial Water Supply, and Primary Contact Recreation.

In Ohio, aquatic habitat quality is typically rated using the Ohio EPA's (Rankin 1989) standardized Qualitative Habitat Evaluation Index (QHEI). The QHEI assesses a variety of stream attributes, including substrate type, siltation/embeddedness, type and amount of instream cover, channel morphology, riparian zone extent and quality, bank erosion, and pool, riffle, and run quality. High quality aquatic habitat is generally characterized by a high diversity of microhabitats, including a variety of velocity-depth combinations and substrate types and an abundance of instream cover (such as snags or overhanging banks). In contrast, modifications such as channelization tend to result in a homogeneous and exposed habitat that is suitable for fewer aquatic species. Aquatic habitat quality was evaluated in the Great Miami River by the Ohio EPA in 1995 and 2000; and by EA Engineering in 1998, 1999, and 2000 (Ohio EPA, 1997 and unpublished data; EA Engineering, 1999, 2000, 2001). Results obtained from these studies indicate good to excellent aquatic habitat quality.

The Great Miami River is not further considered for the purposes of this RFI, due to the distance between the AAs and the river; the primary habitat to be considered regarding the AAs is the adjacent Dicks Creek.

Very little ecologically relevant terrestrial (upland) habitat is evident in the vicinity of the AK Steel Middletown Works facility. Each of the AAs is developed and potential ecologically relevant terrestrial (upland) habitat is limited to mowed lawns and shrub/scrub areas. The southern portion of AAs 2, 3, 4, 5, 6, 7 and 8 and the northern portion of AA 9 lie within the 100 year floodplain of Dicks Creek. The floodplain by definition is a habitat of potential ecological significance. Most of the vicinity near SWMU 23 is occupied by buildings, pavement, or railroad line, or is routinely mowed. However, a 72-acre, largely wooded, upland area is located adjacent to SWMU 23 (Figure 3). This wooded area is partially owned by AK Steel, however a significant portion also is owned by B&O Railroad according to information available via the Butler County Auditor's website. The wooded habitat is bisected by rail lines and a roadway. Most of this woodland lies outside AK Steel property.

2.1.5 General Description of Plants and Animals

2.1.5.1 Current Conditions

This section identifies plant and animal species observed or expected to occur in the habitats identified above. Descriptions of aquatic and terrestrial biological communities are provided below, followed by a discussion of threatened and endangered species.

Aquatic biological communities in Dicks Creek have been extensively studied, as documented in the *CCR* (Arcadis, 2012b). Fish and invertebrate community information for Dicks Creek is not extensively detailed here, because Dicks Creek is subject to interim measures rather than an RFI investigation. In the non-channelized portion of Dicks Creek, some areas contain small patches of curly pondweed *(Potomogeton crispus)* and water weed *(Elodea* spp.) in shallows directly downstream of riffles. In the channelized area of Dicks Creek, the same species are present at greater densities, due to the 100% open canopy. Aquatic vegetation serves as both food and instream cover for fish and aquatic invertebrates and is also consumed by herbivorous wildlife (e.g., waterfowl).

Vegetation along Dicks Creek was characterized in 2004 with particular focus on Reach 2 (ARCADIS, 2012b), as described below. Vegetation in the upland, wooded area near SWMU 23 would not be expected to include the characteristic riparian species identified from Reach 2 of Dicks Creek. However, Amur honeysuckle *(Lonicera maackil)* and poison ivy *(Rhus radicans),* both noted in the Reach 2 area, have also been observed to be abundant near SWMU 23.

All of the AAs are located adjacent to Reach 1 of Dicks Creek, which has recently been remediated under Interim Measures. The vegetation along the creek is young, having been planted as part of the post-remediation restoration of the creek in 2010 and 2012. The specific types of vegetation are identified in the Dicks Creek Year 1 Completion Report (ENVIRON, 2011) and in the Dicks Creek Year 2 Sediment and Floodplain Soil Remediation Design Document (ENVIRONb, 2011). Since the Reach 2 vegetation and ecological description is not applicable to the AAs, it is not included in this RFI WP.

With the exception of a mist net survey conducted along Reach 2 of Dicks Creek by AK Steel in 2011, site-specific surveys of terrestrial animals have not been conducted along Dicks Creek or the Great Miami River. However, various animals and their signs have been observed in the Dicks Creek riparian corridor and floodplain during routine field work. Many of these species likely occur in other nearby habitats as well. Published surveys were also consulted to identify

common vertebrate species expected to occur in the region. The small size of terrestrial habitat patches near the AK Steel facility and the proximity of intense human activity are expected to limit the occurrence of less-common species. The 2011 mist net survey was conducted specifically to evaluate the potential presence of the Indiana bat. While numerous bats were netted, no Indiana bats were among those identified during the survey.

Terrestrial vertebrates include amphibians, reptiles, birds, and mammals. Frogs have frequently been observed along the banks of Dicks Creek. These may have included common species such as Blanchard's cricket frogs *(Acris crepitans)* or green frogs *(Rana clamitans).* Salamanders, though more reclusive, also likely occur in the area. Northern water snake *(Nerodia sipedon),* was observed swimming in Dicks Creek, and various other snake and turtle species may be present.

Abundant bird life has been observed along Dicks Creek, including waterfowl, raptors, shorebirds, scavengers, and songbirds. Belted kingfishers *(Ceryle alcyon)* and great blue herons *(Ardea herodias)* have frequently been observed fishing from the creek, and swallows (Family Hirundinidae) have been observed capturing emergent insects above the creek. Goldfinches (*Carduelis tristis)* have been observed along the edge of wooded habitat, and the presence of woodpeckers (order Picidae) was indicated by holes in trees, as well as the sounds of woodpecker calls and hammering. Hundreds of bird species pass through southwestern Ohio as migrants; however, those most closely associated with habitats of interest are species which commonly breed in the area (ARCADIS, 2012b).

Mammal tracks, including those of beaver *(Castor canadensis),* opossum *(Didelphis virginiana),* and white tail deer *(Odocoileus virginianus),* were repeatedly observed up and down the creek banks (ARCADIS 2012b). Active beaver slides and burrows were observed in both banks, as well as dismantled vegetation along the northern shoreline and woods. The burrows could also have been from muskrats *(Ondatra zibethicus),* as immediate diagnostic tracks were lacking. Other animal tracks and scat were observed, suggesting the presence of raccoons *(Procyon lotor)* and another inconclusively identified mammal species (probably muskrat). Squirrels (*Sciurus* sp.) were observed in trees in the wooded area to the north of the creek. additionally, striped skunk *(Mephitis mephitis)* and red fox *(Vulpes vulpes)* were directly observed during previous field work conducted by ENVIRON.

Terrestrial invertebrates are extremely diverse; ecological risk assessments typically focus on soil-associated species. Soil invertebrates form the base of the terrestrial food web, together with plants. Natural soils may contain more than 1,000 species of invertebrates. Detritivores and decomposers, which feed on decaying plant and animal material, are typically most abundant and include species such as springtails and mites. Other, larger species include earthworms, snails, and insects such as burrowing beetle grubs and fly larvae (Gorsuch et al., 2006).

Additional information on plants and animals in the vicinity of the AA's is provided in the *Interim Measures Remediation Work Plan* (ENVIRON, 2008c). Biological monitoring will be conducted in the future as part of maintenance and monitoring activities for the restored segments of Dicks Creek Reach 1, following completion of the Dicks Creek interim measures (currently scheduled to be completed in 2013). The biological monitoring consists of: (1) re-vegetation surveys; (2) stream habitat surveys; and (3) aquatic biological assessments at specific locations and at specific intervals. The biological monitoring is described in detail in *Restoration of Reach 1 of Dicks Creek, Monroe Ditch, and Outfall 002 Operation and Maintenance Plan, Middletown, Ohio*

included as Attachment 4 to *Dicks Creek Reach 1 Sediment and Floodplain Soil Remediation Design Document*, *Middletown, Ohio* (ENVIRON 2009a). Similar monitoring will occur in the portions of the creek that were remediated in 2012. These monitored areas include stream and streambank segments immediately adjacent to the designated AAs.

2.1.5.2 Threatened and Endangered Species

Information regarding the presence of threatened and endangered species, other species of concern, critical or exemplary habitat, unique geological features, and culturally or archaeologically important features was requested from the Ohio Department of Natural Resources (ODNR) and U.S. Fish and Wildlife Service (USFWS) in 2010. A site-specific threatened and endangered species review was completed by the ODNR Natural Heritage Program in March 2010. There are no existing or proposed state nature preserves or scenic rivers, unique ecological sites, geologic features, animal assemblages, state parks, forests, or wildlife areas within a one mile radius of the project area. Correspondence with the ODNR and USFWS is included in Appendix H of the CCR.

The ODNR has one record of a state threatened species-the Peregrin Falcon (*Falco peregrinus*) in the project area. The Peregrin Falcon is also listed as a federal species of concern. An individual Peregrine Falcon has been observed on-site. Peregrins are one of the most widespread birds of prey, feeding almost exclusively on medium-sized birds. "Peregrin" means wanderer, and they are known to often migrate long distances. They are found in a variety of habitats and prefer to nest on high cliffs along coastlines, rivers, and mountainsides. Increasingly, Peregrine Falcons are found in urban areas, where tall buildings provide suitable nesting sites.

Indiana bat *(Myotis sodalis)* is a federally and state endangered species that is expected to occur in Butler County. The Indiana bat hibernates in caves but forages along small stream corridors with well-developed riparian woods. USFWS indicates that Dicks Creek is within the summer range of the Indiana bat. However, the Indiana bat distribution is limited by its selection of hibernating caves (none of which are located in Ohio) and the susceptibility of those caves to human and natural disturbance. The USFWS has placed seasonal restrictions on the removal of suitable roost trees throughout the potential summer range of the Indiana bat; these restrictions are lifted if a site-specific bat survey identifies no Indiana bats. In response to a request for a site-specific review, the USFWS indicated in September, 2010 that "due to the project type, size, and location, we do not anticipate any impact on federally listed endangered, threatened, or candidate species, or their habitats." Based upon further consultation conducted within permitting processes for the Year 2 and Year 3 Dicks Creek Interim Measures, AK Steel conducted a mist net survey in 2011 within Reach 2 of Dicks Creek to evaluate for the potential presence of Indiana bats. While several bats were netted, none were Indiana bats. The seasonal restrictions on removal of suitable roost trees remains in effect for the remaining Dicks Creek Interim Measures.

Caddisflies of the genus *Hydroptila* were observed in the Great Miami River, based on aquatic survey results. Five species of *Hydroptila* have been listed by the State as threatened species, and one species of *Hydroptila* is a State species of concern. However, a total of 17 *Hydroptila* species are known to occur in Ohio. The CCR notes that because the (aquatic) larvae of this genus have not been fully characterized, specimens cannot be reliably identified to species based on the aquatic life stage (J. Keiper, Cleveland Museum of Natural History, pers. comm.).

2.2 Preliminary Assessment of the Nature and Extent of Contamination

The preliminary assessment of the nature and extent of contamination was conducted using historical information from previously conducted investigations of the Middletown Works facility from site investigations completed in the vicinity of SWMU 23 and along Dicks Creek for the AAs.

2.2.1 Historical Data

Several investigations have been conducted at the Middletown Works, some of which were associated with areas that have been identified as SWMUs, HWMUs, AOCs, and AAs (Figures 1 and 2). The materials managed, environmental releases, previous investigations, and data gaps are summarized in Appendix A of the CCR (Arcadis, 2012b). In addition, groundwater investigations have been conducted and are still ongoing. Environmental investigations in the vicinity of SWMU 23 are limited to water level measurements and sampling conducted during site-wide monitoring events (described further below). Environmental investigations in the vicinity of the AAs are associated with extensive soil/sediment assessment and restoration programs conducted within Dicks Creek (via ongoing Interim Measures).

2.2.1.1 SWMU 23

SWMU 23, consists of three (3) formerly used wastewater disposal lagoons, which occupy a small portion of AK property within the Former Coil Paint plant located toward the western side of Middletown Works (Figure 3) The Former Coil Paint Wastewater Treatment Lagoons, were located on the west side of the existing building at this location. Currently, the former location of the lagoons at the Coil Paint Area is owned by AK Steel Asset Management, but the surrounding parcels including the area of the Former Coil Paint Plant, are owned by others. The main building area currently is owned by NCI Group, based on information available on the Butler County Auditor's website, and is no longer a part of the AK Steel Middletown Works facility.

Based upon AK Steel's historic knowledge of the Coil Paint Plant processes, the following describes the treatment process historically used. The Coil Paint Line had a steel cleaning section to the line prior to the steel entering the paint section of the line. The cleaning section consisted of two alkaline cleaning steps with respective water rinsing sections, a phosphating step followed by a water rinse, and a passivating step followed by a water rinse. The purpose of the alkaline cleaning was to remove any residual dirt or oils. The purpose of the phosphating step was to improve the adherence of paint during the painting step. The purpose of the passivation was to provide corrosion resistance to the steel prior to the paint section of the line.

The treatment of wastewaters from the cleaning section of the Coil Paint Line that occurred prior to discharge to the Coil Paint lagoons consisted of chromium reduction of the spent passivating solution/rinse with ferrous sulfate, equalization of that wastewater stream with spent cleaners, rinse waters and & spent phosphating solutions. The resulting combined wastewater stream was then neutralized with lime. The neutralized slurry was then discharged to the two Coil Paint lagoons for solids settling. The solids were periodically removed from the two lagoons and placed in the third lagoon for solids drying. The dried solids were periodically removed and sent to the on-site landfill(s) at Middletown Works for disposal. No lagoon wastes were stockpiled or otherwise disposed at locations other than the Middletown Works landfill(s). No records were able to be located to identify the specific AK Steel Middletown Works landfill(s) in which the wastes were disposed.

The SWMU 23 wastewater lagoons were closed in the 1980's. Records from historic Middletown Works files indicate that one of the ponds (the sand bed, or Lagoon 3) was closed in 1983. Specific details of that closure are not available. A July 26, 1986 Middletown Works internal engineering report (see Appendix H) indicates that two (2) feet of clay and seeding was required in the remaining 2 lagoons to satisfy OEPA requirements. The engineering report states that clean clay was to be excavated from a new on-site construction area. No permeability data for the clay used in closure is known to be available. The engineering report states that the 2 lagoons were filled with soil and slag from the area immediately surrounding the lagoons, with grading conducted using a bulldozer. A bulldozer and compactor were then used to place a 2 feet thick clay cap in six-inch lifts. A copy of an aerial photograph dating to 1976 (Figure 4) indicates the position of the wastewater lagoons at the west end of the Coil Paint building. Based on historic information, the lagoons occupied an area of approximately 200 by 300 total square feet west of the Coil Paint building, with lagoons 1 and 2 being approximately 100 by 180 feet in dimension, and lagoon 3 (also indicated in some records as the sand bed) being approximately 120 by 200 feet in dimension.

Operations at the former Coil Paint plant generated wastewaters associated with cleaning of steel prior to painting. Paint wastes, spent solvents, and contaminated paints were reported as having been disposed of off-site as hazardous waste (ARCADIS, 2012b). Wastewaters generated at the former Coil Paint plant were treated at a WWTP prior to discharging to the series of three (3) unlined lagoons. Wastewater discharged to the unlined lagoons is reported to have contained metals.

The unlined lagoons were in operation from 1971 to 1978, and were installed in a sand and gravel area west of the Coil Paint Building. Two (2) of the lagoons were used to remove suspended solids from the Coil Paint wastewater stream. The third lagoon was used to dry the solids from the other two lagoons. Water from dewatering the solids was allowed to percolate through a sand and gravel bed into the underlying groundwater. The lagoons were backfilled in the mid-1980s and capped with a vegetated soil cover. The capped lagoons remain in this state today.

The potential exists for release of RCRA metals and volatile organic compound (VOC) constituents to the subsurface environment due to the unlined nature of the lagoons. Existing groundwater monitoring wells are located in the vicinity of SWMU 23 (Figure 5) and have been sampled for VOCs (Table 2). Based on the latest groundwater information included in the site documents (ARCADIS, 2012b), a pair of monitoring wells (GM-1S and GM-1D) are located at the south end of SWMU 23, upgradient to the unit based on available monitoring data. Well GM-2S is positioned at the north end of SWMU 23 in a cross gradient flow location.

Groundwater information summarized in the CCR (ARCADIS, 2012b) from the on-going sitewide groundwater monitoring program conducted by AK Steel, indicates that groundwater from SWMU 23 will migrate in a northeast off-site direction and toward an AK Steel on-site Production Wells 34 and 35 (Figure 5). Although no specific groundwater contamination is indicated by current samples of the existing monitoring wells in the immediate vicinity of SWMU 23, the potential exists for RCRA metals and/or VOC constituents to be present in soil and/or groundwater based on historical operation of the wastewater lagoons. SWMU 23 impacts have not been fully assessed and will be addressed via implementation of this RFI Work Plan.

The SWMU 23 area monitoring wells were installed in October 1987 (ARCADIS, 2012b) with well construction completion depths of 50 feet below ground surface (bgs) for GM-1S and GM-2S, 46 feet bgs for GM-3S, and 110 feet bgs for GM-1D. Copies of individual boring logs and well

completion reports for these wells, and another monitoring well (GM-3S on Figure 5) located east of the Coil Paint building are contained in Attachment A.

Boring log information indicates predominantly mixed sand and gravel deposits throughout the vertical thickness of drilled deposits at each of the above wells, including GM-1D. No bedrock was encountered during drilling of the wells. Measured groundwater depths in both the shallow and deep wells range from 24.42 feet to 27.30 feet from the well top-of-casing (TOC) measuring point (April 2011 data from CCR). It is noted that these measured water table depths are shallower in depth than the top of well screen depths within the respective wells, therefore, the individual well screens are fully submerged below the water table interface. Water level measurement events (ARCADIS, March 2013) indicate that wells GM-1S, and GM-2S, with well screens positioned at approximately 40 – 50 feet bgs and well GM-3S with well screens positioned at approximately 36 to 46 feet bgs, serve to provide measurement data for "intermediate aquifer" monitoring wells. Well GM-1S is identified as an "intermediate" aquifer well in a Geraghty & Miller, Inc. document contained within CCR (Appendix E-4). On the basis of this information, while wells GM-1S, GM-2S and GM-3S have the designation of being shallow aquifer wells, existing well screen interval depths indicate the wells are more appropriately representative of "intermediate depth" aquifer monitoring wells. The screen depth for GM-1D is approximately 100 to 110 feet bgs.

Available groundwater monitoring data from monitoring wells GM-1S, GM-1D and GM-2S do not currently indicate detectable levels of VOCs. Older data from the site indicates limited VOC and metals sampling and analyses were performed in the early stages of sitewide groundwater monitoring at Middletown Works. Records reviewed to date indicate that monitoring well GM-3S has historically been used only for groundwater elevation measurements.

To date, the soils at SWMU 23 have not been sampled and analyzed. Based on the absence of current data for soil and groundwater COPC concentrations, there is release potential for metal constituents and VOCs into the environment from historical operation of the wastewater lagoons. Although there are no documented releases of hazardous constituents from the lagoons, the potential release of constituents was identified as requiring RFI to characterize site conditions further such that the nature and extent of any site contamination can be determined. Sampling of soil and groundwater is considered necessary to evaluate subsurface environmental conditions at SWMU 23.

Investigation of Groundwater Flow Conditions

In 1987, an investigation was conducted to assess groundwater flow and quality beneath the site and the surrounding areas (Geraghty & Miller, Inc. 1989). During the field work phase, 46 shallow and 11 deep monitoring wells were installed and used for water-level collection and sampling for water quality. The facility production well pumping schedule was also evaluated concurrently with the collection of site-wide water level measurements. Lithologic information, pumping data, and plant-wide and regional water-level data were used to create a three-dimensional groundwater flow model to assist with the evaluation of groundwater flow conditions. A copy of the report was submitted to Ohio EPA.

In 1989, AK Steel began a voluntary program to contain groundwater on-site by modifying the operation of the existing groundwater production wells. Updates to and recalibration of the groundwater flow model were completed subsequent to 1989.

Groundwater Quality Investigations 1989 to 1990

As part of the groundwater investigation begun in 1987 to assess groundwater conditions within and at the boundaries of the plant, the groundwater quality was also evaluated in 1989 and 1990 (ARCADIS, 2012). The purpose of the groundwater sampling was to determine groundwater quality primarily at the boundaries of the plant for the upper, intermediate and lower aquifers.

The data from 1989 and 1990 (CCR Appendix E4) illustrated no exceedances of maximum contaminant levels (MCLs) for metals, and metals monitoring ceased. Metals analyzed included arsenic, barium, cadmium, calcium, chromium, iron, lead, magnesium, mercury, nickel, potassium, selenium, silver, sodium and zinc.

Data from 1989 and 1990 indicate the following VOC detections:

- Acetone was detected only in the March 1990 event in GM-1D at 64 ug/L and in GM-2S at 61 ug/L
- Carbon disulfide was detected only in the March 1990 event in GM-1S at 9 ug/L, GM-1D at 5 ug/L and GM-2S at 8 ug/L
- Methylene chloride was detected only in GM-1S in November 1989 at 10 ug/L and in March 1990 at 6 ug/L but was not detected in the June and September 1990 monitoring events. These detections exceeded the MCL of 5 ug/L.
- Trichloroethene was detected only in GM-2S in November 1989 at 10 ug/L, March 1990 at 10 ug/L, June 1990 at 11 ug/L and September 1990 at 8 ug/L. These detections exceeded the MCL of 5 ug/L. This well has been sampled multiple times since (see Table 2) with no further TCE detections.

Site-Wide Groundwater Monitoring

In 1987 and 1988, ARMCO (presently AK Steel) conducted a groundwater investigation by installing sixty-four monitoring wells in the upper, intermediate, and deep aquifers to define the groundwater quality and groundwater flow conditions at the site. The well locations were installed predominantly around the perimeter of the plant including the Slag Processing Area (ARCADIS, 2012b).

In 1989 and 1990, quarterly groundwater sampling events were implemented by collecting groundwater level measurements from all the monitoring wells and ten productions wells. Thirty-three monitoring wells were sampled during this monitoring program implementation. (Geraghty and Miller, Inc. 1989).

Since 1992, semi-annual groundwater monitoring events were initiated by collecting groundwater level measurements from the monitoring well network and the production wells. Monitoring well GM-01S located near SWMU 23 was removed from the Site-Wide Monitoring Program in 2001. Groundwater samples were collected from selected monitoring wells during the first event of each year and submitted for laboratory analysis for VOCs. Only groundwater level measurements are collected during the second event. Semi-annual reports are currently submitted to the Ohio EPA and USEPA in support of the work required by the Consent Decree. These monitoring reports include the site wide groundwater pumping data from AK Steel's production wells, groundwater flow maps from each aquifer, and groundwater quality data (first event only).

Groundwater quality data are collected on an annual schedule of 26 selected wells in the plant-wide monitoring well network and are submitted to characterize groundwater quality in the upper, intermediate and lower aquifer units beneath the plant. Data presented in the CCR, Appendix E-5 summarize the results of site wide groundwater monitoring from 1994 through 2010. The 1994 through 2010 analytical data summarized in the CCR Appendix E-5 from monitoring wells adjacent to SWMU 23, GM-01S, GM-01D, GM-02S and GM-03S, all have had results below the detection limit for the VOCs analyzed in each event in which these wells were sampled. The results of the 1994 through 2010 data for these wells has been extracted from the CCR Appendix E-5 and are presented in Table 2.

2.2.1.2 Additional Areas AA02 through AA09

AAs AA02 through AA09 (Figure 7) are privately owned commercial/industrial or residential properties that immediately abut Dicks Creek, and are located off-site and primarily west of Middletown Works. AK Steel has not conducted any steel-making operations on these parcels. According to historical recollections documented by Miami Conservancy District personnel and/or contractors to the Conservancy District, dredged sediments from Dicks Creek in the late 1960's and/or 1980's conducted by the Miami Conservancy District may have been placed on these properties during construction efforts. Interim Measures are on-going in Dicks Creek and Monroe Ditch to address polychlorinated biphenyls (PCBs) in stream sediment. If sediment dredged from Dicks Creek was placed on adjacent parcels as interviews with previous project employees and local residents indicate, these AA parcels have the potential for presence of PCBs in soil. AK Steel Locations AA02 through AA09 are off of AK Steel property.

Another area, AA01 which is located in a Dicks Creek defined floodplain area, has been addressed under an ongoing IM and will not be part of the investigation Work Plan for assessment of these areas. Additional Area 01 was remediated to remove all unacceptable concentrations of PCBs during the 2012 Dicks Creek remedial construction, and was restored in accordance with the approved Year 2 Dicks Creek Sediment and Floodplain Soil Remediation Design Document (ENVIRON, 2011). Additional details regarding the 2012 scope of work is documented in the Year 2 Dicks Creek Interim Measures Completion Report (KEMRON, 2012).

Although there are no specific documented releases from AK Steel operations at locations AA02 through AA09, interviews of individuals involved in historic dredging activities in Dicks Creek and residents adjacent to the creek indicate the possibility for historic dredged spoil placement to have occurred on these parcels.

The current Dicks Creek channel is nearly straight in the vicinity of AA02-AA09 due to the late 1960's channelization project. Records of interviews of personnel involved in channelization and dredging operations in Dicks Creek in the 1960's and/or 1980's, and individuals who lived in the vicinity of Dicks Creek during dredging operations in these time intervals, indicated that dredge spoils may have been placed on multiple parcels. The records specifically identified that dredge spoils were placed on AA02 through AA09. Most of the parcels were indicated to have dredge spoils placed primarily on the portion closest to Dicks Creek of each parcel. However, interview records for a previous owner of AA03 (Sturgell parcel) indicated that spoils were placed throughout the property along the railroad track. Based on the personal interview information, each of the AAs was identified as requiring RFI.

The CCR provides a review of several historical aerial photographs taken between July 1938 and March 2007. Aerial photo review presented in the CCR indicates no significant land development

or Dicks Creek channel modifications in the vicinity of AA02 through AA09 in photos from 1938, 1956, 1961 and 1962. An October 1968 photo, while indicating no clear changes to the AAs and surrounding properties that would definitively illustrate placement of dredging spoils, confirms that Dicks Creek appeared to have been channelized by this date. The CCR review also indicates that the southern three-fourths of the Sturgell Property (AA03) appeared to have been disturbed, and that the Ormans Welding Property (AA09) is present. Review of a later photo (April 1976) indicated some land disturbance at the southern end of AA02, with perhaps concrete placement on this property. The March 1980 photo indicated additional ground disturbance on AA02 with two circular disturbed areas seen. Later photos from 1986 through 2007 indicate no further significant property changes on AAs AA02 through AA09.

Figure 7 indicates the limits of Interim Measures (IM) remedial construction and restoration activities completed in 2010 (Year 1) and 2012 (Year 2) within the current Dicks Creek floodplain and channel. These IMs, completed immediately adjacent to the AAs, included gridded excavation and sampling to address PCB contamination. The 10' X 20' grids were excavated to pre-determined design depths and sampled to confirm that residual PCB concentrations did not exceed 3.0 mg/kg within grids being restored as stream channel, or 5.0 mg/kg in grids being restored as flood plain. These gridded areas illustrated on Figure 7 have been demonstrated to achieve the PCB action levels established by the Consent Decree. Additional Area AA-01 was included in the gridded excavation and restoration conducted during the Year 2 Dicks Creek IM.

The potential for release of PCBs into the environment onto these areas that are not on the AK Steel Middletown Works facility has been identified as requiring further investigation. Sampling of soil is considered necessary to evaluate surface/subsurface environmental conditions for AAs AA02 through AA09 and determine the nature and extent of potential PCB contamination on these parcels.

2.2.2 Historical Data Evaluation

The groundwater data collected historically from the groundwater monitoring wells at or near SWMU 23 from 1989 through 2010 and included in the CCR Appendices E-4 and E-5, are considered usable in the RFI for this SWMU. This data was deemed usable in the CCR which has been evaluated and accepted by regulatory oversight personnel. The groundwater data have been collected using standard sampling procedures, with review, oversight and approval of the resulting data and interpretations by USEPA and OEPA. These data were determined to be valid and fully usable as presented in the CCR; therefore, the RFI will incorporate the historical field parameters and VOC analytical results for these wells. No soil chemical data is available for SWMU 23, and therefore no historical soil data evaluation is necessary for this site. Additionally, no permeability testing data for the clay used in closure of the lagoons at SWMU 23 is known to be available. Thus, data gaps identified for SWMU 23 include current representative RCRA metals and TCL VOC groundwater data, permeability data for capping materials at SWMU 23 and representative soil samples from within and adjacent to the SWMU. These data gaps are addressed within this RFI Work Plan.

During the 2005 floodplain delineation work related to Dicks Creek Interim Measures, many floodplain soil samples were collected along the creek banks immediately adjacent to the AAs. Grids were established, with excavation to remove any unacceptable PCB concentrations in floodplain soil on the creek banks. During the floodplain soil delineation project, several soil borings (SBs) were installed and sampled within or immediately adjacent to AA02, AA05 and AA09. The locations of these soil borings, as well as the grids in which excavation and restoration have been completed within the Dicks Creek floodplain and channel are illustrated in Figure 7.

Soil boring R1BM-01 was installed adjacent to the southeastern corner of AA 02. Borings R1CS-06 and R1CS-07 were installed immediately north of AA 09. Soil borings R1EM-02, R1EM-03, R1EM-05 and R1EM-06 were installed within AA 05, and boring R1EM-04A was installed immediately south of this Additional Area. Borings R1EM-07 and R1EM-08 were installed to the east of AA-05.

Table 3 summarizes the total PCB analytical results of these soil borings within or immediately adjacent to AAs of Potential Concern. The highest concentration of total PCBs detected was in the R1BM-01 sample collected from 10-11 feet, with an analytical result of 2.495 mg/kg. Samples from borings R1CS-06 and R1CS-07 were the only other analytical results that were at or above 1.0 mg/kg. Analytical results for R1CS-06 and R1CS-07 at depths of 0-1 feet were 1.802 and 1.643 mg/kg, respectively and at 1-2 feet were 1.242 and 0.428 mg/kg, respectively. Soil borings installed on AA 05 included R1EM-02, -03, -05 and -06. The highest detection in any sample taken from these borings was from R1EM-02, with a reported concentration of 0.23 mg/kg.

These data within AA 05 and adjacent to other AAs were collected under an approved work plan, were subject to validation and regulatory review, and were collected in response to the same Consent Decree that is the basis of the AAs RFI. The data are considered valid and fully useable for the RFI. The RFI sampling program has been designed to consider and include these existing data for the AAs. No chemical data are available from samples collected within the boundaries of the other AAs. Therefore, additional sampling and analysis of PCBs is necessary to determine the nature and extent of PCB concentrations in the soils of AA02 through AA09.

2.2.3 Known and Suspected Source Areas of Contamination

RCRA metals or VOC constituents are not known to currently be present above regulatory standards in environmental media at SWMU 23 from historic Coil Paint Plant operations. As has been presented in previous sections of this work plan, SWMU 23 is a potential source area for RCRA metals and/or VOC constituent release to soil and groundwater. The lagoons have been closed for over thirty (30) years.

The AAs 02 through 09 have potential PCB-containing material sourced from the Dicks Creek stream bed and banks, due to reported spoils materials being placed on the properties at the time of stream dredging in the late 1960's and/or 1980's. Limited data from AA 05 and near AA09 and AA02 indicate low levels of PCBs in soil (Table 3 and Figure 7). Other than the historic spoils placement, no ongoing source is known to be present on any of the AAs for PCBs to be released to the environment.

2.2.4 Nature and Extent of Contamination

2.2.4.1 SWMU 23

Historic groundwater sample analyses from monitoring wells located in the vicinity of SWMU 23 (GM-1S, GM-1D, GM-2S) in 1989-1990 do not indicate elevated concentrations of metals. VOCs were detected in 1989-1990 at low concentrations as described in 2.2.1.1. Additional reported VOC data from annual sampling events completed from 1994 through 2011 indicate consistent non-detectable concentrations for VOC COPCs (CCR Appendix E-5). Data gaps for soil and groundwater sampling and analysis for full suite VOC and RCRA metals analysis for groundwater and will be filled via implementation of this RFI WP. No monitoring wells currently exist within the SWMU 23 area to monitor shallow water table conditions. The lack of shallow

aquifer/water table interface groundwater monitoring is also considered a data gap that will be addressed through the RFI. New monitoring wells will be installed through implementation of the RFI to provide the necessary data.

2.2.4.2 Additional Areas AA02 through AA09

The nature and extent of PCB contamination in soil media at the AAs is unknown, and will be determined via implementation of this RFI WP.

2.2.5 Data Gaps

The preliminary assessment of the nature and extent of contamination identified gaps in existing data pertaining to contamination originating from SWMU 23 and the AAs. Additional data are required to evaluate previously unsampled locations (e.g., surface and subsurface soil in the AAs AA02 through AA09) and that additional data are required to evaluate environmental media (e.g., SWMU 23 soils and groundwater) to fully evaluate the nature and extent of contamination originating from the two areas. Identified data gaps include:

SWMU 23:

- Data required to determine if VOC and RCRA metals constituents are present in the surface and subsurface soil adjacent to, below or within SWMU 23 at concentrations exceeding relevant human health and/or ESL;

- No permeability data for the clays used in closure of the lagoons is known to be available. If COPCs are identified in SWMU 23 soils at levels that may leach to groundwater, permeability data will need to be evaluated. This is an additional data gap that may require collection and analysis of surface/near surface samples in a subsequent RFI phase, to provide measurement of capping material permeability;

- Additional data are required to determine if COPC's are present above relevant screening levels in SWMU 23 groundwater, and if so, determine the nature and extent of groundwater contamination exceeding regulatory standards and/or relevant screening levels in the first water-bearing zone. Existing monitoring wells GM-1S and GM-2S are considered as intermediate depth monitoring wells (i.e., top of well screen is several feet below the water table interface). No monitoring wells currently exist within the SWMU 23 area to monitor shallow water table conditions. The lack of shallow aquifer/water table interface groundwater monitoring has been identified as a data gap.

Additional Areas AA02 – AA09

- Additional data are required to determine if PCB constituents are present in the surface and subsurface soil within the AAs at concentrations exceeding relevant human health and/or ESLs;
- Should PCBs be identified at the interface of sediment dredge spoils and native soils at concentrations that exceed leachable screening levels, additional investigation may be necessary to determine if PCBs have leached to groundwater.

Data screening for all parcels will include screening of soil samples to a depth of three (3) feet against ESLs. This depth is consistent with the depths in relevant ecological risk assessment guidance, based upon the potential ecological receptors of interest (ROIs). Additionally, all samples analyzed from soil borings will be screened against relevant USEPA Regional

Screening Levels (RSLs). For any property not owned by AK Steel, residential (e.g., unrestricted) levels will be used for RFI screening purposes. Leachability to groundwater will be assessed based upon soil concentrations detected, with standard regulatory methods such as USEPA Soil Screening Levels, protection of groundwater RSLs and/or other relevant screening values applied for evaluation. This screening approach will assure that the above identified data gaps are addressed in performance of the RFI.

2.2.6 Waste Containment

SWMU 23 closure included filling and vegetative cover being placed at the former wastewater treatment lagoons as described in Section 2.2.1.1. The closure provided reduced potential for infiltration of precipitation, thus reducing potential for migration of constituents of potential concern in the subsurface. The permeability of the clay used for closure of the lagoons is not currently known.

Using historic aerial photographs and drawings, the size of the individual wastewater treatment units that comprise SWMU 23 are as follows: Lagoons 1 and 2 were each approximately 60 by 170 feet in size; Lagoon 3 (sand bed) was approximately 120 by 200 feet. The complete pond area thus encompassed approximately 245 feet by 285 feet. The entire closed SWMU 23 area currently is vegetated (see Figure 5).

No specific documented waste containment measures have been implemented at the AAs.

2.3 Preliminary Site Conceptual Model

A preliminary site conceptual model (SCM) was prepared and presented in the CCR (ARCADIS, 2012b). This section summarizes key points from the CCR related to the SCM. The CCR SCM Figures 48 and 49 are reproduced (and revised as necessary) in this RFI WP as Figures 8 and 9.

For purposes of this RFI WP, the existing preliminary SCMs for SWMU 23 and the AAs have been incorporated. The SCMs for SWMU 23 and for the AAs will be updated and refined following RFI data collection. A Risk Assessment Assumptions Report (RAAR) will be prepared, and the risks to human health and the environment will be assessed as part of the RFI.

It is currently anticipated that SWMU 23 will include an evaluation of risk to both potential human and ecological receptors. The need for ecological risk assessment of the AAs will be dependent upon the RFI data findings. The primary potential ecological receptors related to the AAs would be those associated with the habitats of Dicks Creek. Potential ecological impacts within Dicks Creek are being addressed as part of the Dicks Creek Interim Measures effort. Therefore, pending additional data acquisition and consistent with the Preliminary SCM for the AAs as included in the approved CCR (Fig 45 of the CCR), ecological receptors will not be evaluated for the AAs unless site soil data indicate chemical (PCB) constituents in surface soil (based on the 0-3' analytical results) that would be available to ecological receptors. The SCM (Fig 9) includes a pathway for ecological evaluation should the presence of PCBs above relevant ESLs in surface soil of the AAs be confirmed. Similarly, SWMU 23 (summarized in Figure 8) will have an ecological risk assessment conducted based upon a comparison of surface soil concentrations of VOC and RCRA metals to relevant ESLs.

2.3.1. Primary Sources and Migration Pathways

2.3.2 Preliminary Human Health Site Conceptual Model

Based on information regarding land and groundwater use at and around the site described in the sections above, a preliminary identification of potentially exposed populations under current and reasonably expected future land use was completed in the CCR.

The potential ROIs identified in the CCR for both the AAs 02 through 09 and SWMU 23 include:

2.3.2.1 Receptors of Interest

As defined in the current Preliminary SCM (Figures 8 and 9), human health ROIs are identified as "on-site" and "off-site" and include the following:

AA 02, 05, 06, 07, 08, 09

Off-Site: Routine Workers; Maintenance Workers

AA 03, 04

Off-Site: Residents

SWMU 23

Off-Site: Residents (if migration were to occur from SWMU 23 to off-site residential receptors)

On-Site: Routine Workers; Maintenance Workers; Trespasser

As noted previously, all areas subject to RFI under this work plan are off-site with the exception of on-site workers and potential trespassers at SWMU 23. The potentially exposed populations in off-site areas include residents, routine workers and maintenance workers.

2.3.2.2 Exposure Pathways

Potential pathways by which human receptors may be exposed to chemicals of interest include soil and groundwater dermal contact and ingestion, and inhalation of soil and groundwater particulate and vapor.

Off-site

Potable water is provided to this area by the City of Middletown via well fields 2 miles northwest of the site, and while groundwater off-site is not currently believed to be used for potable purposes, there are no current restrictions in place which would prevent off-site groundwater from being used for potable purposes in the future. As a result, the domestic use of groundwater is considered a potentially complete pathway. The evaluation of potential groundwater exposure of off-site residents will include ingestion, dermal contact, and inhalation during potable (if any) and non-potable residential uses of groundwater.

Off-site residents in residential parcels north and/or south of SWMU 23 could also potentially be exposed to volatile organic vapors from groundwater, if the groundwater contained VOC constituents and a plume were to migrate off site and vertically through the soil column underneath building foundations into indoor air (e.g., vapor intrusion).

For the AAs of Potential Concern, historical reports of deposition of dredge spoils in portions of these parcels indicates that PCB constituent concentrations may exist in soil at unacceptable

concentrations. Off-site residents or workers at the AAs may also be potentially exposed to airborne particulates from on-site surface soil. As presented in the preliminary SCM for the AAs (CCR, ARCADIS, 2012b), potential exposure of residents in areas where historical deposition of dredge spoils occurred is assumed to include incidental ingestion and dermal contact with exposed surface soil and inhalation of (1) particulates from surface soil, (2) vapors from subsurface soil that migrate into outdoor air, and (3) vapors from subsurface soil that migrate through building foundations into indoor air.

On-site

On-site workers for SWMU 23 will be evaluated for dermal contact, ingestion, and inhalation pathways for soil. SWMU 23 on-site workers also will be evaluated for dermal contact, ingestion and inhalation pathways for groundwater.

2.3.2.3 Chemicals of Potential Concern

In areas where exposure pathways are potentially complete, chemicals of potential concern will be identified by comparing concentrations detected in relevant media with the screening values identified in Appendix G of the 2012 CCR (ARCADIS), or the most recent version of the USEPA RSLs. Such identification will also consider background levels and detection frequency.

2.3.3 Preliminary Ecological Site Conceptual Model

Based on information regarding land and groundwater use at and around the site a preliminary identification of potentially exposed ecological populations under current and reasonably expected future land use was completed in the CCR. Figure 8 provides the preliminary SCM for SWMU 23. An ecological risk assessment will be performed for SWMU 23 consistent with this SCM.

As stated above, the potential ecological receptors within Dicks Creek are being addressed through the Dicks Creek interim measures effort. Therefore, pending additional data acquisition and consistent with the preliminary SCM originally developed in the approved CCR (see CCR Figure 45), ecological receptors will not be evaluated for the AAs and the ecological SCM for this effort is limited to SWMU 23 unless analytical data indicate chemical (PCB) contamination in surface soil of the AAs above USEPA Region 5 ESLs. The SCM (Figure 9) includes a pathway for ecological evaluation should the presence of PCBs above ESLs screening levels in surface soil of the AAs be confirmed. Figure 9 identifies currently anticipated ecological ROIs for the 8 AAs should analytical results indicate the need for conduct of an ecological risk assessment. Ecological receptors for the AAs, as applicable, will be refined in the RAAR.

The potential ecological ROIs identified in the CCR for SWMU 23 and the adjacent woodland habitat include:

- Soil Invertebrates;
- Plants; and,
- Terrestrial Wildlife.

The objectives of the ecological risk evaluation are to identify potentially significant risks of adverse effects on ecological resources at the site (if any), and to develop risk-based clean-up levels where potentially significant ecological risks exist. Potential ecological risks will be evaluated for terrestrial areas of the SWMU 23 site where (1) ecologically relevant habitat is present, and (2) historic site activities (e.g., wastewater discharges at SWMU 23) could potentially have resulted in chemical constituents exceeding acceptable levels.

Ecological components of the preliminary site conceptual model for the woodland habitat adjacent to SWMU 23 are described below, as summarized from the CCR. The ecological site conceptual model includes the identification of exposure pathways, ROIs, and assessment endpoints, as well as a discussion of the approach for identifying chemicals of potential ecological concern (COPECs).

2.3.3.1 Exposure Pathways

Potential pathways by which ecological receptors may be exposed to chemicals of interest include dermal uptake from soil, soil ingestion, and ingestion of food (i.e., plant and animal tissue). For soil-dwelling organisms, all exposure pathways are integrated by comparing soil concentrations to toxicological benchmarks. For wildlife, doses obtained through ingestion of soil and food are directly calculated for each exposure pathway and summed for comparison to toxicity benchmarks. Data are not available to directly assess dermal exposures for wildlife; however, this is not considered a significant data gap, because dermal exposures are expected to be much less important than ingestion due to the protective effects of fur and feathers. The assessment of amphibians and reptiles is also limited by the availability of appropriate toxicological data. Ecological receptors that will be exposed primarily through direct contact with soil include soil invertebrates and plants. Wildlife receptors include herbivores (e.g., mourning dove, meadow vole), invertivores (e.g., American robin, short-tailed shrew), and carnivores (e.g., American kestrel, red fox). Wildlife receptors are exposed via prey ingestion (for bioaccumulative compounds only), as well as incidental soil ingestion (Figure 8).

2.3.3.2 Receptors of Interest

It is not feasible to complete ecological risk calculations for all species occurring in the vicinity of the site. Such an effort would also be duplicative because of the similarity of exposure patterns among closely related species and those with like feeding guilds. For these reasons, representative ROIs are selected for quantitative risk assessment purposes. The selected receptors are representative of entire classes of organisms (i.e., functional groups).

Potential receptors identified for the woodland habitat are as follows:

- *Soil invertebrates* – Soil-associated invertebrates, such as earthworms, live in constant and direct contact with surface soil that may be impacted by COPECs. The soil invertebrate community influences the suitability of soils for various plant species and serves as a prey base for higher trophic level organisms;

- *Plants – Plant* roots are in constant and direct contact with soil that may be impacted by COPECs. Plant communities provide food for herbivores and essential habitat for many animal species;

- *American robin (Turdus migratorius)* – The American robin is selected to represent the invertivorous bird feeding guild. The American robin feeds primarily on worms and other terrestrial invertebrates. Robins are common throughout the United States during the breeding season and inhabit moist forests, swamps, and open woodland (U.S. EPA 1993);

- *Short-tailed shrew (Blarina brevicauda)* – Short-tailed shrews represent the invertivorous mammal feeding guild. Short-tailed shrews feed primarily on soil invertebrates such as earthworms and slugs (U.S. EPA 1993). Shrews prefer cool, moist locations (U.S. EPA 1993);

- *Mourning dove (Zenaida macroura)* – Mourning doves represent the herbivorous bird feeding guild for terrestrial areas. Mourning doves are almost exclusively herbivores, consuming mostly seeds. Exposure parameters for mourning doves were identified as part of the development of the U.S. EPA's Ecological Soil Screening Levels (U.S. EPA, 2005). Mourning doves are more likely to occur in the woodland habitats of interest than the herbivorous bird species (e.g., quail) included in the U.S. EPA's Wildlife Exposure Factors Handbook (U.S. EPA, 1993);

- *Meadow vole (Microtus pennsylvanicus)* - Meadow voles, primary consumers representing the herbivorous mammal feeding guild, are found over most of the northern half of the United States (U.S. EPA 1993). Meadow voles prefer grassy habitat in moist to wet areas (U.S. EPA 1993). Voles often constitute a major portion of the diet of some carnivorous birds and mammals (e.g., raptors, mink);

- *American kestrel (Falco sparverius)* - The American kestrel is selected to represent the carnivorous bird feeding guild. Kestrels nest in tree cavities and prefer woodland edges and open areas such as grasslands, old fields, and marshes (Brewer et al. 1991). They are found in areas with human activity more often than other raptors. Kestrels feed on insects, amphibians, reptiles, birds, and mammals (U.S. EPA 1993); and,

- *Red fox (Vulpes vulpes)* - Red foxes are top-level carnivores and are selected to represent the carnivorous mammal feeding guild. They are the most widely distributed carnivore in the world, and they have been observed on-site. Red foxes prefer broken, varied habitat (U.S. EPA 1993).

Specific ecological receptors for SWMU 23 and the AAs (if required based upon PCB concentrations identified during the RFI) will be identified in the RAAR.

2.3.3.3 Assessment Endpoints

Assessment endpoints are explicit expressions of the environmental value that is to be protected, operationally defined by an ecological entity (e.g., fish, birds, mammals) and its attributes (e.g., community structure, survival, growth, reproduction). Assessment endpoints are selected based on ecological relevance, susceptibility (which is a combination of toxicological sensitivity and potential for exposure), and relevance to management goals. The following assessment endpoints are selected for the woodland habitat area:

- *Soil invertebrates* – Survival and viability of invertebrate community, including species composition, relative abundance and function, trophic structure;

- *Plant* -Survival and viability of plant community, including species composition, relative abundance and function, trophic structure; and,

- *Terrestrial Wildlife, including birds, mammals* – Survival and viability of wildlife community, including species composition, relative abundance and function, and trophic structure.

2.3.3.4 Chemicals of Potential Ecological Concern

Chemicals of potential ecological concern will be identified for the woodland habitat adjacent to SWMU 23 in a Screening Ecological Risk Assessment (SERA), based on chemical concentrations in surface soil. Similarly, the potential for ecological impact of PCBs in soil at the AAs will be evaluated based upon PCB concentrations in surface soils, if present. The identification of

COPECs will employ the screening values identified in Attachment 4 of the Consent Decree (i.e., Eco SSLs and Region 5 ESLs) and will be conducted in accordance with U.S. EPA (1998, 2001) guidance.

2.4 Description of Interim Stabilization Measures

2.4.1 SWMU23

The SWMU 23 wastewater lagoons consisted of unlined lagoons that were in operation from 1971 to 1978. As previously discussed, the lagoons were closed in the 1980s. As discussed in Section 2.2.1.1 of this RFI Work Plan, two of the lagoons were capped with clay and vegetative cover established; the third lagoon had previously been closed prior to this capping effort. No information has been identified regarding the methods used in closure of the third lagoon. The current condition of the former lagoons indicate they are closed level with surrounding ground and capped with a vegetated soil cover.

2.4.2 Additional Areas AA02 – AA09

The AA02-AA09 areas are all off-site properties. No specific interim stabilization measures have been implemented on these properties by AK Steel.

2.5 Data Quality Objectives

Data Quality Objectives (DQOs) were developed in accordance with EPA's seven step process to serve as a basis for designing a plan for collecting data of sufficient quality and quantity to support the goals of the RFI/CMS (EPA 2006). The seven steps include: (1) State the Problem; (2) Identify the Goals of the Investigation; (3) Identify Information Inputs; (4) Define the Boundaries of the Investigation; (5) Develop a Decision Rule; (6) Specify Performance Criteria; and (7) Develop a Plan for Obtaining Data. The DQOs were developed after the historical data for the site were assessed for usability and evaluated, and data gaps were identified.

The DQO process identified the following six problems to be addressed by the goals of the RFI:

1. Determining the nature and extent of contamination originating from SWMU 23, if any;
2. Determining the nature and extent of contamination at each Additional Area (02 through 09);
3. Evaluating potential, current and future ecological and human heath exposures, both short term (acute) and long term (chronic), as a result of releases from SWMU 23 to environmental media, if any;
4. Evaluating potential, current and future ecological and human health exposures, both short term (acute) and long term (chronic), as a result of PCBs in soil at each Additional Area (02 through 09); and,
5. Determining if a Corrective Measures Study (CMS) is necessary for SWMU 23 or one or more of the AAs, based on the site specific data and information gathered during the RFI.
6. Evaluate the overall integrity of existing containment structures at SWMU 23.

The DQO planning team consists of the AK Steel Project Manager, and the KEMRON technical team, which includes the Project Manager, chemists, geologists, engineers, risk assessors, and quality assurance (QA) specialists. The duties of the team members are discussed in the attached *RFI/CMS Quality Assurance Project Plan* (QAPP). The decision maker for the DQO planning team is the AK Steel Project Manager. All final decisions must be formally approved

by United States et al. and be consistent with the terms of the Consent Decree. The DQOs for the RFI of SWMU 23 and the AAs AA02 through AA09 are presented in Table 4.

The data collected under this scope of work will be used in the preparation of a human health risk assessment and an ecological risk assessment and in the evaluation. Therefore, it is critical that the final data be definitive data for purposes of decision making.

3.0 RFI Work Plan

The RFI is designed to collect and analyze the data necessary for evaluating the nature and extent of potential contamination, assess the risk to human health and environment posed by the contamination, and collect information needed to prepare for a CMS, if needed. This RFI Work Plan presents a summary of tasks comprising the investigative phase of work to be performed for both SWMU 23 and AAs AA02 through AA09. Environmental sampling performed for both investigation areas will utilize common types of environmental drilling equipment, and include sampling and analysis of collected soil samples for identified COPCs; namely RCRA metals and VOCs in soil for SWMU 23, and PCBs in soil for AAs AA02 through AA09. Groundwater will be sampled from both new and existing monitoring wells in the vicinity of SWMU 23 for RCRA metals, aluminum (AL), zinc (ZN), and VOCs. No groundwater sampling is proposed for the AAs at this time.

Elements of the RFI Work Plan presented in this section include:

- Sampling Objectives
- Project Management
- Data Collection QA
- Data Management and Reporting
- Detailed Descriptions of Methods and Procedures for Performing the Required Tasks
- Public Involvement
- Proposed Schedule for Conducting the Work

3.1 Work Plan and Sampling Objectives

The key objectives of the RFI of AAs AA02 through AA09, and SWMU 23 will be to:

- Characterize the nature and extent of contamination for the subject parcels; and,
- Gather environmental data necessary to support an assessment of risk to human health and the environment posed by any identified releases of constituents to environmental media.

3.2 Project Management

The project management team consists of the AK Steel Project Coordinator and Project Manager, the KEMRON Project Manager, KEMRON field team, and the drilling and laboratory subcontractors. The duties of the team members are discussed in the *Quality Assurance Project Plan* (QAPP) included as Attachment B. AK Steel and KEMRON have the overall responsibility for management of the project.

Project Coordinator (AK Steel)

The primary function of the AK Steel Project Coordinator (Pat Gallo) is to ensure that technical, financial, and scheduling objectives are achieved successfully. The project coordinator will approve all external reports (deliverables) before their submission to USEPA et al.

Project Manager (AK Steel)

The AK Steel Project Manager (James Kemp) will be responsible for implementing the project and has the authority to commit resources necessary to meet project objectives and requirements. The AK Steel Project Manager will work directly with the KEMRON Project Manager to monitor and direct daily activities; communicate health and safety information to project personnel; serve as a liaison between KEMRON, the subcontractors, regulatory

personnel, and the AK Steel Project Coordinator. The AK Steel Project Manager will work directly with KEMRON Project Manager.

Project Manager (KEMRON)
The KEMRON Project Manager (Mary Lou Rochotte) has responsibility for ensuring that the project meets the objectives of USEPA et al.'s and KEMRON's quality standards. The KEMRON Project Manager will report directly to the AK Steel Project Manager.

Project Scientists (KEMRON)
Field investigation sampling will be supported by a team of KEMRON scientists, engineers and technicians experienced in performance of environmental sampling on hazardous waste sites. The KEMRON team will report directly to the KEMRON Project Manager.

Subcontractors
Subcontractors will be responsible for those activities which they are contracted to conduct (e.g., drilling soil borings, analyzing samples, etc.) and ensuring that these activities meet the project's objectives. Subcontractors will perform work under the direction of the KEMRON Project Manager and/or a designated KEMRON Field Team Leader.

An organizational chart for the project management team is included as Figure 10.

3.3 Data Collection Quality Assurance
A Quality Assurance Project Plan (QAPP) for this RFI investigation is presented in Attachment B. The *SWMU 23 and AAs 02 through 09 RFI QAPP, Revision 1* (KEMRON, 2013) incorporates procedures necessary to document all monitoring, sampling, field measurements, and sample analyses to be performed during the RFI and includes a description of QA objectives and data reduction and reporting requirements. Data records, field logs, analytical laboratory reports, and chain-of-custody documentation will be reviewed for accuracy and completeness, assuring adherence to high scientific and technical standards.

The COPCs at SWMU 23 (RCRA metals and VOCs) and AA02 through AA09 (PCBs) are also included in the RFI/CMS currently in progress by another contractor at SWMUs 38 and 39. For consistency in the work conducted under the Consent Decree, the same analytical laboratory, TestAmerica, is anticipated to conduct analyses of the samples from SWMU 23 and AA02 through AA09. The laboratory SOPs are incorporated within the *SWMU 23 and AA02 through AA09 RFI QAPP, Revision 1* (Attachment B).

3.4 Data Management and Reporting
A Data Management Plan (DMP) for the RFI is included as Attachment C. The DMP addresses the procedures necessary to document and track data and results associated with the RFI. The DMP describes the project; project organization and responsibility; sample identification; chain-of-custody guidelines; and data flow, transformation, reduction, transfer, validation, analysis, reporting, tracking, and security. All quality assurance/quality control (QA/QC) procedures will be structured in accordance with applicable USEPA and OEPA requirements, regulations, guidance, and technical standards, as documented in the *RFI QAPP*. AK Steel will provide United States et al. all locational, soil, water, and groundwater data in an electronic database suitable for display in a GIS format as part of the final project reporting.

3.5 Methods and Procedures for Performing the Required Tasks

Detailed descriptions for performing environmental investigation of SWMU 23 and AAs AA02 through AA09 are presented in Section 4 of this Work Plan.

3.6 Public Involvement

A Public Involvement Plan (PIP) already is in existence for Interim Measures work being performed under the Consent Decree, and for the SWMU 38 and 39 RFI. A PIP for this RFI is provided as Attachment D. AK Steel and KEMRON will fully implement this PIP to conform to all public involvement requirements of the Consent Decree. All land owners on which the RFI will occur will be contacted to request access for purposes of implementing the RFI. The general public will be kept abreast of RFI activities via the fact sheets and letters that are prepared consistent with the PIP.

3.7 Proposed Schedule for Conducting the Work

A schedule for the implementation of the RFI is included as Figure 11. The schedule includes the selection of subcontractors, implementation of field work, data analysis, and the preparation and submittal of various reports. Milestones listed in the schedule include the preparation and submittal of the: (1) RAAR; (2) Environmental Covenant (if determined to be applicable for SWMU 23); (3) RFI Report; (4) Fact Sheet after the completion of the field work and the approval of the RFI Report. The schedule will be updated upon final approval of this Work Plan, and as necessary based upon completion of key work elements throughout conduct of the RFIs for SWMU 23 and for the 8 AAs.

3.8 Site Access Requests for Sampling on Additional Areas

AK Steel will request access from individual property owners for the sampling proposed in this RFI WP on AA02 through AA09 private properties. Sampling will occur on the AAs following approval of this RFI WP and execution of access agreements for the AAs.

The currently proposed technical approach for investigation of SWMU 23 does not require access agreements since all sampling is proposed on property owned by AK Steel. Should the initial RFI results indicate that additional future RFI work is required on properties not owned by AK Steel, access agreements will be sought with the appropriate property owners at that time.

4.0 Sampling Plan (SWMU 23 and Additional Areas)

Environmental sample collection will follow standard operating procedures (SOPs) that are consistent with standard practice for environmental sample collection. KEMRON will implement SOPs where possible that already have been reviewed and approved under the Consent Decree to assist in streamlining the RFI process. The SOPs applicable to field work for this RFI and significant information regarding sample management and analytical laboratory standards, methods and procedures is provided in the RFI Quality Assurance Project Plan (QAPP) in Attachment B. Individual SOPs for field work are identified by number, with summary information regarding the field procedures, with detailed information provided in the field SOPs.

Environmental sample collection for SWMU 23 and AAs AA02 through AA09 will involve sampling of soil using commonly available soil boring technologies with either hollow-stem auger assisted split-spoon sample collection or push-tube type sample collection such as with a Geoprobe unit. Groundwater sampling at SWMU 23 will be conducted using standard EPA-approved methodologies as well.

The bulk of sampling to be performed during site investigation work at both sites (SWMU 23 and AAs) will be completed using a direct push-tube sampling rig or utilizing rotosonic drilling. Drilling will be conducted in accordance with SOP # 12-SBC-AKMW. Drilling method will be determined and selected based upon expected subsurface conditions.

Individual soil boring locations (soil boring and well borings) will be marked on location and cleared of subsurface utilities prior to start of drilling. Subsurface utility clearance will involve notification of the state-sponsored utility location service, as well as AK Steel engineering staff. Utility clearance will require a minimum of 48 hours prior to start of any drilling. In accordance with KEMRON's Corporate Health and Safety Plan, individual boring locations are manually cleared to a depth of 4 feet for purposes of avoiding the presence of potential unmarked utilities.

When sampling with push tube methods, soil samples will be collected on a continuous basis using standard 4-foot length sample tube. Individual samples will be retained within a clear acetate sleeve which will be split in half lengthwise upon retrieval. Individual soil cores will be visually examined by the geologist who will also log the core as samples are collected. Hollow Rotosonic methods will allow for collection of a continuous soil core. The field geologist will retain soil samples for lab analysis from the above specified depth intervals in order to characterize soil quality.

Upon reaching the soil boring termination depth, individual soil borings will be backfilled with granular bentonite which will be hydrated with water in place. Boring locations will be measured with a tape measure from a nearby surface structure and will be georeferenced using a portable Global Positioning System (GPS) device. Residual soil sample material from soil cores not retained for analysis will be contained and properly managed as Investigation Derived Waste (IDW).

4.1 SWMU 23 Sampling

Environmental sample collection within SWMU 23 will involve the completion of soil borings within the confines of the former lagoons, and four (4) additional soil borings located outside of the lagoon area perimeter but within AK Steel owned property, at locations shown on Figure 12. Soil sampling will terminate upon encountering the water table interface or encountering bedrock, whichever occurs first. Continuous soil sampling to the depth of the water table interface,

estimated at 20 ft. or more, is considered sufficient in determining whether residual constituents from historical surface disposal is evident within the subsurface soils.

A total of four new groundwater monitoring wells will be installed at SWMU 23. Three wells (two shallow and one intermediate) will be installed on the east and northeast portions of SWMU 23 to provide groundwater monitoring locations at the currently interpreted side gradient and down-gradient positions, based on the latest available potentiometric surface maps for the area (ARCADIS, 2012). The wells located to the east will be a nested pair of wells to provide both shallow and intermediate monitoring. A fourth well will be installed on the west side of SWMU 23 to provide a shallow monitoring well in the currently interpreted upgradient location. Three of the new monitoring wells will be completed as shallow depth/water table interface monitoring wells with screen intervals set at approximately 20 – 30 feet bgs (a minimum of 9 feet of screen submergence below water table). The fourth will be completed as an intermediate depth monitoring well with screen intervals set at approximately 40-50 feet bgs. Installation and sampling of three (3) water table monitoring wells and one (1) intermediate depth monitoring well will provide shallow groundwater quality, as well as complement data from existing intermediate depth wells (GM-1S, GM-2S) and deeper aquifer monitoring well GM-1D.

Groundwater samples will be collected from the new monitoring wells and existing monitoring wells GM-1S, GM-1D, GM-2S and GM-3S which are located near SWMU 23 (Figures 5 and 12). The existing wells are assumed to be functional; however, sampling will occur only following a well inspection and competency check. Groundwater monitoring will be completed to determine the nature and extent of COPCs.

Additionally, all soil borings and monitoring well installations will occur on AK Steel owned property for this phase of the RFI. Once the data are available from the RFI field effort described in this work plan, a determination will be made regarding any need for additional environmental media sampling to determine SWMU 23 impacts off AK Steel owned property. Recent photographs of the SWMU 23 lagoon area are presented in Attachment E.

Existing Monitoring Well Inspection

The existing monitoring wells, which are currently used for water level monitoring, will be inspected for usability for groundwater sample collection. Boring logs are available for each of the existing wells (contained in Attachment A). Each well will be measured for water level and total depth to see if the depth measurement equates with historical completion depth. The bottom will also be sounded for sediment buildup.

Available information from AK Steel indicates that well GM-1D was last sampled in April 2011. Data for this well, summarized in an annual groundwater report prepared by Arcadis, Inc. (September 2011), which was analyzed for trichloroethene (TCE), indicated no detectable TCE concentrations in deeper aquifer material groundwater at the SWMU 23 area. In addition to this reported data, the CCR also indicates that upgradient wells GM-1S and GM-1D, and cross gradient well GM-2S do not currently contain detectable levels of VOCs (ARCADIS, May 2012b).

It is anticipated that wells GM-1S, GM-1D, GM-2S and GM-3S will function adequately for sampling during this RFI due to the presence of significant medium to coarse sand and gravel materials logged at the existing well locations. Should well inspection efforts indicate that the wells appear to be silted in, then an effort will be made to redevelop the affected wells. Inspections and redevelopment will be completed prior to installation of new wells. Should individual wells be determined non-functional then new well installations may be recommended.

Monitoring Well Installation and Sampling

Four (4) new monitoring well installations are planned for this RFI investigation. These new wells will be installed in accordance with SOP # 12-MWI-AKMW, with three (3) new wells screened in the shallow water table (approximately 20-30 ft bgs), and one (1) new well screened at an intermediate depth (approximately 40-50 feet bgs). Figure 12 indicates the planned location of each new monitoring well. This figure also shows the general historical groundwater gradient direction from annual potentiometric surface maps (1995 – 2011) for the intermediate and lower aquifer monitoring wells based upon data provided in the semi-annual sitewide groundwater monitoring reports prepared by others.

Each monitoring well, including the existing wells GM-1S, GM-1D, GM-2S and GM-3S and the four (4) new monitoring wells at SWMU 23, will be sampled using low-flow sampling methodology as described in SOP # 12-GWS-01.

Each well will have a representative sample collected, with samples analyzed for Target Compound List (TCL) VOCs (EPA Method 8260), and RCRA metals, Al and Zn (EPA Methods 6010 and/or 6020).

At the present time, no vapor intrusion pathway sampling and analysis is planned, based on the absence of detectable VOCs in the existing groundwater monitoring wells at SWMU 23. Should VOCs be detected in groundwater samples associated with SWMU 23 during the RFI, the concentrations of the detections will be evaluated and a determination will be made as to whether sampling and analysis of soil and groundwater for a potential vapor intrusion pathway is appropriate.

Soil Boring Completions

Figure 12 indicates the planned locations for soil boring completions. Three (3) soil borings will be completed within each of the areas once occupied by Lagoons 1 and 2 (northern and central smaller lagoons). Six (6) soil borings will be completed within the larger southern Lagoon 3. Boring locations will be staked in the field using measurements from existing key features (building structures, monitoring wells, etc.) as the lagoons are now buried with no visible surface expression of lagoon boundaries. Four (4) additional soil borings will be completed on each side of the lagoons within the AK Steel owned property to sample soil at locations exterior of the former lagoons (Figure 12), with the northeast ,east and west side borings being completed as water table monitoring wells. (Note that in addition to providing soil samples, boring locations SB-4 and SB-15 will be completed with shallow monitoring well installations, and SB-16 will be completed with an intermediate depth well installation. The shallow monitoring well location paired with SB-16 will not be sampled for soil.) Four additional surface soil samples (0 to 1 ft below ground surface) will be collected as indicated on Figure 12 for use in evaluating direct contact exposure pathways.

Continuous soil sampling will be advanced (via push tube or rotosonic drilling) until the water table is reached. Continuous soil sampling to the depth of the water table interface, estimated at 20 ft. or more, is considered sufficient in determining whether residual constituents from historical surface pond discharges have impacted subsurface soils. Available data from monitoring wells indicates the water table to be within 25 feet of the ground surface. Existing borings logs for the SWMU 23 area wells indicate essentially medium to coarse sand and gravel as the predominant geologic formation at SWMU 23.

The field geologist will log individual soil cores for recovery percentage, soil type, and any notable environmental characteristics (discolorations, moisture, odors, non-native material, etc.). The field geologist will also be equipped with a photoionization detector (PID) to field screen individual soil samples for the presence of VOC in soil headspace samples. Soil samples will be retained at 2 foot sample intervals for headspace testing and for potential analytical sample selection. Individually collected samples will be kept in a cooler with ice, with separate headspace portions retained under ambient conditions. Photographs will be taken of individual open soil cores for documentation purposes.

Soil samples collected for VOC analysis will be obtained in accordance with KEMRON SOP # 12-SSP-01. This SOP specifies EPA Method 5035 (e.g., EnCore, Terracore or equivalent) will be followed for VOC soil sample collection at RCRA sites. Soil samples for VOC analysis will be collected and containerized at 2-foot intervals upon collection and cutting open of the core liner. An additional small volume of sample will be collected for in-field headspace VOC determination (using a PID), placed in a small zip-lock bag and set aside for ambient temperature stabilization. Following collection of the VOC sample and headspace fractions, the remainder of the recovered core sample will be measured, photographed, and descriptively logged by the field geologist. Following this, the 2-foot interval will be removed from the core liner, placed into a stainless steel bowl, homogenized, then samples will be collected for potential analysis of RCRA metals, Al and Zn.

Soil samples will be analyzed for VOC (EPA Method 8260) and RCRA metals, Al and Zn (EPA Method 6010 and/or 6020). Two (2) soil samples per soil boring will be retained and submitted for laboratory analysis. Additional soil samples may be submitted based on field observation of soil samples, and/or field screening results.

Borings SB-2, SB-6 and SB-9 will have samples collected within the cap material. Cap material thickness will be described. The cap material samples will be analyzed for RCRA metals, Al and Zn, VOCs and grain size (ASTM Method D-422). A decision on the need for permeability testing of the cap material will be made after laboratory analytical results of the soil borings are received and evaluated. If there COPC concentrations beneath the lagoons do not exceed relevant leaching values, no permeability testing will be performed. If COPC concentrations beneath the lagoons exceed the relevant leaching values, additional samples of the cap material will be collected for permeability testing (ASTM Method D-5084) and leakage through the cap(s) will be estimated using USEPA's HELP model (or other model with prior USEPA et al. approval). Please see the RFI QAPP (Attachment B) for further information regarding permeability testing methodology.

For lagoon soil borings, a minimum of two soil samples will be collected from 1) native material immediately below the lagoon backfill and 2) native material immediately above the water table. Additional samples beneath the surface cap material may be submitted for analysis based on the field-recorded PID reading. Field headspace screening of soil samples will be conducted in accordance with SOP# 12-PID-AKMW. Headspace screening results will be recorded on the field soil boring log. Anomalous headspace results, if detected, will be used to determine if one or more additional samples are submitted for laboratory analysis.

For soil borings outside of the lagoons, a minimum of two soil samples will be collected. One sample will be the native material immediately above the water table while the second sample will be selected based on the field-recorded PID reading and/or other evidence of contamination (e.g., staining).

Following completion of field headspace testing and final selection of samples for laboratory analysis, remaining soil sample material will be properly containerized, managed and disposed off-site as IDW. IDW will be temporarily staged on AK Steel secure property (location to be determined) designated for IDW temporary storage prior to disposal.

Open soil borings will be backfilled with granular bentonite and periodically hydrated during and after filling to the ground surface to seal the boreholes (per Chapter 9 the OEPA Technical Guidance Manual for Groundwater Investigations). The ground surface will be patched with soil. Individual soil boring GPS locations will be measured with portable GPS for mapping location determination. Existing monitoring wells will also be GPS-located for mapping reference.

At the present time, no vapor intrusion pathway sampling and analysis is planned. Should VOCs be detected in soil or groundwater samples associated with SWMU 23 during the RFI, the concentrations of the detections will be evaluated and a determination will be made as to whether sampling and analysis of groundwater and/or soil for a potential vapor intrusion pathway is appropriate

4.2 Additional Areas AA02 through AA09 Sampling

Proposed environmental sample collections within the AAs are illustrated on Figures 13-20, and will involve the completion of soil borings within the property boundary of each of the designated AAs. The majority of soil borings will be completed at the Dicks Creek end of each property, and the actual number of completed borings on each property is dependent on the size of the property. Recent photographs of the AAs are presented in Attachment
F.

As with the SWMU 23 drilling investigation, soil borings completed on the AAs properties will be completed using push-tube or hollow rotosonic drilling technology. Subsurface soil sampling will utilize continuous soil sampling completed to a depth at which native material or bedrock is encountered, whichever occurs first. Surface cover materials such as coarse gravel, concrete, vegetative cover or other debris will not be sampled as part of the RFI. Removal of surface materials may be necessary prior to initiation of sampling at a soil boring. Additionally, utility surveys will be required on each parcel, and access agreements must be executed prior to any sample location marking or soil boring installation. The proposed location of borings will be finalized after access agreements are in place and utilities are marked. Limitations imposed by the property owners regarding soil boring locations, as well as engineering limitations encountered on-site (utilities, structures, etc.) may result in a reduction in the feasibility of installing the soil borings as currently depicted in Figures 13-20. However, if such limitations are encountered, the boring location will be adjusted as necessary to assure complete delineation of PCBs in soil.

The primary drilling and sampling objective on the AAs will be to determine the presence and thickness of potential dredged Dicks Creek sediment material used as fill for site grading or filling. Soil boring completion depths will be terminated when original soil horizons/root zones are encountered. Soil samples will be logged on a continuous basis with photographs taken of individual open soil cores for documentation purposes. PID headspace screening will be conducted on collected samples as described in SOP# 12-PID-AKMW. Based on the COPCs being limited to PCBs, and the absence of any PCB source that could result in non-aqueous phase liquids at the sites, no monitoring well installations are currently planned for completion for this portion of the RFI.

Soil samples will be retained at continuous 1-foot sample intervals for logging purposes and for potential analytical sample selection. Detailed soil boring logs will be generated by the field geologist at each location. Field observations of recovered soil samples will note in detail changing soil characteristics with depth. The intent is to be able to differentiate surficial fill (non-dredged fill materials), from potential dredged fill material, from original native materials. Individual soil boring conditions and observations will dictate the depth of samples submitted for analysis from each boring, with a minimum of 4 samples submitted for analysis per boring (or fewer if sediment deposit depth is less than 3 feet below ground surface).

The number of samples submitted for analysis from each borehole will be based on the thickness of the sediment in the borehole, visual observations, PID readings and analytical results. For this assessment, it is anticipated that (at a minimum) the following soil samples will be submitted from each boring for PCB analysis:

- Soil intervals 0-1' bgs, 1-2' bgs and 2-3' bgs; and
- Subsurface 1 ft. interval at base of sediment deposit (above native material horizon, if definable in field and if not included in 0-3' intervals).

Additional samples will be collected and analysis will proceed if necessary based on the following decision criteria:

- Top of native material sample (below sediment deposit interface, and if not included in 0-3' interval) will be collected. Laboratory analysis will be conducted if analytical results from the base of sediment sample (see above) exceed the relevant soil leaching to groundwater screening level(s);
- Subsurface soil below 3' depth and base of sediment sampling interval will be biased to any visual observations and field measurements potentially indicating presence of PCBs (such as oily materials/sheen, discoloration, elevated PID readings, etc.);
- For borings that meet or exceed ten (10) feet in depth, if analytical results indicate that either the 2-3' bgs interval or base of sediment deposit interval exceed the relevant RSL for COPCs, at least one mid-depth sample will be analyzed from that soil boring. Decisions regarding the number of mid-depth samples to be analyzed from borings less than ten (feet) in depth will be based upon the thickness of the sediment observed at each location, the PID readings and visual observations and the COPC concentrations determined to be present at the 2-3' and base of sediment intervals as compared to ESLs and/or RSLs.

Historical interview information (Call record regarding Miami Conservancy memorandum, September 2005) indicates the potential for dredged spoils to have been placed on each of the AAs, with focus on the portion of the properties closest to Dicks Creek. For AA03 (Sturgell parcel), the call record suggests that material from the creek was spread throughout the parcel, especially on the eastern side along the railroad tracks. As this information suggests the possibility of more widespread placement of fill material, a larger number of investigative soil borings is planned for this property to provide a distribution of sample points throughout the property. Additionally, to minimize disruption to residents on both AA03 and AA04, and since no existing chemical data provides information on PCB concentrations for these parcels, sampling is planned for both parcels to reduce the likelihood of multiple mobilizations on the parcels for completion of PCB constituent delineation.

It is possible that shallow bedrock may be present beneath one or more of the AAs as possible bedrock outcropping in adjacent creek bank exposures has been observed near some of the AAs (refer to photos in Attachment F). No drilling into or sampling of bedrock will be conducted in this RFI, since no PCB source would be anticipated in bedrock. The source of potential PCB contamination is former sediment dredged from the stream and possibly placed on the parcels; therefore, there is no reasonable suspicion of PCBs being contained in bedrock.

Proposed sample locations will be marked in the field. Following all utility notification and marking, and identification of any features on the property that may limit conduct of sampling (such as trees, home features, etc.) the final sample locations will be determined and borings will be installed. The soil borings will be logged in the field, and samples collected and managed as described above.

Soil samples will be analyzed for PCB arochlors via EPA Method 8082. Samples will be collected continuously as described above from the uppermost soil intervals to the base of the boring, concluding with a sample of native material. Soil samples which appear to contain sediment characteristics (fine-grained soils) will be the focus of sampling and laboratory analysis. Soil samples which appear to define an interface between possible fill soil and underlying native soil (undisturbed soil horizon) will also be retained for analysis (from above and below this horizon). An attempt will be made to penetrate into native soil/clay at least two feet. If a boring reaches bedrock no further subsurface penetration will be necessary at the AAs.

USEPA RSLs and/or ESLs established for PCB aroclors, as incorporated into the approved CCR, will be used for screening of AAs sample results, and determination of the need for additional samples to delineate the horizontal extent of PCB concentrations. Soil borings will be installed on approximately seventy-five (75) foot centers on each AA. As illustrated in the figures, fewer of the total anticipated sample locations on residential AAs (AA03 and AA04) are illustrated as contingency samples; this will allow less disturbance to residents. On other AAs where property owners do not reside at the parcel, initial sample collection is focused on the portion of the parcel closest to Dicks Creek, where available information indicates material from the creek was most likely placed as fill, and considering prior sampling locations during Interim Measures delineation investigations. The proposed boring locations on Figures 13 through 20 are based on currently available information, including existing analytical results, historic interview records and aerial photographs. These locations will be sampled to the extent feasible based on the presence of buildings, utilities or other engineering limitations present on each parcel, and any limitations imposed by property owners via final access agreements to be acquired. Final sampling locations will be selected and marked in the field based on the above limitations. If necessary, additional step-out sampling will be conducted to provide complete delineation of PCBs.

In order to minimize the time required on parcels with full time residents, laboratory analysis of initial samples collected in the upper three (3) feet of soil will be conducted on a 24 hour turnaround time to determine if additional samples are needed for horizontal delineation. Deeper samples collected at these locations may be analyzed on a standard turnaround. If the 24 hour turnaround results indicate PCB arochlor concentrations greater than the relevant RSLs and/or ESLs are detected, additional samples will be collected at the contingency step-out locations as illustrated on Figures 13-20. Use of contingency sampling locations identified on the figures and additional step-outs as necessary, as described in the RFI DQOs, will assure that PCB concentrations are fully delineated.

If a future remobilization can occur on parcels without excessive limitations or restrictions, short term turnaround time may not be deemed necessary as a second mobilization could be conducted if determined necessary to define the vertical and horizontal extent of PCB contamination at each Additional Area. We note that any parcel with significant access limitations to overcome may require a multi-phased approach be implemented to complete delineation of the nature and extent of PCB concentrations.

Vertical delineation will be deemed complete when native soil, clay or bedrock is encountered, whichever occurs first. If refusal occurs before reaching identifiable native material/bedrock, an attempt will be made to offset to another boring location within approximately three lateral feet of the first attempted location, assuming no engineering limitations are present to prevent such offset. This approach will assure delineation of PCB concentrations in non-native fill material (e.g., sediment material placed on the parcels on each parcel.

Following completion of field logging and final selection of samples for laboratory analysis, remaining soil sample material will be managed and disposed off-site as IDW. Open soil borings will be backfilled with granular bentonite to the ground surface to seal the boreholes. The ground surface will be patched with clean topsoil brought to the site in areas that are vegetated; areas that are covered with aggregate or concrete will be patched as agreed with the property owner. Individual soil boring locations will be GPS-located for mapping location documentation.

While groundwater is included in the SCM for the AAs, the first phase of the RFI will be limited to soil data collection. This is based on the existing data that, while limited, illustrates low PCB concentrations in all sampled locations on and immediately adjacent to the AAs. Following evaluation of the PCB data from soil sampling, a decision will be made regarding the need for groundwater sampling and analysis on or immediately adjacent to the AAs. The decisions regarding the need for groundwater data will be made based on concentrations of any detected PCBs, comparison to relevant soil screening values for leaching to groundwater, and technical discussion and consultation with USEPA et. al. The decision point will be based on evaluation of the soil data and an update of the existing SCM.

4.3 Soil Sample Collection

Soil sampling will be performed using a truck-mounted push-tube type drilling machine or a rotosonic rig.

Individual soil samples will be collected after the cores are logged and photographed by the field geologist. In general, for a 4-foot core with 100% recovery, one soil sample for each 2 feet (1 foot for AAs) of core run is placed into a ziplock bag or laboratory sample container and reserved for potential analysis. Collected samples will be placed into a cooler with ice until a final determination is made as to sample selection for laboratory analysis. Sub-samples used for PID headspace screening are generally kept under ambient conditions separate from potential lab samples. It is likely that final selection of the laboratory samples will be made following completion of a boring. Specific methodology for selection of samples for laboratory analysis is defined in the previous sections.

4.4 Sample Labeling

Soil and/or groundwater samples selected for laboratory analysis will be given unique sample identifiers/labels which identify the type of sample collected and the location and/or depth from which it was collected. QA/QC samples are also given unique sample designations for tracking

purposes (see QAPP Attachment B). Field collection records and/or field book notations will be used to fully identify individual samples and the collection methods used in obtaining the sample.

The following sample designation system will be used during this sampling activity:

SWMU23, or AA## = Location identifier
GM# = Monitoring well identifier (existing SWMU 23 wells)
SB# = Soil boring, followed with number 1, etc.
(0-2) = Sample depth in feet
A = Analytical sample
B = Field duplicate sample (QA sample)
C = Equipment blank sample (QA sample)
Examples of sample label nomenclature, include:

SWMU23-SB1(0-2)A indicates a SWMU23 soil sample was collected at soil boring SB 1 at a depth of 0-2 feet bgs.

SWMU23-GM1SA indicates a groundwater sample obtained from well GM-1S.

AA05-SB2(0-1)C indicates that an equipment blank sample was collected following collection of the 0-1 foot depth sample at boring SB2 at site AA05. The field notebook and/or sample collection record will indicate the specific nature of this collected sample (ie., lab provided DI water was used/poured over/through the decontaminated hand auger directly into the provided sample containers, etc.).

KEMRON will use individual sample collection records at each sampling location. The sample collection record will identify the sampling location by sample number and site address/property name. A description of the collected sample core will also be logged (geologic description of soil type) on the collection record. The form will also indicate the core interval selected for laboratory analysis, and other pertinent information, such as weather conditions, date/time of collection, on-site and immediate surrounding land use, etc.

4.5 Analytical Testing

Laboratory analysis will be conducted by TestAmerica analytical laboratory. This laboratory has been approved for analytical testing of environmental samples for other Consent Decree related work, including Dicks Creek Interim Measures and SWMU 38 and 39 RFI field work. If necessary, another laboratory may be selected in the future, subject to approval through a revision or addendum to the SWMU 23 and AAs RFI QAPP. The analytical laboratory will provide the appropriate sample containers for each required analysis for each sampling location. Sampling technicians will collect and place the individual samples into appropriate labeled bottles. Sample bottles will be placed into Ziploc bags and placed into a cooler with ice for preservation in the field. Sample coolers will be maintained under direct custody of KEMRON until the samples are returned to the laboratory for analysis. If samples are shipped to the laboratory using an overnight commercial delivery service, individual samples and sample coolers will be appropriately packaged for shipment in accordance with SOP # 07-PAS-00.

Collected soil and/or groundwater samples will be analyzed for PCB aroclors for the AAs 02 through 09, and for VOCs and RCRA metals, Al and Zn at SWMU 23, as detailed in the RFI QAPP, Revision 1 (KEMRON, 2013). At a minimum, a standard analytical turnaround time of 10-14 days will be requested of the laboratory. Analytical results for the collected environmental

samples will be reviewed and validated per the RFI QAPP. As described above, a more rapid turnaround time of sample analysis is anticipated for making decisions in the field regarding the need for additional samples on parcels that are currently residential land use, and potentially on other parcels, depending upon site-specific limitations that currently are not known. AAs AA03 and AA04 are the two properties in residential land use status, and thus will be the starting point of the RFI field effort and the focus areas for rapid turnaround on analytical results.

4.6 Geotechnical Testing

Laboratory analysis will be conducted by Terracon laboratory (former HC Nutting laboratory). This laboratory has been approved for geotechnical testing of samples for other Consent Decree related work, including SWMU 38 and 39 RFI field work. If necessary, another laboratory may be selected in the future, subject to approval through a revision or addendum to the SWMU 23 and AAs RFI QAPP. The analytical laboratory will provide the appropriate sample containers for each required analysis for each sampling location. Sampling technicians will collect and place the individual samples into appropriate labeled bottles

5.0 RFI Report

Following completion of the RFI for SWMU 23 and AAs AA02 through AA09, and submittal and approval of a RAAR, an RFI Report which incorporates the results of the RFI investigations will be prepared and submitted for review and approval. The RFI Report will be based on the information and data from the environmental media sampled and analyzed during the RFI together with the historical analytical results for groundwater monitoring wells at/near SWMU 23. Historical groundwater data at/near SWMU 23 as discussed in section 2.2.2 above is deemed usable for this RFI as stated in section 3.1.2.2.1 of the CCR. Additionally, previous analytical data from the Dicks Creek floodplain (Environ August 2005, December 2006 and March 2008) and sediment (Environ August 2006) delineation reports were determined to be usable in Section 3.1.2.1 of the CCR. Sufficient data will be available to:

1) Characterize the nature and extent of contamination both within and migrating beyond the boundaries of each of the subject parcels of the RFI; and,

2) Support an assessment of risk to human health and the environment posed by any identified releases of constituents of potential concern and consistent with the approach presented in the approved RAAR.

In addition, the RFI Report will provide an adequate basis for decisions to be made regarding the need for a CMS, or other response actions as appropriate. The RFI Report will include AK Steel's recommendation regarding the need for a CMS to evaluate any potential corrective measures or potential remedial alternatives to address site conditions.

The RFI Report will include analysis of the RFI data, risk assessment and analysis, and will be the basis for determining if a CMS is required, as described in the following subsections.

The RFI for SWMU 23 will be based on the current and anticipated future land use of the property as industrial. The currently planned soil borings and new well installations at SWMU 23 will be installed on AK Steel owned property. Application of industrial land use standards will require application of an environmental covenant to the AK Steel parcel, as specified in Ohio Administrative Code §§ 5301.88 through 5301.92.

The AAs are not owned by AK Steel. While multiple parcels have been in industrial or commercial use for many years, AK Steel will investigate each parcel to present both an unrestricted and an industrial/commercial future use scenario for RFI purposes. While an industrial/commercial scenario may be considered and presented for each AA as part of the human health risk assessments, no COPCs will be screened out based upon current land use of any of the AAs; all human health risk assessment will be conducted based upon unrestricted land use for those parcels not owned by AK Steel.

5.1 Data Analysis

The RFI Report will present an analysis of all existing and investigation data, and additional information from the CCR that is relevant to the RFI. A detailed description of the source(s), type(s), and extent of contamination and the migration pathways at SWMU 23 and each Additional Area will be presented. The RFI Report will describe the extent of contamination in relation to background levels indicative of the area as well as in relation to relevant Media Cleanup Standards.

5.2 Analysis of Risk

The RFI Report will include assessment of human health and ecological risks under reasonable maximum exposure conditions for both current and reasonably expected future land use scenarios. Risk assessment methodologies will be based upon current USEPA risk assessment policy and guidance.

The risk assessments will discuss any significant areas of uncertainty that may affect the assessment of risk, the potential impacts of such uncertainties with regard to potential over or under estimation of risk, and the technical steps and approaches implemented to address such uncertainties. At a minimum, the uncertainty discussion will include:

- Identification of key uncertainties and degree of scientific consensus with respect to each;
- Significant data gaps and a discussion of the whether additional data gathering would be expected to significantly affect confidence in the risk assessment results;
- Key science policy judgments or default assumptions used to bridge information gaps and the basis for these judgments or assumptions; and,
- Any significant elements of quantitative uncertainty analysis embedded in the risk estimate.

5.2.1 Human Health Risk Assessment

A Human Health Risk Assessment will be prepared to evaluate potential risks to human health posed by SWMU 23 and each Additional Area (02 through 09). The risk analysis will include the following major steps: (1) data evaluation; (2) exposure assessment; (3) toxicity assessment; and (4) risk characterization.

The exposure assessment will analyze how and to what extent human receptors may be exposed to the COCs based on current and reasonably anticipated future land uses under a scenario in which no action is taken (e.g., a “no cleanup action” scenario). As noted previously, SWMU 23 will be assessed implementing the assumption that the historical and current industrial land use will be the reasonably anticipated future land use; unrestricted land use will not apply. The only exception to this statement will be if the RFI data indicate that COCs have migrated off-site such that residential property may be impacted. If such migration is indicated by RFI data, risk assessment will be conducted to address the potentially affected properties and unrestricted use analysis may be required.

Since the AAs are not owned by AK Steel, human health risk assessment will be consistent with residential use of each parcel. As noted previously, commercial/industrial land uses are also anticipated to be analyzed in the risk assessment for the AAs.

All areas and/or media at SWMU 23 and the AAs where risks exceed a lifetime cancer risk of 1.0E-06 and all areas and/or media with a hazard index of 1 or greater will be identified for additional analysis. For each of these risk or hazard levels, Media Cleanup Standards (MCSs) will be determined to estimate the acceptable chemical concentration. MCSs based on risk assessment will be calculated by rearranging the risk assessment equations to derive media and chemical-specific constituent concentrations based on acceptable risk and hazard goals and expected or potential exposures identified and evaluated in the completed risk assessments. MCSs may also be based on background concentrations if the background-based MCSs are greater than risk-based values.

The human health risk assessment will also evaluate and present cumulative excess lifetime cancer risks of 1.0E-04 and 1.0E-05.

5.2.2 Ecological Risk Assessment

An Ecological Risk Assessment will be prepared to evaluate potential ecological risk posed by SWMU 23 and, if determined necessary based on analytical data comparisons to ESLs, one or more Additional Area (02 through 09). The Ecological Risk Assessment will include the following elements:

- Project scoping, planning and study objectives;
- Conceptual site model;
- Ecological exposure areas;
- COCs, fate and transport, sources of data, and analytical procedures used;
- Potential ecological receptors;
- Assessment and measurement endpoints;
- Stressor-response and exposure profiles; and,
- Risks to assessment endpoints, including risk estimates and adversity evaluations.

The ecological risk analysis will include an evaluation of both current and potential future potential risks to ecosystems based on available transport mechanisms at SWMU 23 and the AAs. The evaluation will be based on the following lines of evidence (where appropriate): (1) chemical sampling in potentially exposed habitats and reference sites; (2) toxicity testing; (3) tissue analyses; (4) biological community assessments; (5) habitat assessments of potentially exposed areas; and (6) assessments of ecological impacts on receptors. The toxicity analysis will be based on impacts at the individual level (e.g., mortality, growth and reproductive impairments) and those occurring at higher levels of biological organization (i.e., at population, community, and ecosystem levels).

5.3 Conclusions and Recommendations

Based on the data analysis and the analyses of human health and ecological risks, the RFI Report will include conclusions and recommendations regarding the potential risks posed by constituents at each site, and the need for any subsequent CMS. A CMS will be prepared and submitted if determined necessary for SWMU 23 and/or any of the AAs 02 through 90 based upon the RFI. If a CMS is required, a CMS Work Plan will be submitted 30 days following EPA final approval of the RFI, as specified in the PMP (ARCADIS, 2012a).

6.0 PROGRESS REPORTING

AK Steel currently submits monthly progress reports for all work conducted under the Consent Decree. These same monthly progress reports will be used to update United States et al. on activities that occurred during each calendar month. As determined necessary and/or upon request of United States et al., periodic conference calls may also be held to discuss the progress of the RFI, including data and other findings as they are generated. Email communications also will be used to inform the United States et al. of any significant information related to the RFI that is determined to be appropriate for distribution outside of the normal monthly reporting schedule.

The public will be kept informed of RFI progress as described in the PIP (Attachment D). AK Steel will maintain an Information Repository to provide information to the public. Additionally, communications with property owners will be as determined and agreed upon via access agreements. The AK Steel hotline also will be maintained, and any questions received regarding the SWMU 23 and/or AAs AA02 through AA09 RFI will be addressed upon receipt via calls to the hotline.

6.1 Monthly Progress Reports

The current monthly progress reports submitted by AK Steel to the United States et al. will be present the RFI progress for each calendar month. These monthly reports include:

- A description of activities performed and estimate of the percentage of the RFI completed;
- Summaries of all findings in the reporting period, including results of any sampling and analysis, pilot studies, or other studies;
- Summaries of any deviations from approved work plans for the RFI during the reporting period and the reasons for such deviations;
- Summaries of all formal contacts with representatives of the local community, public interest groups or State government other than OEPA during the reporting period;
- Summaries of all contacts made regarding access to off-site property;
- Summaries of all problems encountered during the reporting period;
- Actions being taken to rectify problems;
- Changes in relevant personnel during the reporting period; and,
- Projected work for the next reporting period.

Copies of the monthly reports are maintained in the information repository. Copies of laboratory/monitoring data, inspection reports, and contractor reports or other documents providing a description of work performed for AK Steel as part of the RFI may be provided to the United States et al. upon request.

6.2 Information Repository

A public Information Repository was established in the Middletown Public Library to ensure that the public has access to all the project reports, data, and documents regarding the Interim Measures remediation. A similar but separate Information Repository will be established to address the RFI/CMS reports, data, and documents prepared under the Consent Decree. The RFI/CMS Repository will include copies of the Consent Decree and attachments, documents that have been approved by the United States et al., monthly progress reports, correspondence with the regulators, AK Steel fact sheets, AK Steel press releases and Middletown Journal newspaper stories. Additional details on the information repository, and other public information regarding this RFI, are provided in Attachment D.

The address, phone number, and hours of operation for the repository are provided below.

Midpointe Middletown Public Library
125 S. Broad Street
Middletown, OH 45044
513-424-1251
Hours of Operation:
Monday through Friday, 9:00 a.m. – 7:00 p.m.;
Saturday, 9:00 a.m. – 5:00 p.m.;
Sunday, 1:00 p.m. – 5:00 p.m.

The repository is handicapped accessible and contains photocopying capabilities.

7.0 SCHEDULE

The PMP (ARCADIS, 2012a) establishes priorities for conduct of RFI/CMS for the SWMUs, HWMUs and AAs at Middletown Works. SWMU 23 and AAs AA02 through AA09 were selected as high priority sites for conduct of an RFI.

A schedule for the implementation of the SWMU 23 and AAs AA02 through AA09 RFI is included as Figure 11. The schedule includes the completion of access agreements, selection of subcontractors, implementation of field work, laboratory analysis, data analysis, and the preparation of the various subsequent RFI component reports. Milestones included in the schedule include the preparation and submittal of the: (1) RAAR; (2) Environmental Covenant for SWMU 23; and, (3) RFI Report.

It should be noted that the schedule includes assumptions regarding regulatory review of documents. Should review of documents extend beyond the scheduled time frames, the schedule will be adjusted accordingly to extend the following scheduled response submittal and review dates. Additionally, should the RFI work as initially planned not complete the delineation required for the RFI at all locations, additional mobilizations may be required to conduct step-out investigation elements (e.g., additional monitoring wells for SWMU 23, and/or additional soil borings for either SWMU 23), including potential conduct of work on properties that require additional access agreements. This work may require one or more additional phases of investigation, which would be conducted after access agreements to the property(ies) were completed.

The schedule (Figure 11) will be finalized as part of the final issuance of this RFI Work Plan following approval by United States et al.

8.0 REFERENCES

ARCADIS. 2012a. *Project Management Plan, AK Steel Corporation, Middletown Works, Middletown, Ohio.* August 15, 2012.

ARCADIS. 2012b. *Current Conditions Report, AK Steel Corporation, Middletown Works, Middletown, Ohio.* Version 5. May 11, 2012.

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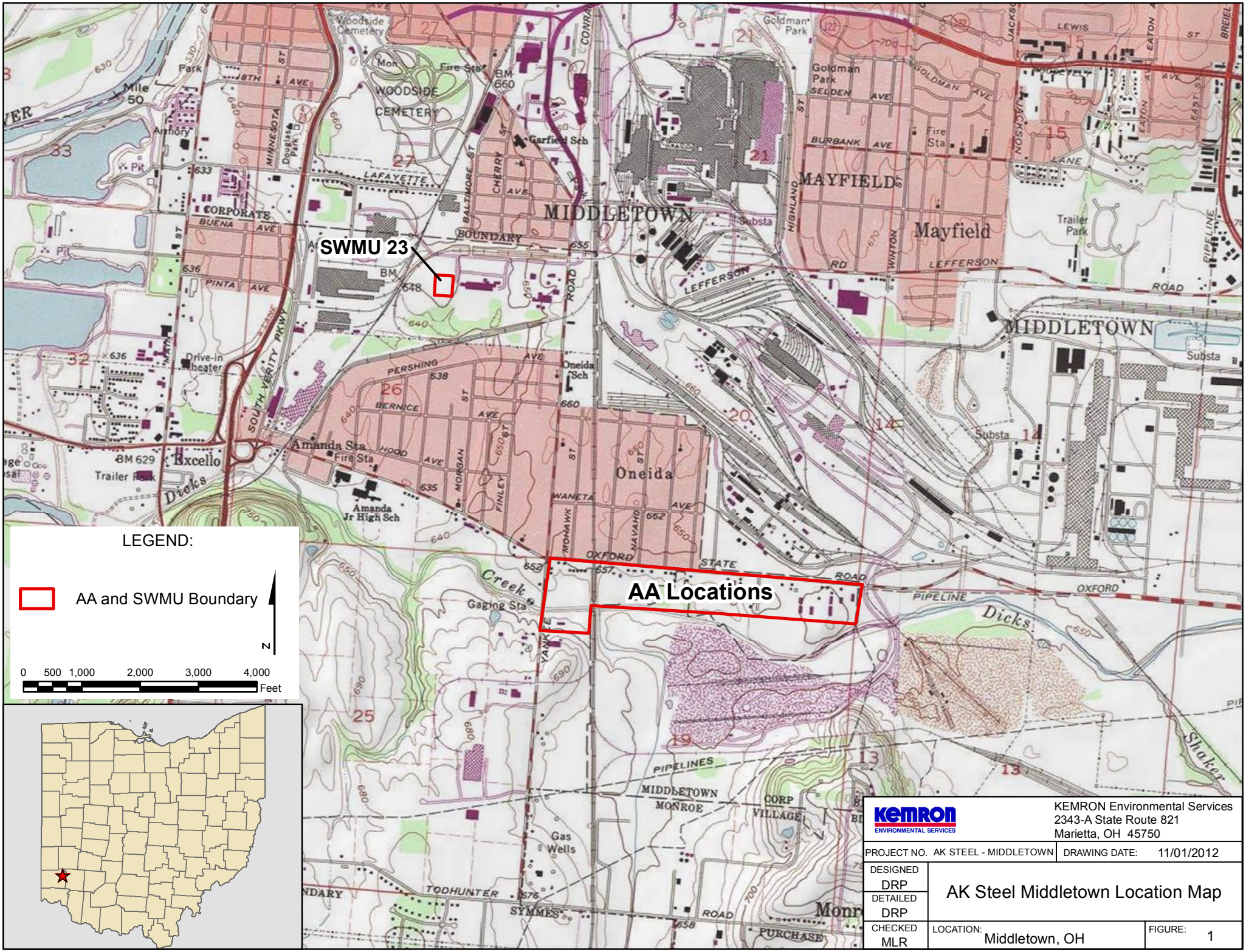
FIGURES



KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO.	AK STEEL - MIDDLETOWN	DRAWING DATE:	11/01/2012
DESIGNED	DRP	AK Steel Middletown Location Map	
DETAILED	DRP		
CHECKED	MLR	LOCATION: Middletown, OH	FIGURE: 1

KEMRON ENVIRONMENTAL SERVICES

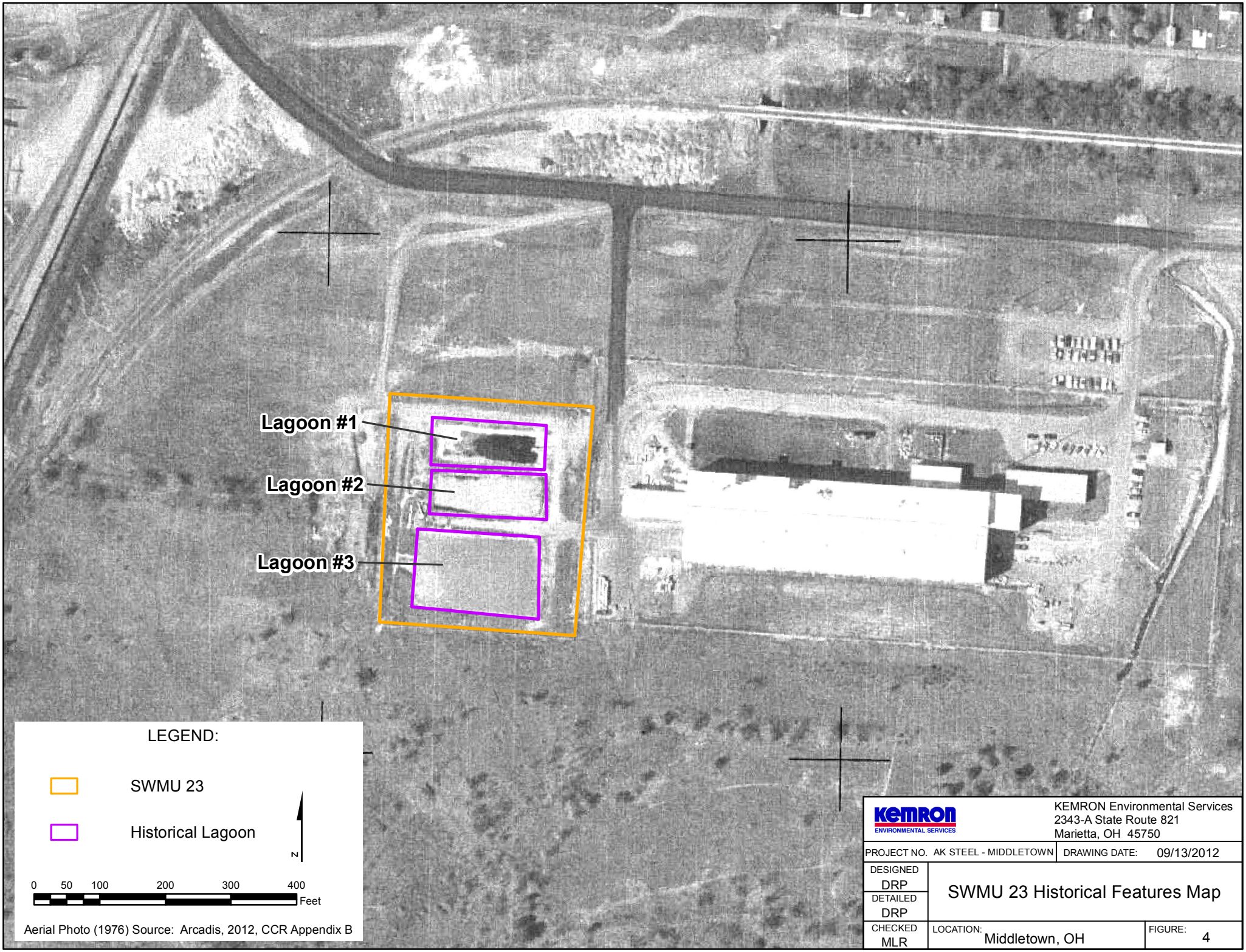
KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO. AK STEEL - MIDDLETOWN	DRAWING DATE: 04/15/2013		
DESIGNED DRP	General Location Map; SWMU 23 and Additional Areas		
DETAILED DRP			
CHECKED MLR	LOCATION: Middletown, OH	REVISION: 1	FIGURE: 2

KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO. AK STEEL - MIDDLETOWN		DRAWING DATE: 09/13/2012	
DESIGNED DRP	SWMU 23 Area		
DETAILED DRP			
CHECKED MLR	LOCATION: Middletown, OH	FIGURE: 3	



KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO. AK STEEL - MIDDLETOWN		DRAWING DATE: 09/13/2012	
DESIGNED DRP	SWMU 23 Historical Features Map		
DETAILED DRP			
CHECKED MLR	LOCATION: Middletown, OH	FIGURE: 4	

KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO. AK STEEL - MIDDLETOWN		DRAWING DATE: 04/11/2013	
DESIGNED DRP	SWMU 23 Existing Features Map		
DETAILED DRP			
CHECKED MLR	LOCATION: Middletown, OH	REVISION: 1	FIGURE: 5

LEGEND:

Parcel Boundary

Additional Area Location

0 100 200 400 600 800 Feet

N

KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO. AK STEEL - MIDDLETOWN		DRAWING DATE: 04/15/2013	
DESIGNED DRP	Additional Areas: AA02 through AA09 Overview Map		
DETAILED DRP			
CHECKED MLR	LOCATION: Middletown, OH	REVISION: 1	FIGURE: 6

AA 02
AA 04
AA 03
AA 07
AA 05
AA 08
AA 09
AA 06

R1BM-01
R1CS-06
R1CS-07
R1EM-02
R1EM-03
R1EM-04A
R1EM-05
R1EM-06
R1EM-07
R1EM-08

LEGEND:

- Historical Soil Boring
- Additional Area Location
- Dicks Creek Remediation Grid

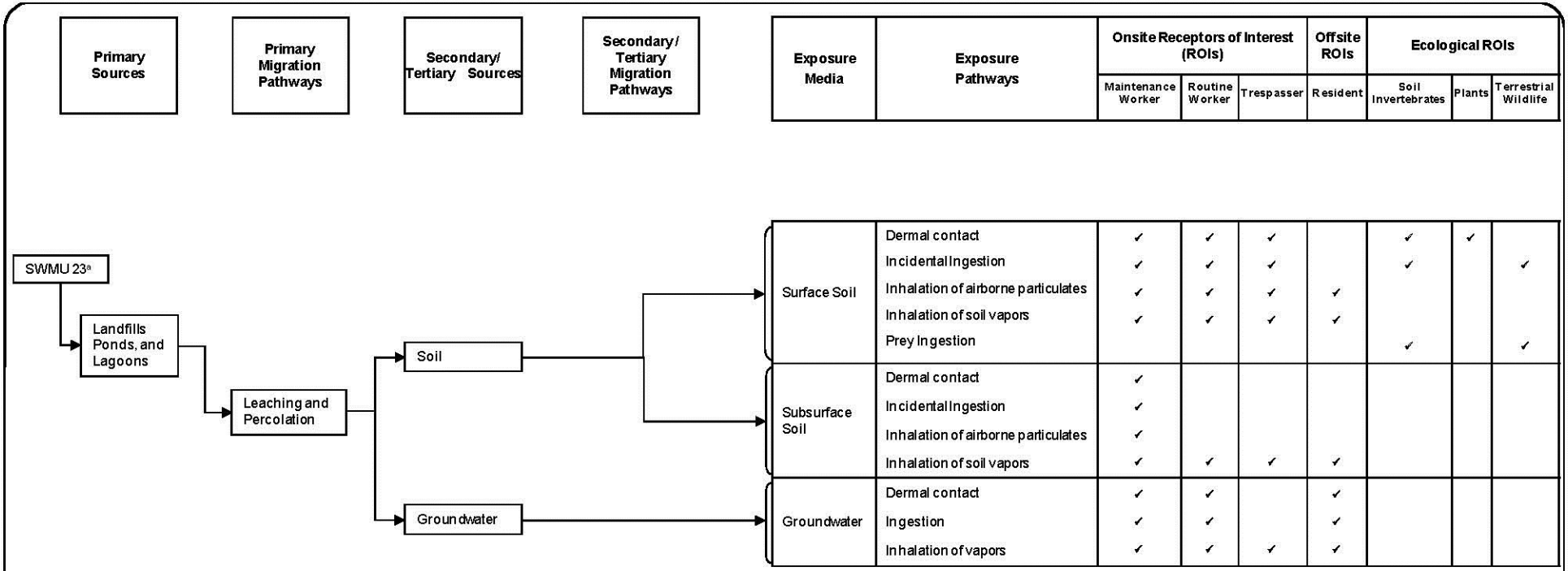
N

0 100 200 400 600 800 Feet

KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO. AK STEEL - MIDDLETOWN		DRAWING DATE: 04/25/2013	
DESIGNED DRP	Additional Areas: Existing Features Map		
DETAILED DRP			
CHECKED MLR	LOCATION: Middletown, OH	REVISION: 1	FIGURE: 7



Exposure Media	Exposure Pathways	Onsite Receptors of Interest (ROIs)			Offsite ROIs	Ecological ROIs		
		Maintenance Worker	Routine Worker	Trespasser	Resident	Soil Invertebrates	Plants	Terrestrial Wildlife
Surface Soil	Dermal contact	✓	✓	✓		✓	✓	
	Incidental Ingestion	✓	✓	✓		✓		✓
	Inhalation of airborne particulates	✓	✓	✓	✓			
	Inhalation of soil vapors	✓	✓	✓	✓			
	Prey Ingestion					✓		✓
Subsurface Soil	Dermal contact	✓						
	Incidental Ingestion	✓						
	Inhalation of airborne particulates	✓						
	Inhalation of soil vapors	✓	✓	✓	✓			
Groundwater	Dermal contact	✓	✓		✓			
	Ingestion	✓	✓		✓			
	Inhalation of vapors	✓	✓	✓	✓			

SWMU 23: Former Coil Paint Wastewater Treatment Lagoons

Notes:

✓ Potentially complete exposure pathway, currently and in the future

a. The adjacent property contains septic tanks and leach fields.

SOURCE: CCR, ARCADIS, 2012

Figure 8 - Preliminary Site Conceptual Model for SWMU 23, AK Steel Corporation, Middletown, Ohio.

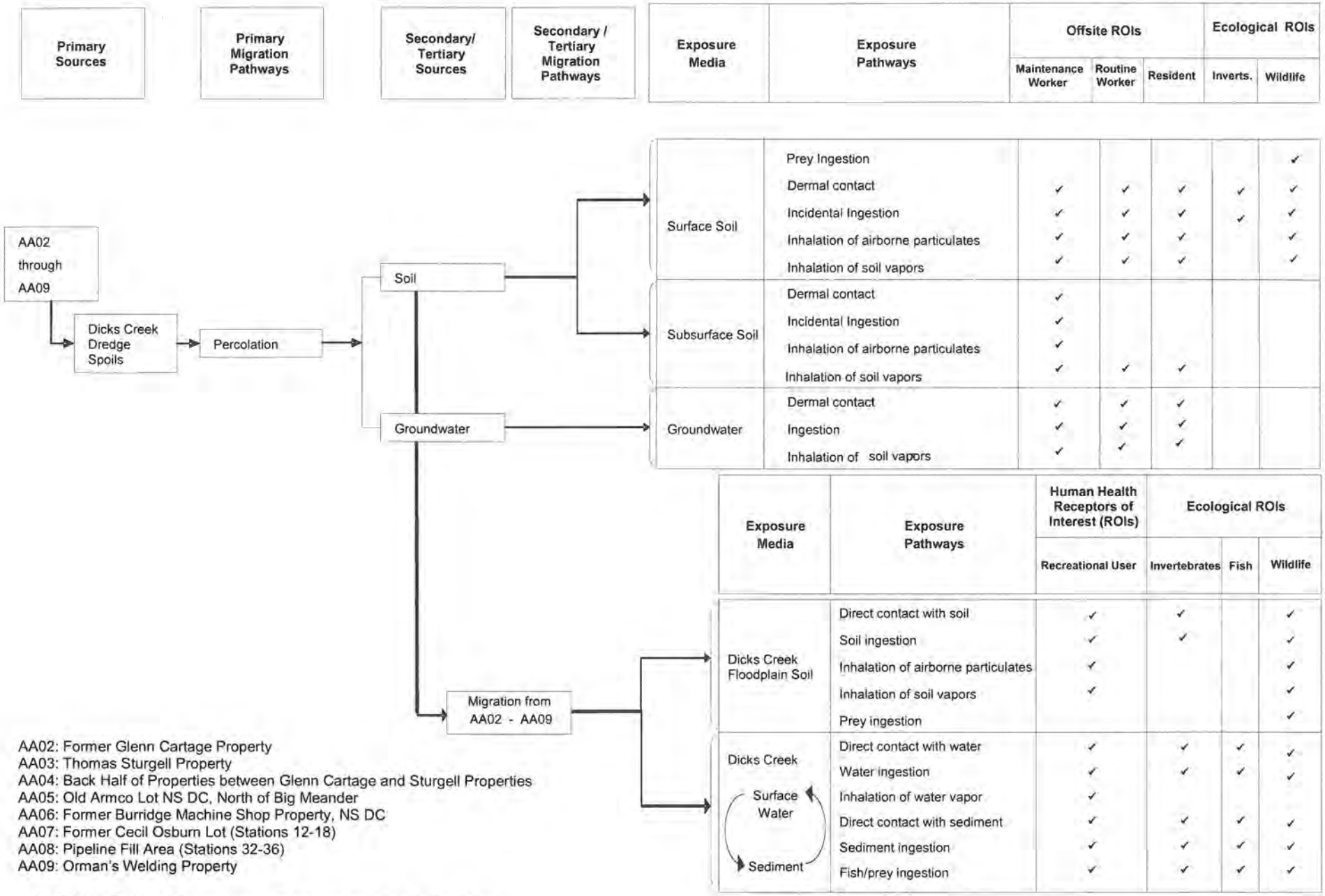
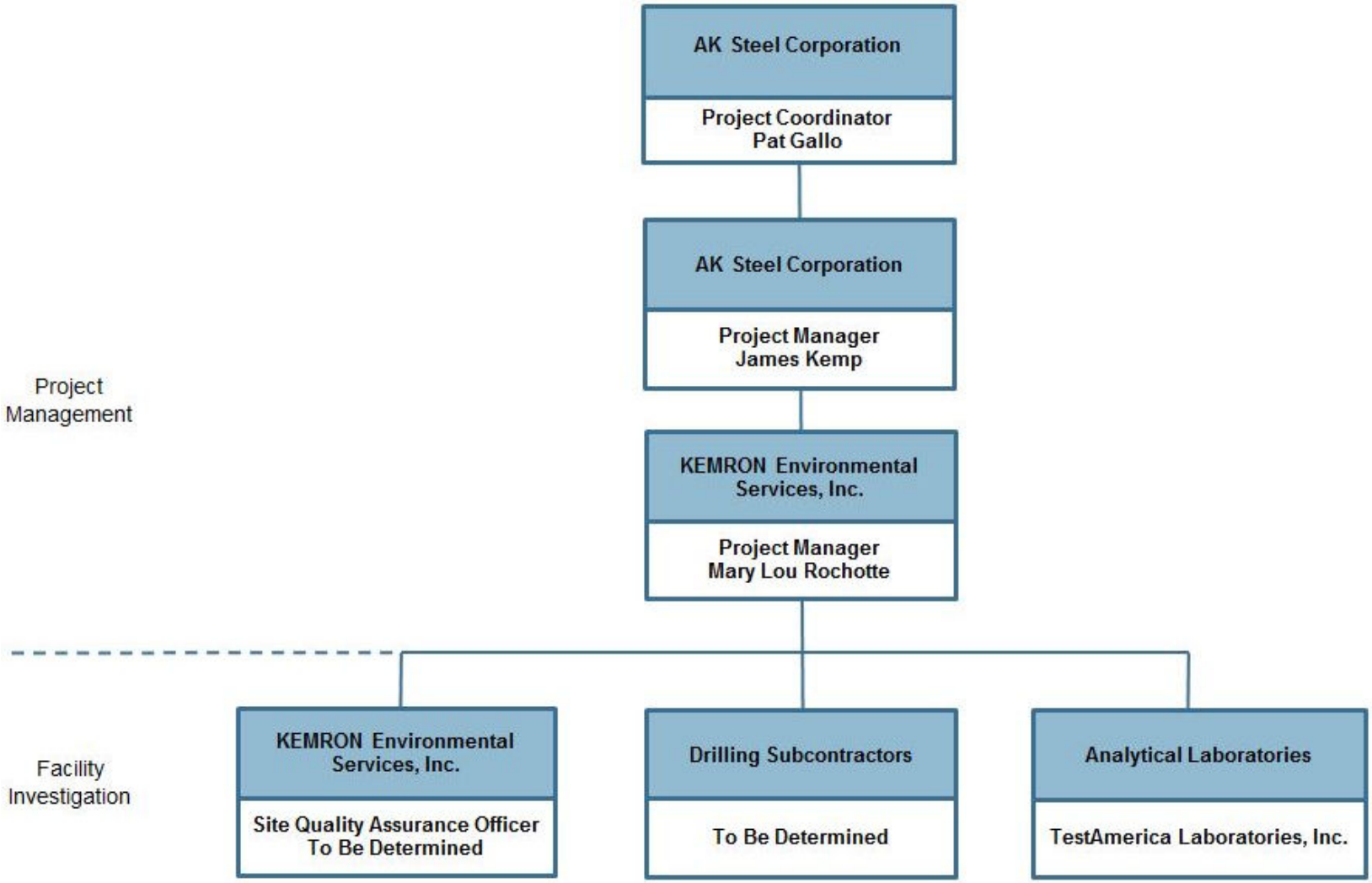


Figure 9 – Preliminary Site Conceptual Model for Additional Areas 02 -09, AK Steel Corporation, Middletown, Ohio.

REVISION: 1 Date: 04/26/2013



KEMRON ENVIRONMENTAL SERVICES	KEMRON Environmental Services 2343-A State Route 821 Marietta, OH 45750	
PROJECT NO. AK STEEL - MIDDLETOWN	DRAWING DATE: 11/01/2012	
DESIGNED DRP	RFI Management Table of Organization	
DETAILED DRP		
CHECKED MLR	LOCATION: Middletown, OH	FIGURE: 10

Task Mode	ID	Task Name	Start	Finish	Duration
	0	**RFI, SWMU 23 & AAs**	**Mon 10/8/12**	**Wed 6/3/15**	**693 days**
	1	**RFI Work Plan, SWMU 23 & AAs**	**Mon 10/8/12**	**Mon 7/29/13**	**211 days**
	2	**Prepare Draft RFI Work Plan, SWMU 23 & AAs and submit to EPA**	**Mon 10/8/12**	**Wed 11/28/12**	**38 days**
	11	EPA Review of RFI Work Plan, SWMU 23 & AAs	Thu 11/29/12	Wed 2/27/13	65 days
	12	Receive EPA Comments	Thu 2/28/13	Thu 2/28/13	1 day
	13	Prepare & Issue Final RFI WP SWMU & 8 AAs	Thu 2/28/13	Fri 4/26/13	57 edays
	14	EPA Conditional Approval of RFI Work Plan, 8 AAs	Fri 4/26/13	Mon 6/3/13	38 edays
	15	Prepare and submit 8 AAs RTCs	Mon 6/3/13	Mon 6/24/13	21 edays
	16	Meeting to confer on SWMU 23 comments	Mon 6/17/13	Mon 6/17/13	1 day
	17	Respond to SWMU 23 meeting comments	Mon 6/17/13	Mon 7/1/13	14 edays
	18	Meeting to confer on SWMU 23 comments	Wed 7/17/13	Wed 7/17/13	1 day
	19	Respond to SWMU 23 meeting comments	Wed 7/17/13	Mon 7/22/13	5 edays
	20	EPA Review of response to comments	Mon 7/22/13	Mon 7/29/13	7 edays
	21	EPA Final Approval of RFI Work Plan, SWMU 23	Mon 7/29/13	Mon 7/29/13	0 edays
	22	**RFI Public Involvement Letter & RFI Fact Sheet**	**Mon 10/8/12**	**Fri 8/2/13**	**214 days**
	23	**Prepare RFI Public Involvement Letter & RFI Fact Sheet and submit to EPA**	**Mon 10/8/12**	**Thu 4/11/13**	**134 days**
	29	EPA Review and Approval of RFI Fact Sheet	Fri 4/12/13	Thu 5/30/13	35 days
	30	Distribute RFI Public Involvement Letter and RFI Fact Sheet	Mon 7/29/13	Fri 8/2/13	4 edays
	31	**RCRA Facility Investigation**	**Mon 7/29/13**	**Fri 3/7/14**	**159 days**
	32	Contractor Bid and Selection	Mon 7/29/13	Sat 10/12/13	75 edays
	33	**SWMU 23 activities**	**Sat 10/12/13**	**Fri 2/14/14**	**89 days**
	34	Mobilize subcontractor	Mon 10/14/1	Tue 10/15/13	2 days
	35	Utility Clearance	Sat 10/12/13	Sat 10/26/13	2 ewks
	36	Well Check	Sat 10/12/13	Wed 10/16/13	4 edays
	37	Install soil borings and monitoring wells, collect soil and groundwater samples	Sat 10/26/13	Sat 11/16/13	3 ewks
	38	Laboratory Analysis	Sat 11/16/13	Mon 12/16/13	30 edays
	39	Data Validation	Mon 12/16/1	Wed 1/15/14	30 edays
	40	Data Management	Wed 1/15/14	Fri 2/14/14	30 edays
	41	**8 AAs activities**	**Tue 7/30/13**	**Fri 3/7/14**	**158 days**
	42	Finalize Access Agreements	Tue 7/30/13	Sun 10/13/13	75 edays
	43	Utility Clearance	Sun 10/13/13	Sun 10/27/13	2 ewks
	44	Drill soil borings	Sat 11/16/13	Sat 12/7/13	3 ewks
	45	Laboratory Analysis	Sat 12/7/13	Mon 1/6/14	30 edays
	46	Data Validation	Mon 1/6/14	Wed 2/5/14	30 edays
	47	Data Management	Wed 2/5/14	Fri 3/7/14	30 edays
	48	Demobilization of subcontractor	Sat 12/7/13	Sun 12/8/13	1 eday
	49	**Risk Assessment Assumption Report**	**Mon 1/6/14**	**Sun 7/6/14**	**130 days**
	50	Prepare Risk Assessment Assumption Report (RAAR)	Mon 1/6/14	Fri 3/7/14	60 edays
	51	Submit RAAR	Fri 3/7/14	Fri 3/7/14	1 day
	52	EPA review of RAAR	Fri 3/7/14	Mon 4/21/14	45 edays
	53	Prepare and issue Final RAAR	Mon 4/21/14	Thu 5/22/14	31 edays
	54	EPA final approval of RAAR	Thu 5/22/14	Sun 7/6/14	45 edays
	55	**RCRA Facility Investigation Report, Risk Assessments, and Environmental Covenant (as necessary)**	**Sun 7/6/14**	**Wed 6/3/15**	**238 days**
	56	Analyze risk and prepare Risk Assessments and Environmental Covenant (as necessary)	Sun 7/6/14	Mon 11/3/14	120 edays
	57	Analyze data and prepare RCRA Facility Investigation (RFI) Report	Sun 7/6/14	Mon 11/3/14	120 edays

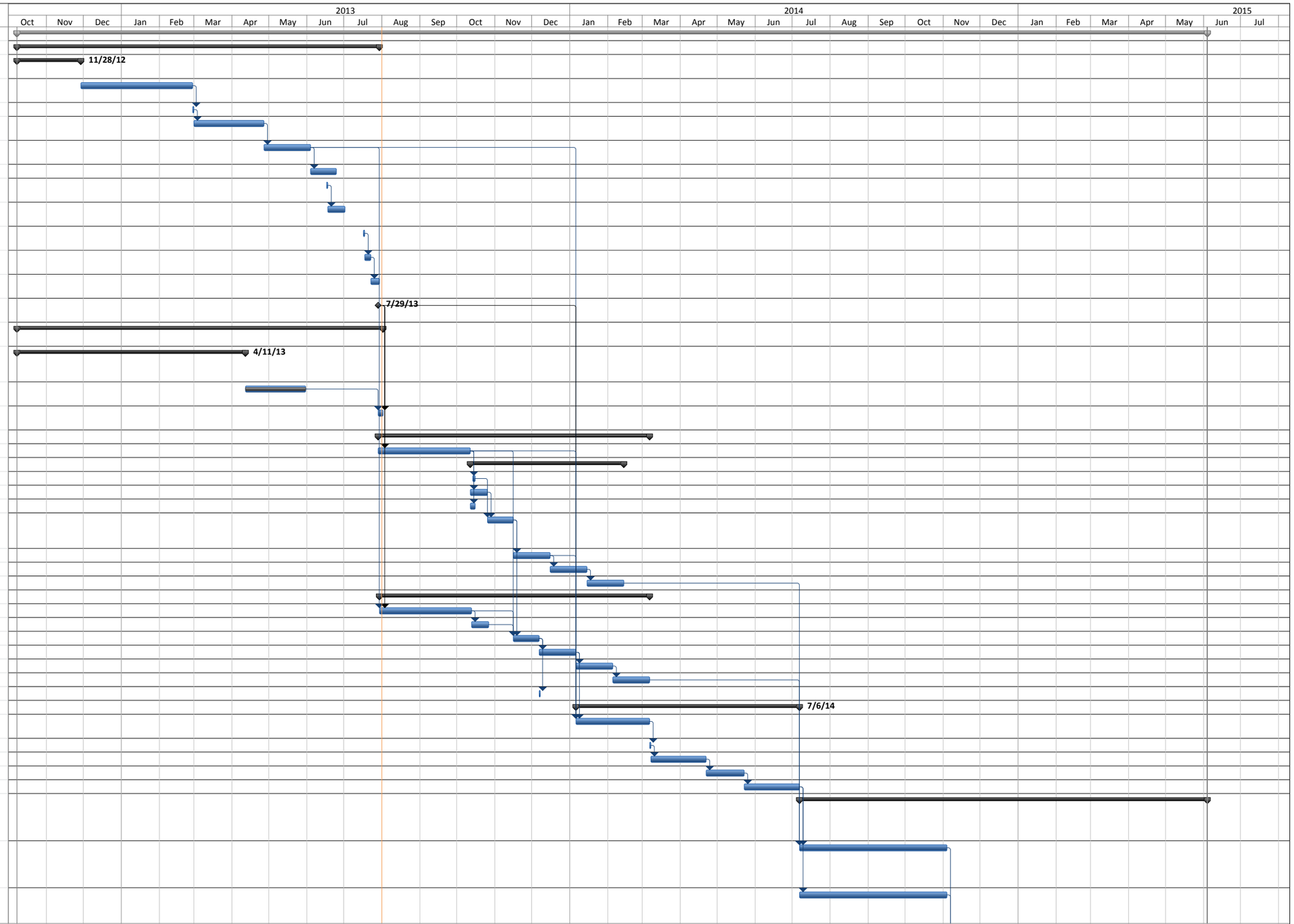


Figure 11
Project: RFI, SWMU 23 & AAs
Date: Thu 8/1/13

Note: Additional comments from EPA will alter schedule

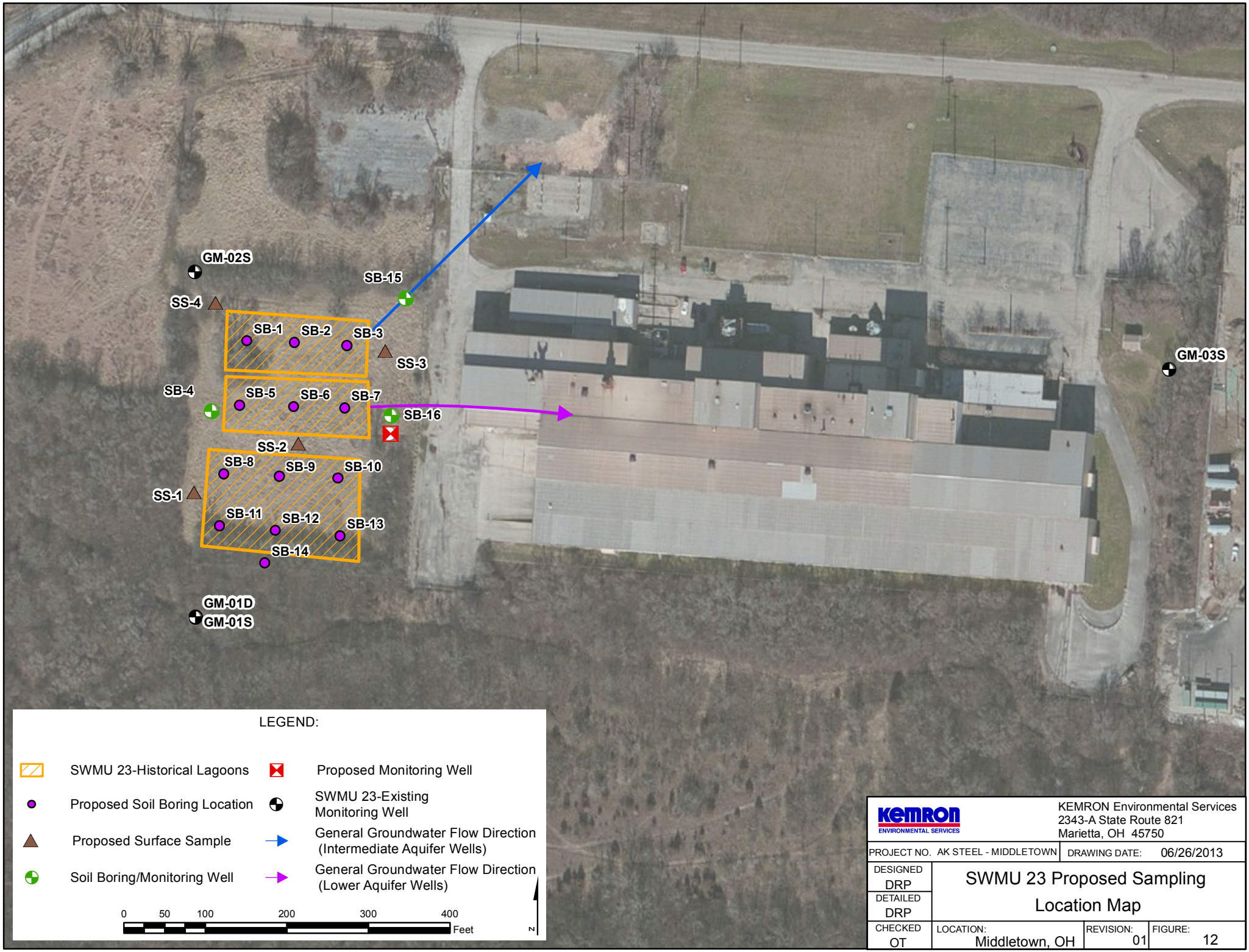
Task	Summary	External Milestone	Inactive Summary	Manual Summary Rollup	Finish-only		
Split	Project Summary	Inactive Task	Manual Task	Manual Summary	Deadline		
Milestone	External Tasks	Inactive Milestone	Duration-only	Start-only	Progress		

Task Mode	ID	Task Name	Start	Finish	Duration
	58	Submit RFI Report, including Risk Assessments and Environmental Covenant (as necessary)	Tue 11/4/14	Tue 11/4/14	1 day
	59	EPA review of RFI Report	Tue 11/4/14	Fri 12/19/14	45 edays
	60	Prepare responses to comments and revise RFI Report	Fri 12/19/14	Mon 2/2/15	45 edays
	61	EPA review of revised RFI Report	Mon 2/2/15	Thu 3/19/15	45 edays
	62	EPA Comments received	Fri 3/20/15	Fri 3/20/15	1 day
	63	Prepare and issue Final RFI Report	Fri 3/20/15	Mon 5/4/15	45 edays
	64	EPA final approval of RFI Report	Mon 5/4/15	Wed 6/3/15	30 edays

Figure 11
Project: RFI, SWMU 23 & AAs
Date: Thu 8/1/13

Note: Additional comments from EPA will alter schedule

Task, Split, Milestone, Summary, Project Summary, External Tasks, External Milestone, Inactive Task, Inactive Milestone, Inactive Summary, Manual Task, Duration-only, Manual Summary Rollup, Manual Summary, Start-only, Finish-only, Deadline, Progress



KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO. AK STEEL - MIDDLETOWN		DRAWING DATE: 06/26/2013	
DESIGNED DRP	SWMU 23 Proposed Sampling Location Map		
DETAILED DRP			
CHECKED OT	LOCATION: Middletown, OH	REVISION: 01	FIGURE: 12

KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO. AK STEEL - MIDDLETOWN		DRAWING DATE: 04/25/2013	
DESIGNED DRP	AA02: Parcel Q6542101000005 GARLAND II LLC Proposed Sampling Locations		
DETAILED DRP			
CHECKED MLR	LOCATION: Middletown, OH	REVISION: 1	FIGURE: 13

KEMRON ENVIRONMENTAL SERVICES

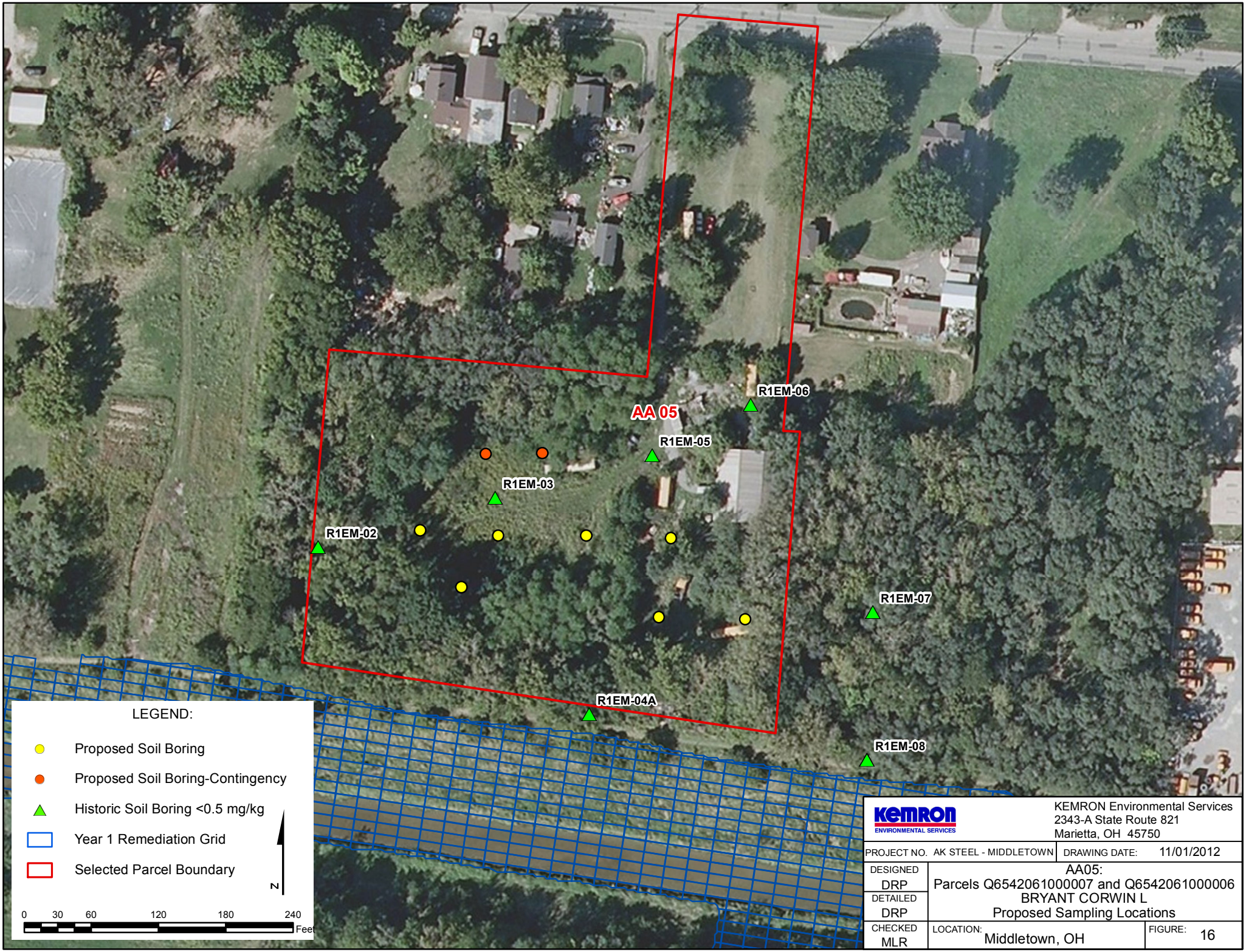
KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO. AK STEEL - MIDDLETOWN		DRAWING DATE: 04/25/2013	
DESIGNED DRP	AA03: Parcels Q6542101000019 and Q6542101000010 STURGELL PAULA Proposed Sampling Locations		
DETAILED DRP			
CHECKED MLR	LOCATION: Middletown, OH	REVISION: 1	FIGURE: 14

KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

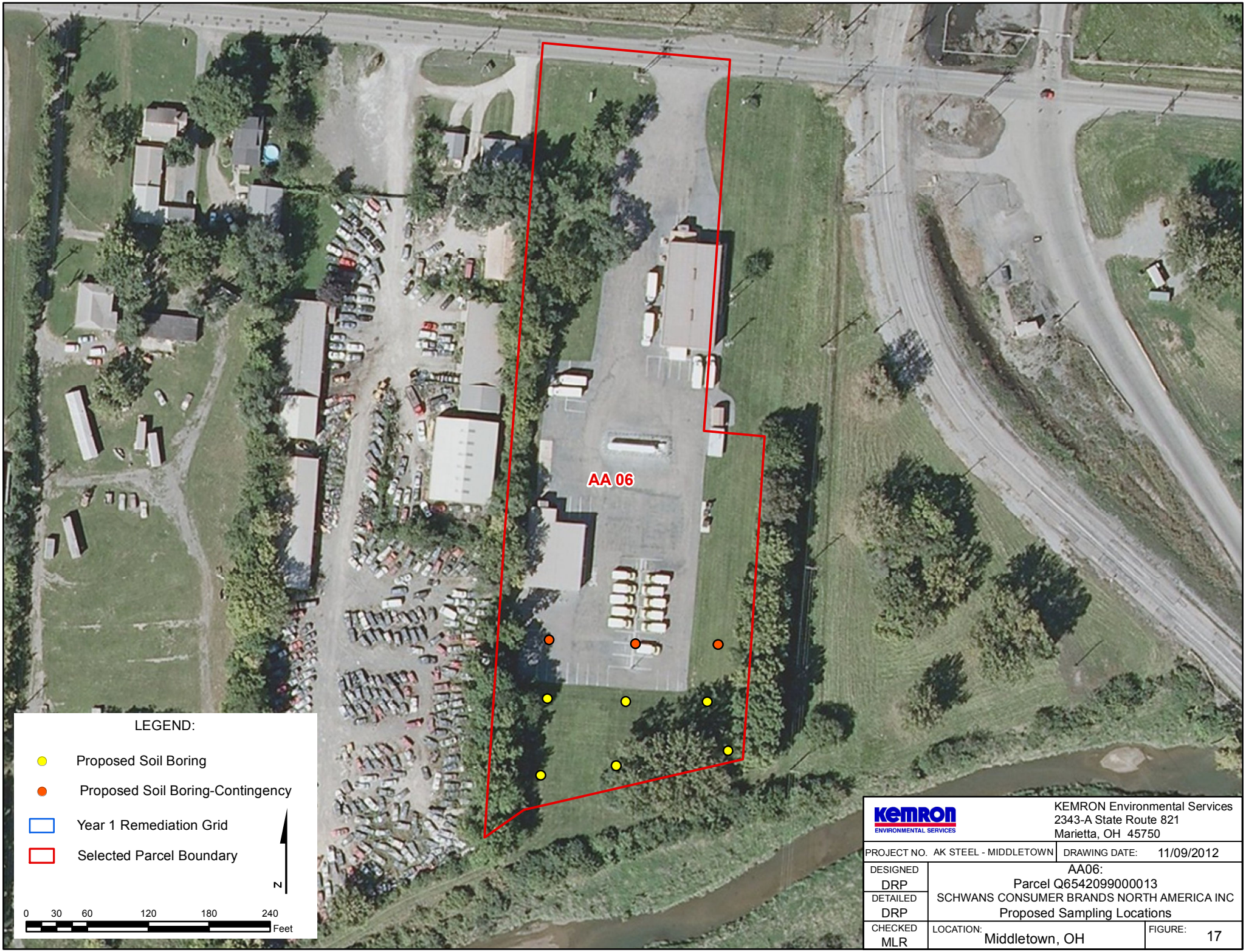
PROJECT NO. AK STEEL - MIDDLETOWN		DRAWING DATE: 04/25/2013	
DESIGNED DRP	AA04: Proposed Sampling Locations		
DETAILED DRP			
CHECKED MLR	LOCATION: Middletown, OH	REVISION: 1	FIGURE: 15



KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

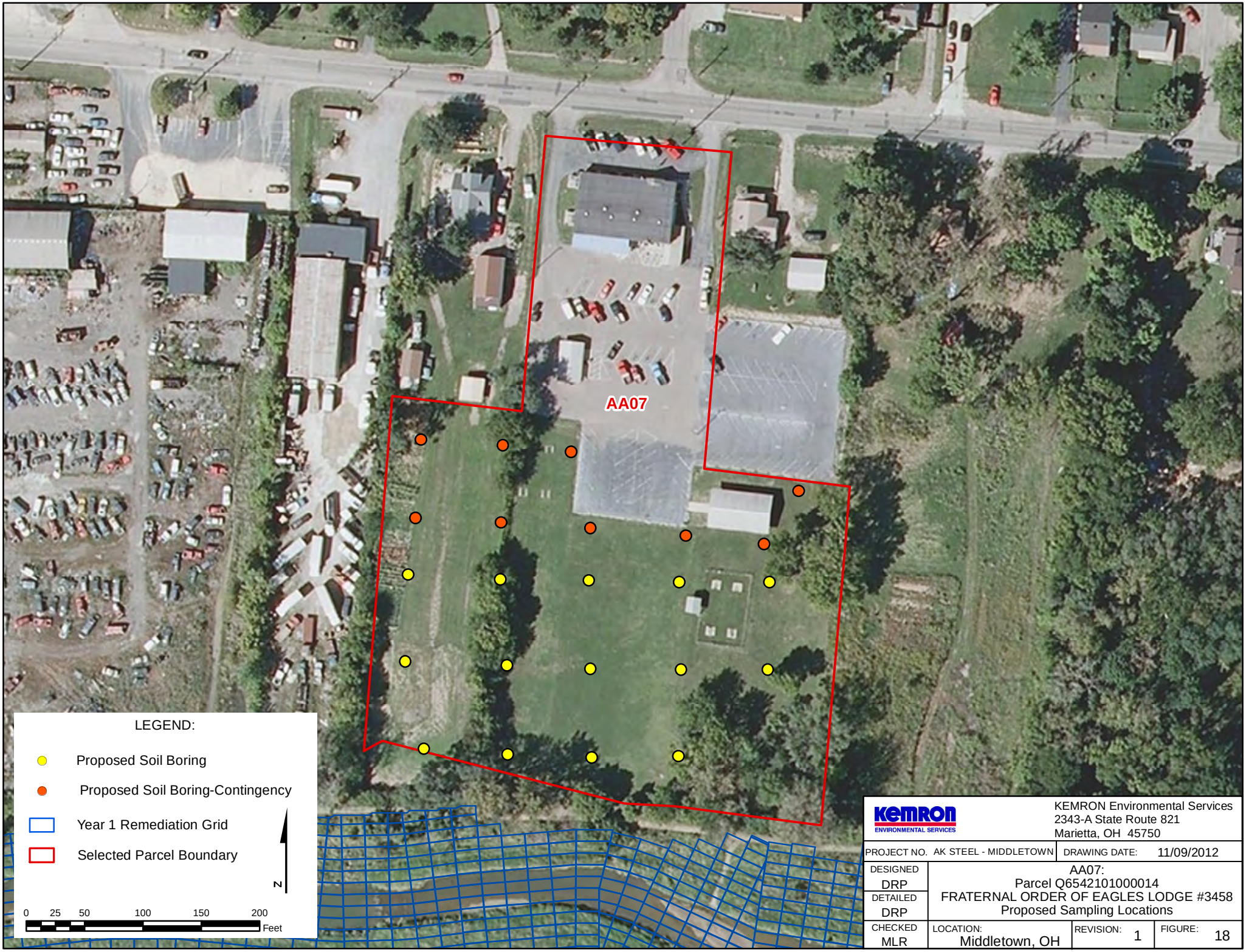
PROJECT NO.	AK STEEL - MIDDLETOWN	DRAWING DATE:	11/01/2012
DESIGNED	DRP	AA05: Parcels Q6542061000007 and Q6542061000006 BRYANT CORWIN L Proposed Sampling Locations	
DETAILED	DRP		
CHECKED	MLR	LOCATION: Middletown, OH	FIGURE: 16



KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

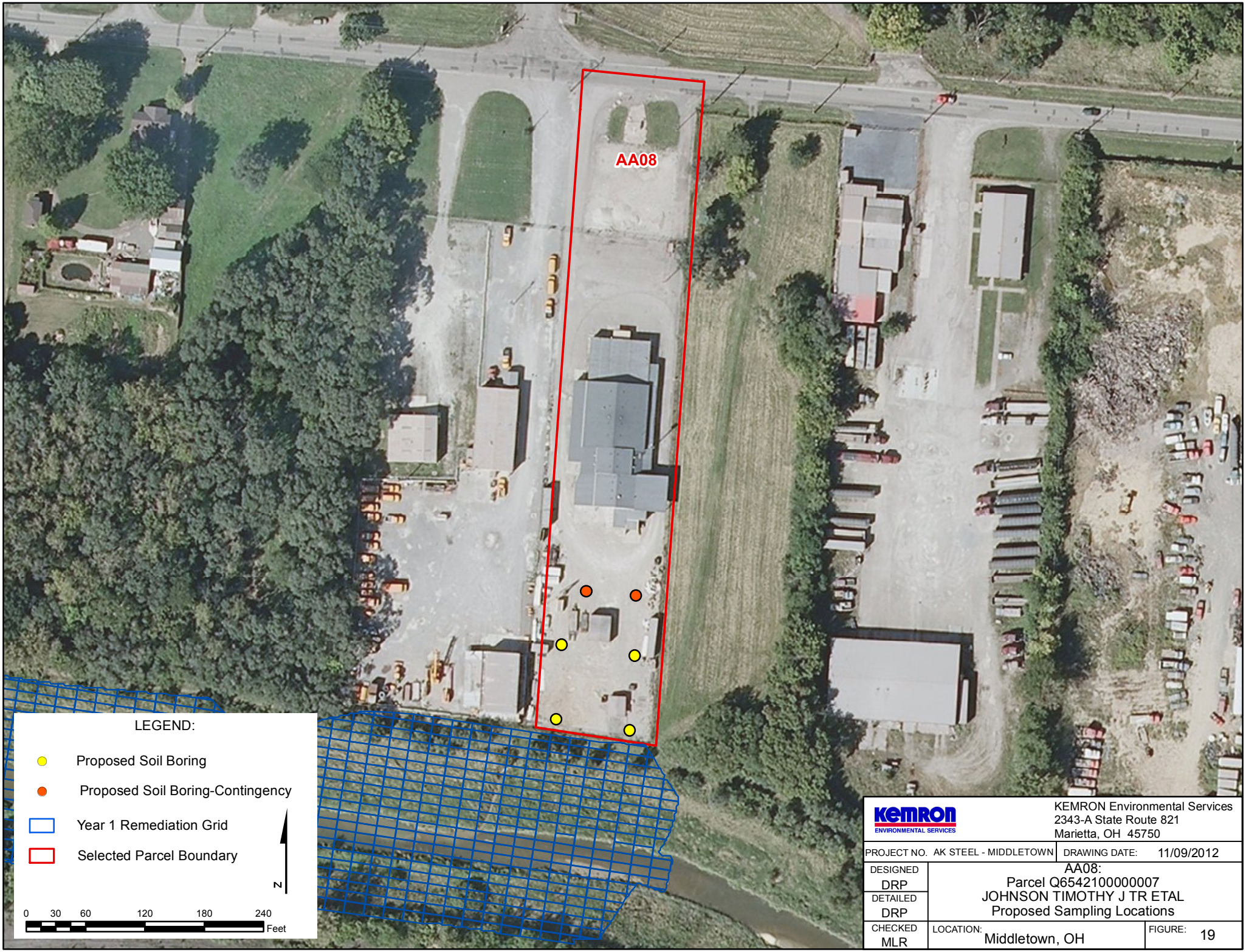
PROJECT NO.	AK STEEL - MIDDLETOWN	DRAWING DATE:	11/09/2012
DESIGNED	DRP	AA06: Parcel Q6542099000013 SCHWANS CONSUMER BRANDS NORTH AMERICA INC Proposed Sampling Locations	
DETAILED	DRP		
CHECKED	MLR	LOCATION: Middletown, OH	FIGURE: 17



KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

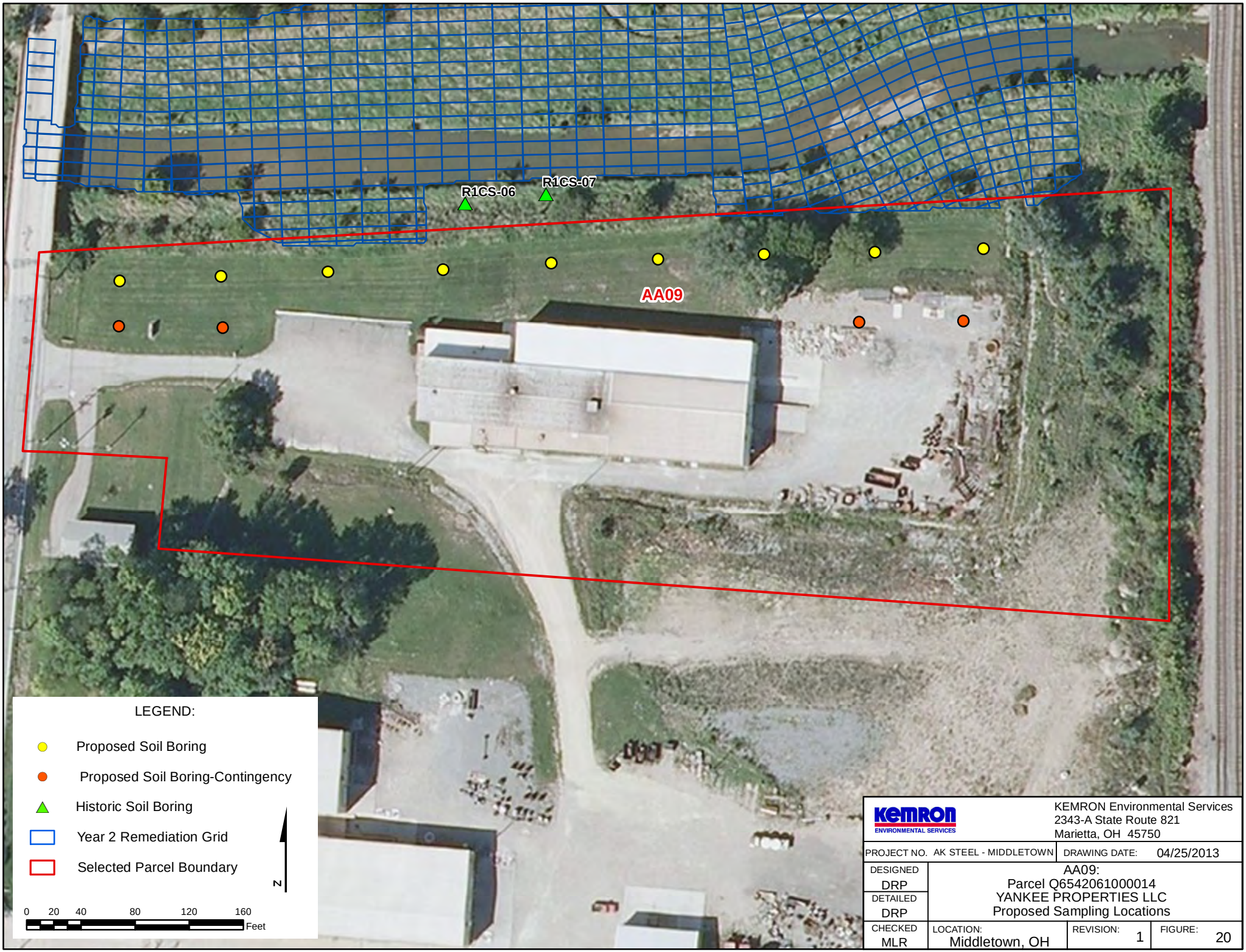
PROJECT NO. AK STEEL - MIDDLETOWN		DRAWING DATE: 11/09/2012	
DESIGNED DRP	AA07: Parcel Q6542101000014 FRATERNAL ORDER OF EAGLES LODGE #3458 Proposed Sampling Locations		
DETAILED DRP			
CHECKED MLR	LOCATION: Middletown, OH	REVISION: 1	FIGURE: 18



KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO. AK STEEL - MIDDLETOWN		DRAWING DATE: 11/09/2012	
DESIGNED DRP	AA08: Parcel Q6542100000007 JOHNSON TIMOTHY J TR ETAL Proposed Sampling Locations		
DETAILED DRP			
CHECKED MLR	LOCATION: Middletown, OH	FIGURE: 19	



KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO. AK STEEL - MIDDLETOWN		DRAWING DATE: 04/25/2013	
DESIGNED DRP	AA09: Parcel Q6542061000014 YANKEE PROPERTIES LLC Proposed Sampling Locations		
DETAILED DRP			
CHECKED MLR	LOCATION: Middletown, OH	REVISION: 1	FIGURE: 20

TABLES

Table 2
SWMU 23 Area Groundwater Monitoring Data Summary

	MCL (ug/L)	5	70[(cis)]	5				5
Well No.	**Date**	**TCE (ug/L)**	**1,2-DCE (ug/L)**	**Benzene (ug/L)**	**Chloroform (ug/L)**	**Carbon Disulfide (ug/L)**	**Acetone (ug/L)**	**Methylene Chloride (ug/L)**
GM-01S	Nov-89	BDL<5	BDL<5[(tr)]	BDL<5	BDL<5	NR	NR	10
GM-01D	Nov-89	BDL<5	BDL<5[(tr)]	BDL<5	BDL<5	NR	NR	BDL<5
GM-02S	Nov-89	10	BDL<5[(tr)]	BDL<5	BDL<5	NR	NR	BDL<5
GM-03S	Nov-89	NS	NS	NS	NS	NS	NS	NS
GM-01S	Mar-90	BDL<5	BDL<5	BDL<5	BDL<5	9	BDL<20	6
GM-01D	Mar-90	BDL<5	BDL<5	BDL<5	BDL<5	5	64	BDL<5
GM-02S	Mar-90	10	BDL<5	BDL<5	BDL<5	8	61	BDL<5
GM-03S	Mar-90	NS	NS	NS	NS	NS	NS	NS
GM-01S	Jun-90	BDL<5	BDL<5[(tr)]	BDL<5	BDL<5	NR	NR	BDL<5
GM-01D	Jun-90	BDL<5	BDL<5[(tr)]	BDL<5	BDL<5	NR	NR	BDL<5
GM-02S	Jun-90	11	BDL<5[(tr)]	BDL<5	BDL<5	NR	NR	BDL<5
GM-03S	Jun-90	NS	NS	NS	NS	NS	NS	NS
GM-01S	Sep-90	BDL<5	BDL<5[(tr)]	BDL<5	BDL<5	NR	NR	BDL<5
GM-01D	Sep-90	BDL<5	BDL<5[(tr)]	BDL<5	BDL<5	NR	NR	BDL<5
GM-02S	Sep-90	8	BDL<5[(tr)]	BDL<5	BDL<5	NR	NR	BDL<5
GM-03S	Sep-90	NS	NS	NS	NS	NS	NS	NS
GM-01S	Mar-94	BDL<5	BDL<5	BDL<5	BDL<5	NR	NR	NR
GM-01D	Mar-94	BDL<5	BDL<5	BDL<5	BDL<5	NR	NR	NR
GM-02S	Mar-94	BDL<5	BDL<5	BDL<5	BDL<5	NR	NR	NR
GM-03S	Mar-94	NS	NS	NS	NS	NS	NS	NS
GM-01S	Mar-95	BDL<5	BDL<5	BDL<5	BDL<5	NR	NR	NR
GM-01D	Mar-95	BDL<5	BDL<5	BDL<5	BDL<5	NR	NR	NR
GM-02S	Mar-95	BDL<5	BDL<5	BDL<5	BDL<5	NR	NR	NR
GM-03S	Mar-95	NS	NS	NS	NS	NS	NS	NS
GM-01S	Mar-96	BDL<5	BDL<5	BDL<5	NR	NR	NR	NR
GM-01D	Mar-96	BDL<5	BDL<5	BDL<5	NR	NR	NR	NR
GM-02S	Mar-96	BDL<5	BDL<5	BDL<5	NR	NR	NR	NR
GM-03S	Mar-96	NS	NS	NS	NS	NS	NS	NS
GM-01S	Mar-97	BDL<5	BDL<5	BDL<5	NR	NR	NR	NR
GM-01D	Mar-97	BDL<5	BDL<5	BDL<5	NR	NR	NR	NR
GM-02S	Mar-97	BDL<5	BDL<5	BDL<5	NR	NR	NR	NR
GM-03S	Mar-97	NS	NS	NS	NS	NS	NS	NS
GM-01S	Mar-98	BDL	BDL	BDL	NR	NR	NR	NR
GM-01D	Mar-98	BDL	BDL	BDL	NR	NR	NR	NR
GM-02S	Mar-98	BDL	BDL	BDL	NR	NR	NR	NR
GM-03S	Mar-98	NS	NS	NS	NS	NS	NS	NS
GM-01S	Mar-99	BDL	BDL	BDL	BDL	NR	NR	NR
GM-01D	Mar-99	BDL	BDL	BDL	BDL	NR	NR	NR
GM-02S	Mar-99	BDL	BDL	BDL	BDL	NR	NR	NR
GM-03S	Mar-99	NS	NS	NS	NS	NS	NS	NS
GM-01S	Apr-00	BDL	NR	BDL	NR	NR	NR	NR
GM-01D	Apr-00	BDL	NR	BDL	NR	NR	NR	NR
GM-02S	Apr-00	BDL	NR	BDL	NR	NR	NR	NR
GM-03S	Apr-00	NS	NS	NS	NS	NS	NS	NS
GM-01S	Apr-01	BDL	NR	NR	NR	NR	NR	NR
GM-01D	Apr-01	BDL	NR	NR	NR	NR	NR	NR
GM-02S	Apr-01	BDL	NR	NR	NR	NR	NR	NR
GM-03S	Apr-01	NS	NS	NS	NS	NS	NS	NS
GM-01S	Apr-02	NS	NS	NS	NS	NS	NS	NS
GM-01D	Apr-02	BDL	NR	NR	NR	NR	NR	NR
GM-02S	Apr-02	NS	NS	NS	NS	NS	NS	NS
GM-03S	Apr-02	NS	NS	NS	NS	NS	NS	NS

Table 2
SWMU 23 Area Groundwater Monitoring Data Summary

	MCL (ug/L)	5	70(cis)	5				5
Well No.	**Date**	**TCE (ug/L)**	**1,2-DCE (ug/L)**	**Benzene (ug/L)**	**Chloroform (ug/L)**	**Carbon Disulfide (ug/L)**	**Acetone (ug/L)**	**Methylene Chloride (ug/L)**
GM-01S	Apr-03	NS	NS	NS	NS	NS	NS	NS
GM-01D	Apr-03	BDL	NR	NR	NR	NR	NR	NR
GM-02S	Apr-03	NS	NS	NS	NS	NS	NS	NS
GM-03S	Apr-03	NS	NS	NS	NS	NS	NS	NS
GM-01S	Apr-04	NS	NS	NS	NS	NS	NS	NS
GM-01D	Apr-04	BDL	BDL(cis)	NR	NR	NR	NR	NR
GM-02S	Apr-04	NS	NS	NS	NS	NS	NS	NS
GM-03S	Apr-04	NS	NS	NS	NS	NS	NS	NS
GM-01S	Apr-05	NS	NS	NS	NS	NS	NS	NS
GM-01D	Apr-05	BDL	BDL(cis)	NR	NR	NR	NR	NR
GM-02S	Apr-05	NS	NS	NS	NS	NS	NS	NS
GM-03S	Apr-05	NS	NS	NS	NS	NS	NS	NS
GM-01S	Apr-06	NS	NS	NS	NS	NS	NS	NS
GM-01D	Apr-06	BDL	BDL(cis)	NR	NR	NR	NR	NR
GM-02S	Apr-06	NS	NS	NS	NS	NS	NS	NS
GM-03S	Apr-06	NS	NS	NS	NS	NS	NS	NS
GM-01S	Apr-07	NS	NS	NS	NS	NS	NS	NS
GM-01D	Apr-07	BDL	NR	NR	NR	NR	NR	NR
GM-02S	Apr-07	NS	NS	NS	NS	NS	NS	NS
GM-03S	Apr-07	NS	NS	NS	NS	NS	NS	NS
GM-01S	Apr-08	NS	NS	NS	NS	NS	NS	NS
GM-01D	Apr-08	BDL	NR	NR	NR	NR	NR	NR
GM-02S	Apr-08	NS	NS	NS	NS	NS	NS	NS
GM-03S	Apr-08	NS	NS	NS	NS	NS	NS	NS
GM-01S	Apr-09	NS	NS	NS	NS	NS	NS	NS
GM-01D	Apr-09	BDL	NR	NR	NR	NR	NR	NR
GM-02S	Apr-09	NS	NS	NS	NS	NS	NS	NS
GM-03S	Apr-09	NS	NS	NS	NS	NS	NS	NS
GM-01S	Apr-10	NS	NS	NS	NS	NS	NS	NS
GM-01D	Apr-10	BDL	NR	NR	NR	NR	NR	NR
GM-02S	Apr-10	NS	NS	NS	NS	NS	NS	NS
GM-03S	Apr-10	NS	NS	NS	NS	NS	NS	NS
GM-01S	Apr-11	NS	NS	NS	NS	NS	NS	NS
GM-01D	Apr-11	BDL<5	BDL<5(cis)	BDL<5	BDL<5	NR	NR	BDL<5
GM-02S	Apr-11	NS	NS	NS	NS	NS	NS	NS
GM-03S	Apr-11	NS	NS	NS	NS	NS	NS	NS
GM-01S	Apr-12	NS	NS	NS	NS	NS	NS	NS
GM-01D	Apr-12	BDL<5	BDL<5(cis)	BDL<5	BDL<5	NR	NR	BDL<5
GM-02S	Apr-12	NS	NS	NS	NS	NS	NS	NS
GM-03S	Apr-12	NS	NS	NS	NS	NS	NS	NS

Notes:

BDL	Below Detection Limit.
ug/L	Micrograms Per Liter.
NS	Not Sampled.
NR	Not Reported
1,2-DCE	1,2-Dichloroethene (total)
(tr)	trans-1,2-Dicholoroethene
(cis)	cis-1,2,-Dichloroethene
TCE	Trichloroethene

Source:CCR, ARCADIS, 2012b

Table 3
Summary of Existing PCB Data for Additional Areas

Sample Date	Soil Boring Name	Start Depth	End Depth	Units	Matrix	Parameter	Analytical Result	Units	Lab Qualifier	PCB Method
3/30/2005	R1BM-01	0.0	1.0	ft	Soil	Total PCBs	0.93120	mg/kg		Homologue
3/30/2005	R1BM-01	1.0	2.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
3/30/2005	R1BM-01	10.0	11.0	ft	Soil	Total PCBs	2.49505	mg/kg		Homologue
3/30/2005	R1BM-01	12.0	13.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
3/7/2005	R1CS-06	0.0	1.0	ft	Soil	Total PCBs	1.80195	mg/kg		Homologue
3/7/2005	R1CS-06	1.0	2.0	ft	Soil	Total PCBs	1.24175	mg/kg		Homologue
3/7/2005	R1CS-07	0.0	1.0	ft	Soil	Total PCBs	1.64285	mg/kg		Homologue
3/7/2005	R1CS-07	1.0	2.0	ft	Soil	Total PCBs	0.42795	mg/kg		Homologue
6/15/2005	R1EM-02	0.0	1.0	ft	Soil	Total PCBs	0.14610	mg/kg		Homologue
6/15/2005	R1EM-02	1.0	2.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
6/15/2005	R1EM-02	12.0	13.0	ft	Soil	Total PCBs	0.23000	mg/kg		Homologue
6/14/2005	R1EM-03	0.0	1.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
6/14/2005	R1EM-03	1.0	2.0	ft	Soil	Total PCBs	0.11625	mg/kg		Homologue
6/14/2005	R1EM-03	19.0	20.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
3/30/2005	R1EM-04A	0.0	1.0	ft	Soil	Total PCBs	0.26960	mg/kg		Homologue
3/30/2005	R1EM-04A	1.0	2.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
3/30/2005	R1EM-04A	9.0	10.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
3/30/2005	R1EM-04A	10.0	11.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
6/14/2005	R1EM-05	0.0	1.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
6/14/2005	R1EM-05	1.0	2.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
6/14/2005	R1EM-05	19.0	20.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
6/14/2005	R1EM-06	0.0	1.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
6/14/2005	R1EM-06	1.0	2.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
6/14/2005	R1EM-06	13.0	14.0	ft	Soil	Total PCBs	0.11225	mg/kg		Homologue
6/15/2005	R1EM-07	0.0	1.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
6/15/2005	R1EM-07	1.0	2.0	ft	Soil	Total PCBs	0.24885	mg/kg		Homologue
6/15/2005	R1EM-07	16.0	17.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
3/30/2005	R1EM-08	0.0	1.0	ft	Soil	Total PCBs	0.53225	mg/kg		Homologue
3/30/2005	R1EM-08	1.0	2.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
3/30/2005	R1EM-08	9.0	10.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue
3/30/2005	R1EM-08	10.0	11.0	ft	Soil	Total PCBs	0.01135	mg/kg	U	Homologue

Source: ENVIRON. 2005. Data Summary Report: Floodplain Soil (March 7-June 15, 2005). August 2005.

Table 4: SWMU 23 and Additional Areas of Potential Concern (AA-02 through AA-09) - Data Quality Objectives **Revision 2, June 2013**

Step 1: State the Problem	Step 2: Identify Goals of the RFI	Step 3: Identify Information Inputs	Step 4: Define Boundaries of the RFI	Step 5: Develop a Decision Rule	Step 6: Specify Performance or Acceptance Criteria	Step 7: Develop Plan of Obtaining Data
I. Assess the concentrations of COPCs in environmental media at SWMU 23. **II.** Assess the concentrations of PCBs in soil at each AA. **III.** Determine the nature and extent of constituent releases from SWMU 23, if any. **IV.** If COPC concentrations at SWMU 23 and/or one or more AA are determined to exceed relevant screening values, assessment of risk to current and future human health and the environment is necessary. **V.** The risk assessment will establish whether the sites pose an unacceptable risk	**1.** Determine if any RCRA metals and/or VOC constituents have been released from SWMU 23.	Information inputs include: 1. Sampling and analysis of existing and newly installed and developed groundwater monitoring wells at SWMU 23 on AK Steel property, and sampling and analysis of cap material, near surface and subsurface soils at SWMU 23. Laboratory analysis will include RCRA metals and VOCs for all samples. Cap material samples also will have grain size analysis performed. 2. Historic groundwater metals and VOC analytical results from the existing wells near SWMU 23 from 1989 through 2010 and included in the CCR Appendices E-4 and E-5, are considered usable in the RFI for this SWMU. 3. Existing potentiometric surface maps for the shallow aquifer will be evaluated from the on-going sitewide groundwater study. 4. New potentiometric surface maps will be created from the RFI groundwater monitoring event. 5. Soil boring and well logs from existing and the new soil	The RFI boundaries will be determined by the concentrations detected in the groundwater sample analyses from the first water bearing zone and the soil samples collected at SWMU 23.	If RCRA metals and/or VOC constituents are detected above relevant USEPA Regional screening levels, step-out sampling will be performed. For groundwater, the subject wells will first be resampled for confirmation of the elevated result. RCRA metals concentrations detected will be compared to the up-gradient well concentrations to assist in evaluating whether the metals may be naturally occurring. Background concentrations in soil may also be used for evaluation of data. If it is determined that a RCRA metals concentration exceeding a relevant USEPA Regional screening level (RSL) is not consistent with naturally occurring concentrations, or if one or more validated VOC constituent result is detected above the applicable RSL, step-out locations will be identified and access agreements pursued to allow complete delineation of the extent of groundwater and soil impacts. Vapor intrusion pathway analysis will be conducted if RFI data indicate VOC impacts from SWMU 23 that may result	Laboratory analytical data will be deemed usable based on data review and validation as described in the RFI QAPP. Additionally, precision and accuracy metrics are specified in the RFI QAPP. Analytical results that are “J flagged” will be included and used in the data set. If data are rejected (e.g., “R” flagged) or otherwise determined to be invalid via data review and validation, the data will not be usable for decision making. Field and laboratory SOPs are included in the RFI QAPP and will be implemented to establish performance and acceptance of the field and laboratory work elements. Existing groundwater monitoring wells will be inspected to assure functionality prior to sampling. Unless otherwise determined with USEPA and OEPA, historical data that have not been validated or for which original laboratory reports are not available will be considered screening data and will not be included in the site risk assessment.	Data will be obtained as follows: 1. A total of four new groundwater monitoring wells will be installed. Three new groundwater monitoring wells will be installed on AK Steel owned property in locations that are currently interpreted as being side and down gradient of the closed lagoons. The well planned on the eastern edge will consist of a nested pair for monitoring of both shallow and intermediate aquifers. One (1) additional upgradient shallow water table monitoring well will be installed along the west side of SWMU 23 2. The new and existing monitoring wells at SWMU 23 will be sampled with laboratory analysis of RCRA metals and VOC constituents to determine the current concentrations of these COPCs in groundwater. 3. Depth to groundwater will be measured in each of the wells and a potentiometric surface map prepared. 4. Soil borings will be installed to sample near surface and subsurface soils as well as cap materials, and determine concentrations of RCRA metals and VOCs in soils.

Step 1: State the Problem	Step 2: Identify Goals of the RFI	Step 3: Identify Information Inputs	Step 4: Define Boundaries of the RFI	Step 5: Develop a Decision Rule	Step 6: Specify Performance or Acceptance Criteria	Step 7: Develop Plan of Obtaining Data
to potential current and future receptors, and thus whether a Corrective Measures Study is necessary. **VI.** The overall integrity of existing containment structures at SWMU 23 has not been evaluated.		borings and wells installed at SWMU 23 will be used.		in a complete pathway to one or more "occupied" structures as defined by USEPA. If vapor intrusion evaluation is determined to be necessary based on soil or groundwater data, additional access agreements and a work plan addendum will be required.		5. Logs of the soil borings and wells will be examined and relevant information compiled. 6. All analytical results will be reviewed, validated, compiled and compared to established screening values consistent with the Consent Decree and CCR. 7. If elevated constituent concentrations are present such that the extent of groundwater impacts has not been defined, an additional phase of groundwater investigation will be developed in the downgradient direction. If elevated constituent concentrations are present such that the extent of soil impacts has not been defined, an additional phase of soil investigation will be developed. Prior to implementing further investigations, well and soil sampling locations and access to the locations will be required to be established.
	2. Determine the extent and concentration of PCB constituents present on each AA.	Information inputs include: 1. Existing IM soil boring analytical data and boring logs. 2. New soil borings to be installed, sampled and analyzed for PCB concentrations at each AA. 3. Historic interview records	The boundaries of the first phase of investigation are the parcel boundaries as shown on RFI figures.	Using Regional Screening Levels (per the CCR) for soil sample evaluation, soil on each AA will be sampled and analyzed to fully delineate vertical and horizontal extent of PCB constituents on each parcel. These samples will be collected from 1 ft intervals. The following intervals will be analyzed:	Laboratory analytical data will be deemed usable based on data review and validation as described in the RFI QAPP. Additionally, precision and accuracy metrics are specified in the RFI QAPP. Analytical results that are "J flagged" will be included and used in the data set. If data are	1. Soil borings will be installed to determine the horizontal and vertical extent of PCBs in any dredge spoils historically placed on each AA. 2. Logs of the soil borings will be examined and relevant information compiled. 3. All analytical results will be reviewed, validated, compiled and

Step 1: State the Problem	Step 2: Identify Goals of the RFI	Step 3: Identify Information Inputs	Step 4: Define Boundaries of the RFI	Step 5: Develop a Decision Rule	Step 6: Specify Performance or Acceptance Criteria	Step 7: Develop Plan of Obtaining Data
		and aerial photographs regarding each AA.		• 0-1', 1-2' and 2-3'; • the deepest sediment interval based on field observations; Additional samples from native soil at the base of the boring or mid-intervals may be analyzed based on field observations or analytical results. Sample locations proposed for sampling are identified on 75' horizontal spacing, subject to field limitations that may be encountered and pending access agreements being established. Contingency soil sampling locations will be sampled as illustrated on AA-specific maps (and as refined in the field based on site-specific limitations) to define the extent of PCB impacts. Vapor intrusion pathway evaluation will not be conducted unless detected PCB soil sample concentrations indicate that the vapor pathway is a potentially complete pathway.	rejected (e.g., "R" flagged) or otherwise determined to be invalid via data review and validation, the data will not be usable for decision making. Field and laboratory SOPs are included in the RFI QAPP and will be implemented to establish performance and acceptance of the field and laboratory work elements. Data from soil borings installed during previous PCB investigations in the area as illustrated in the RFI Work Plan are fully usable for the RFI.	compared to established screening values consistent with the Consent Decree and CCR. 4. If elevated constituent concentrations are present such that the extent of PCB constituent concentrations has not been defined on each AA, contingency borings will be installed and sampled. Additional step-out borings will be installed if determined necessary to complete the delineation of PCB concentrations. Prior to implementing step-out investigation beyond the initially planned contingency sample locations, it is possible access agreement modifications and/or additional utility clearances may be required.
	3. Complete human health and ecological risk assessments to determine whether current and potential	Characterize constituent concentrations in each environmental medium for SWMU 23 (soil and groundwater) potentially	The boundaries of the risk assessment include SWMU 23 and each AA. For SWMU 23, the	If RFI results provide sufficient delineation of the nature and extent of impacts from SWMU 23 and each AA to support calculation of risk estimates	Laboratory data incorporated into the 2012 CCR, collected under the Consent Decree, and new data that are not rejected via data review and validation	Data collection planning is described in previous goals and steps.

Step 1: State the Problem	Step 2: Identify Goals of the RFI	Step 3: Identify Information Inputs	Step 4: Define Boundaries of the RFI	Step 5: Develop a Decision Rule	Step 6: Specify Performance or Acceptance Criteria	Step 7: Develop Plan of Obtaining Data
	future on-site and off-site risks exist to human health and the environment related to SWMU 23 and each AA. Identify all media and areas where there exists an excess lifetime cancer risks within the range of 1E-06, 1E-04 and 1E-04 and /or a hazard index equal to or greater than 1 in accordance with standards specified in the March 2006 Consent Decree. Refine COPCs to define Contaminants of Concern (COC) by medium for SWMU 23 and each AA. Develop media specific standards as necessary for COCs.	impacted by Coil Paint plant lagoon releases to the environment and characterize constituent concentrations in each AA (soil) impacted by historic dredge spoils placement (AA02 through AA09). Characterize potential scenarios for human and ecological exposure to SWMU 23 COCs, and AA COCs, under current and reasonably expected future use scenarios.	boundaries radiate outward as necessary to define the nature and extent of constituents in soil and groundwater media, and if determined necessary, vapors. For the AAs, the boundaries are the property boundaries for PCB in soil for both human health and ecological receptors as deemed necessary.	associated with reasonable maximum exposure (RME) under both current and reasonably anticipated future land uses, additional RFI data collection will not be necessary. Otherwise, additional RFI data collection will be planned and executed to complete risk evaluation. The need for ecological risk assessment of the AAs will be dependent upon the RFI data findings. Pending additional data acquisition and consistent with the Preliminary SCM for the AAs as included in the approved CCR (Fig 45 of the CCR), ecological receptors will not be evaluated for the AAs unless site soil data indicate chemical (PCB) constituents in surface soil (based on the 0-3' analytical results) that would be available to ecological receptors.	will be fully usable as decision making data. Historic data that does not meet these criteria will not be used for decision making unless an evaluation of the data with United States et al. determines such data to be usable. If not usable for risk assessment, the data will be considered screening data.	
	4. Evaluate the overall integrity of existing containment structures at SWMU 23 if analytical results from media sampling indicate potential for the release of COPCs from the lagoons.	Information inputs include: 1. Sampling and analysis of subsurface soils at SWMU 23 within the lagoon footprint. Laboratory analysis will include RCRA metals, AL, ZN and VOCs for all samples. Cap material samples also will have grain size analysis performed.	Integrity evaluation will be limited to the soil caps placed on the three lagoons at SWMU 23 used for Coil Paint wastewater treatment.	The cap material samples will be analyzed for RCRA metals, VOCs and grain size. A decision on the need for permeability testing of the cap material will be made after laboratory analytical results of the soil borings are received and evaluated. If there is no evidence of COPCs beneath the lagoons above relevant regulatory leaching values , no	Laboratory analytical data will be deemed usable based on data review and validation as described in the RFI QAPP. Additionally, precision and accuracy metrics are specified in the RFI QAPP. Analytical results that are "J flagged" will be included and used in the data set. If data are rejected (e.g., "R" flagged) or	Data collection planning is described in previous goals and steps.

		2. Sampling and geotechnical analysis of cap material for permeability (if warranted).		permeability testing will be performed. If COPCs are detected beneath the lagoons above the relevant leaching levels, additional samples of the cap material will be collected for permeability testing (per QAPP SOPs/ASTM methods) and leakage through the cap will be estimated using US EPAs HELP model (or other model with prior USEPA approval).	otherwise determined to be invalid via data review and validation, the data will not be usable for decision making. Field and laboratory SOPs are included in the RFI QAPP and will be implemented to establish performance and acceptance of the field and laboratory work elements. Landfill cap permeability measurements, if required, will be compared to RCRA Subtitle C or Subtitle D Cap standards, depending on detected concentrations of COPCs in the former lagoon soils (eg., hazardous versus non-hazardous landfill cap permeability standards)	

ATTACHMENT A

EXISTING SWMU-23 SOIL BORING/MONITORING WELL LOGS

GERAGHTY & MILLER, INC.
Ground-Water Consultants

OB Nº C1311AR1	BORE HOLE Nº GM-1S
PROJECT Armco	LOCATION Middletown, Ohio
DRILLING CONTRACTOR H.C. Nutting	DRILLING EQUIPMENT Mobile Drill B-61, Hollow Stem Auger
HYDROGEOLOGIST Craig Stevens	DRILLER Hobert Herd/Don Adams
DATE START / TIME 10/13/87	DATE FINISH / TIME 10/14/87
SURFACE ELEVATION	TOTAL DEPTH 51'
WELL CASING 2" PVC	SCREEN TYPE 2" PVC
LENGTH 10'	SLOT

GROUND WATER			
DATE	TIME	DEPTH	WEATHER

	CASING	CORE	SAMPLER	TUBE
TYPE	PVC		S-Spoon	
DIAMETER	2"		1.6" OD	
HAMMER WEIGHT			140 lbs	
FALL			30"	

REMARKS

BORE HOLE LOG

DEPTH (FT.)	SAMPLE Nº	BLOW COUNT PER 6 IN.	RECOVERY (FT.)	LITHOLOGIC DESCRIPTION	REMARKS	GRAPHIC LOG
	1	3-5-12	1.0	Fine SAND (90%); tr gravel; brown, dry, organics	Large, well rounded pale to white gray gravel in cuttings from 2'-5'.	
5	2	8-14 20	1.0	Med-cs SAND (50%); gravel (50%); brown-pale brown and tan, dry and damp.		
10	3	7-18 19	1.4	Med SAND (60%); gravel (20%); cs sand (15%); med brown-tan, damp-moist, well sorted.		
15	4	19-23 24	1.3	GRAVEL (50%); medium sand (25%); cs sand (25%); light brown, gravel is pale colored, dry, damp, poorly sorted		
20	5	32-33	.7	Cs SAND (40%); gravel (40%); silt (15%); light brown-tan, damp, moist.		
25	6	18-24 32	1.5	Cs SAND (40%); med sand (40%); gravel (20%); light-med. brown, moist. Fe staining at 36.2' tr Manganese.		

GERAGHTY & MILLER, INC.
Ground-Water Consultants

JOB NO. C1311AR1
BORE HOLE NO. GM-1S
PROJECT Armco
LOCATION Middletown, Ohio
REMARKS

DEPTH (FT.)	SAMPLE NO	BLOW COUNT PER 6 IN.	RECOVERY (FT.)	LITHOLOGIC DESCRIPTION	REMARKS	GRAPHIC LOG
	7	16-32	.8	Cs sand (70%); gravel (20%); silt (10%); pale gray-tan, wet (saturated?)		
35	8	28-70	.6	Medium SAND (35%); cs sand (30%); gravel (25%); silt (10%); pale gray-light brown, saturated.		
40	9	18-36 38	1.5	Top: Med SAND (50%); cs sand (50%); brown, saturated, well sorted. Btm: Med SAND (60%); gravel (30%); tr silt, light brown, saturated.		
45	10	20-32 36	1.5	Med SAND (30%); cs sand (30%); gravel (25%); fine sand (15%); tr silt grading into med SAND (25%); cs sand (25%); gravel (50%); gray-brown, saturated, Fe staining on gravel		
50	11	32-54	1	Med SAND (25%); cs sand (25%); gravel (35%); tr fine sand; tr silt; grayish brown, saturated.		

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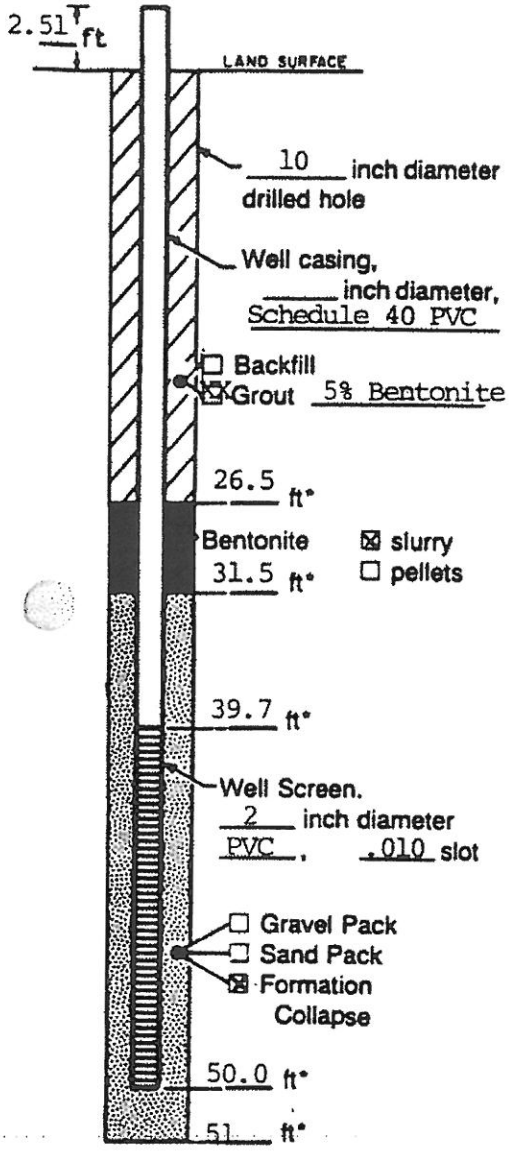
Ground-Water Consultants

WELL CONSTRUCTION LOG



Measuring Point is Top of Well Casing Unless Otherwise Noted.

*Depth Below Land Surface

Project Armco/C1311AR1 Well GM-1S

Town/City Middletown

County Butler State Ohio

Permit No. _____

Land-Surface Elevation

and Datum _____ feet ☐ surveyed

_____ ☐ estimated

Installation Dates(s) 10/13/87 - 10/14/87

Drilling Method Hollow Stem Auger

Drilling Contractor H. C. Nutting

Drilling Fluid 60 gallons city water

Development Techniques(s) and Date(s)

Brainard-Kilman hand pump and surge block (1/14/88)

Fluid Loss During Drilling _____ gallons

Water Removed During Development 160 gallons

Static Depth to Water 41.89 (1/16/88) feet below M.P.

Pumping Depth to Water _____ feet below M.P.

Pumping Duration _____ hours

Yield _____ gpm Date _____

Specific Capacity _____ gpm/ft

Well Purpose Water level monitoring

Remarks

Prepared by George H. Colvin

GERAGHTY & MILLER, INC.
Ground-Water Consultants

NO. C1311AR1	BORE HOLE NO. GM-1D
PROJECT: Armco	LOCATION: Middletown, Ohio
DRILLING CONTRACTOR: Reynolds	DRILLING EQUIPMENT: Cable Tool
HYDROGEOLOGIST: David L. O'Brien	DRILLER: Steve Back
DATE START / TIME: 10/2/87	DATE FINISH / TIME: 10/28/87
SURFACE ELEVATION:	TOTAL DEPTH: 110.2
WELL CASING: Schedule 40 PVC	SCREEN TYPE: Schedule 40 PVC
LENGTH: 10'	SLOT: 0.010

GROUND WATER					CASING	CORE	SAMPLER	TUBE
DATE	TIME	DEPTH	WEATHER	TYPE	PVC			
				DIAMETER	4"			
				HAMMER WEIGHT				
				FALL				

REMARKS

DEPTH (FT.)	SAMPLE NO	BLOW COUNT PER 6 IN.	RECOVERY (FT.)	BORE HOLE LOG: LITHOLOGIC DESCRIPTION	REMARKS	GRAPHIC LOG
	1			Top Soil; organic matter; brown		
5	2			Cs GRAVEL to COBBLES; fine to med sand; tr clay; brown		
10	3			Med to cs SAND (70%); fine sand (10%); gravel (20%); brown		
15	4			Med to cs SAND (50%); gravel (50%); light brown, poorly sorted		
20	5			GRAVEL (50%); v cs sand (25%); med to cs sand (25%); light brown to tan		
	6			GRAVEL (50%); med to cs SAND (50%); light brown		
30	7			Med to v cs SAND (60%); gravel (40%); light brown		

GERAGHTY & MILLER, INC.
Ground-Water Consultants

JOB NO. 1311AR1

BORE HOLE NO. GM-1D

PROJECT Armco

LOCATION Middletown, Ohio

REMARKS

DEPTH (FT.)	SAMPLE NO.	BLOW COUNT PER 6 IN.	RECOVERY (FT.)	LITHOLOGIC DESCRIPTION	REMARKS	GRAPHIC LOG
35	8			Med to cs SAND (90%); gravel (10%); light brown		
40	9					
	10					
45	11			V cs SAND (80%); med to cs sand (20%) brown to grey		
50	12			V fine to fine SAND (60%); v cs SAND (30%); silt (10%); brown to tan		
55	13					
60	14					
65	15			Med SAND (80%); v cs sand (20%); tr silt light brown to tan		
70				Cs to v cs SAND (50%); med sand (25%); gravel and cobbles (25%) lt brn to tan		

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JOB NO. C1311AR1
BORE HOLE NO. GM-1D
PROJECT Armco
LOCATION Middletown, Ohio
REMARKS

DEPTH (FT.)	SAMPLE NO	BLOW COUNT PER 6 IN.	RECOVERY (FT.)	LITHOLOGIC DESCRIPTION	REMARKS	GRAPHIC LOG
75	16					
	17			Med to v cs SAND (50%); gravel (50%); light brown to tan		
80	18			V cs SAND to fine GRAVEL (60%); fine to med sand (20%); silt (20%); grey		
85	19			Fine to med SAND (60%); fine to med gravel (30%) silt (10%); tr silt; few cobbles grey to tan		
90	20					
95	21			Med to cs SAND (70%); fine gravel (20%) silt (10%); grey		
100	22			med SAND (85%); fine to med gravel (10%) tr fine; light brown to grey		
105	23					

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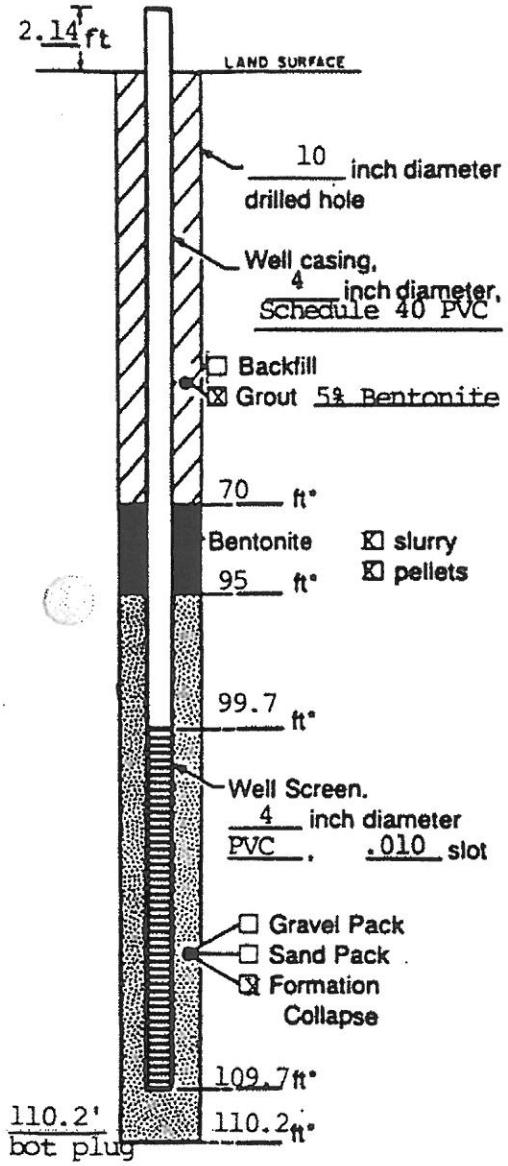
Ground-Water Consultants

WELL CONSTRUCTION LOG



Measuring Point is Top of Well Casing Unless Otherwise Noted.

*Depth Below Land Surface

Project Armco C1311AR1 . Well GM-1D

Town/City Middletown

County Butler State Ohio

Permit No. _____

Land-Surface Elevation

and Datum _____ feet ☐ surveyed

_____ ☐ estimated

Installation Dates(s) 10/21/87 - 10/28/87

Drilling Method Cable Tool

Drilling Contractor Reynolds

Drilling Fluid 1450 gallons city water

Development Techniques(s) and Date(s)
Submersible pump (1/8/88)

Fluid Loss During Drilling _____ gallons

Water Removed During Development 2679 gallons

Static Depth to Water 42.18 (1/8/88) feet below M.P.

Pumping Depth to Water 43.30 feet below M.P.

Pumping Duration 3.3 hours

Yield _____ gpm Date 1/8/88

Specific Capacity 12.14 gpm/ft

Well Purpose Water level monitoring

Remarks 10" casing depth below land surface - 83.1' with a final stick-up of 2.5'; bentonite pellets 95' to 90'; bentonite slurry 90 to 70'.

Prepared by David L O;Brien

GERAGHTY & MILLER, INC.
Ground-Water Consultants

JOB NO: C1311AR1	BORE HOLE NO: GM-2S
PROJECT: Armco	LOCATION: Middletown, Ohio
DRILLING CONTRACTOR: H.C. Nutting	DRILLING EQUIPMENT: Mobile Drill B-61, Hollow Stem Auger
HYDROGEOLOGIST: Craig Stevens	DRILLER: Hobert Herd/Don Adams
DATE START / TIME: 10/14/87	DATE FINISH / TIME: 10/15/87
SURFACE ELEVATION:	TOTAL DEPTH: 51.5'
WELL CASING: 2" PVC	SCREEN TYPE: 2" PVC
LENGTH: 10'	SLOT:

GROUND WATER					CASING	CORE	SAMPLER	TUBE
DATE	TIME	DEPTH	WEATHER	TYPE	PVC		S-Spoon	
				DIAMETER	2"		1.6" OD	
				HAMMER WEIGHT			140 lbs	
				FALL			30"	

REMARKS

DEPTH (FT.)	SAMPLE NO	BLOW COUNT PER 6 IN.	RECOVERY (FT.)	BORE HOLE LOG — LITHOLOGIC DESCRIPTION	REMARKS	GRAPHIC LOG
	1	8-15 16	.9	TOPSOIL Fine SAND (25%); v fine sand (25%); gravel (30%); silt (20%); med brown, dry		
5	2	6-7-8	1.0	GRAVEL (50%); med sand (20%); cs sand (20%); tr fine sand; light brown-tan, damp.		
10	3	9-15 16	1.5	GRAVEL (35%); cs sand (30%); med sand (20%); tr fine sand; tr silt; light brown-tan, moist, Fe staining.	High percentage of limestone in soil samples	
15	4	12-18 30	1.3	Same as above; faint chemical odor.		
20	5	15-18 18	1.3	Same as above; stronger chemical odor. GRAVEL; brown-rust orange, saturated, well rounded		
25	6	15-32 31	1.5	Mid .5': Fine SAND (55%); gravel (30%); cs sand (15%); gray brown, moist, Fe staining. Btm .5': Fine-cs SAND (80%); gravel (20%) rust orange brown, moist, Fe staining; chemical odor.		

GERAGHTY & MILLER, INC.
Ground-Water Consultants

JOB NO: C1311AR1
BORE HOLE NO: GM-2S
PROJECT: Armco
LOCATION: Middletown, Ohio
REMARKS:

DEPTH (FT.)	SAMPLE NO	BLOW COUNT PER 6 IN.	RECOVERY (FT.)	LITHOLOGIC DESCRIPTION	REMARKS	GRAPHIC LOG
	7	17-15 18	1.5	Med SAND (70%); tr fine sand; tr med sand; tr gravel; med brown, wet, well sorted, v faint chemical odor.		
35	8	32-54	1.3	Same as above	Water table at 38'	
40	9	25-70	1.2	Med SAND (35%); cs sand (35%); gravel (20%); tr fine; brown, saturated		
45	10	18-30	0	No recovery; but same as above		
50	11	49-90	1.0	SILT (45%); clay (35%); tr fine sand; tr gravel; olive gray, saturated, compact, dense		

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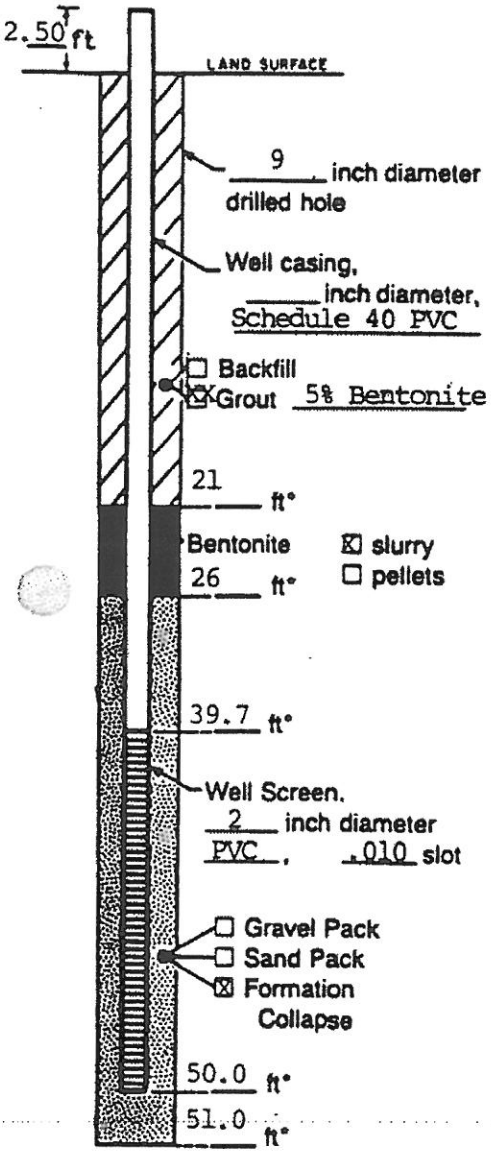
WELL CONSTRUCTION LOG



Measuring Point is Top of Well Casing Unless Otherwise Noted.

*Depth Below Land Surface

Project Armco/C1311AR1 Well GM-2S

Town/City Middletown

County Butler State Ohio

Permit No. _____

Land-Surface Elevation

and Datum _____ feet ☐ surveyed

_____ ☐ estimated

Installation Dates(s) 10/14/87 - 10/15/87

Drilling Method Hollow Stem Auger

Drilling Contractor H. C. Nutting

Drilling Fluid 90 gallons city water

Development Techniques(s) and Date(s)

Brainard-Kilman hand pump and surge block (1/14/88)

Fluid Loss During Drilling _____ gallons

Water Removed During Development 266 gallons

Static Depth to Water 40.75 (1/16/88) feet below M.P.

Pumping Depth to Water _____ feet below M.P.

Pumping Duration _____ hours

Yield _____ gpm Date _____

Specific Capacity _____ gpm/ft

Well Purpose Water level monitoring

Remarks

Prepared by George H. Colvin

GERAGHTY & MILLER, INC.
Ground-Water Consultants

JOB NO: C1311AR1	BORE HOLE NO: GM-3S
PROJECT: Armco	LOCATION: Middletown, Ohio
DRILLING CONTRACTOR: H.C. Nutting	DRILLING EQUIPMENT: Mobile Drill B-61, Hollow Stem Auger
HYDROGEOLOGIST: Craig Stevens	DRILLER: Hobert Herd/Don Adams
DATE START / TIME:	DATE FINISH / TIME:
SURFACE ELEVATION:	TOTAL DEPTH: 49.5'
WELL CASING: 2" PVC	SCREEN TYPE: 2" PVC
LENGTH: 10'	SLOT: .010

GROUND WATER					CASING	CORE	SAMPLER	TUBE
DATE	TIME	DEPTH	WEATHER	TYPE	PVC		S-Spoon	
				DIAMETER	2"		1.6" OD	
				HAMMER WEIGHT			140 lbs	
				FALL			30"	

REMARKS

DEPTH (FT.)	SAMPLE NO	BLOW COUNT PER 6 IN.	RECOVERY (FT.)	BORE HOLE LOG: LITHOLOGIC DESCRIPTION	REMARKS	GRAPHIC LOG
	1	6-18-14	1.5	Fine SAND (90%); tr silt; brown with ochre tint, dry; topsoil in top 2.4"		
5	2	25-24-20	1.3	Med SAND (35%); cs sand (20%); gravel (35%); tr fine; light brown, damp	Abundance of limestone in sample.	
10	3	9-15-25	1.4	Same as above; moist.		
15	4	25-36-38	1.5	Clayey silt (70%); fine sand (20%); fine gravel (10%); olive gray, wet-saturated, dense, compacted.	1.5-2.0' of water perched on fine grained silt unit	
20	5	15-25-50	1.5	Fine SAND (50%); silt (30%); tr fine gravel, brown to rust brown, wet horizontal partings.		
25	6	11-28-53	1.5	Med SAND (50%); cs sand (40%); tr gravel, light brown; damp-moist, well sorted.		

GERAGHTY & MILLER, INC.
Ground-Water Consultants

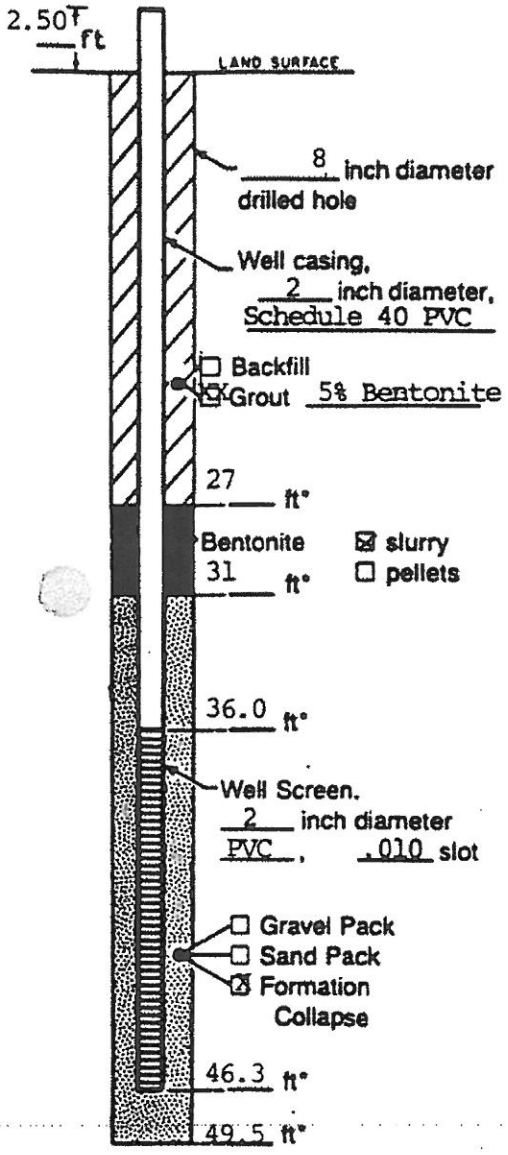
JOB NO: C1311AR1
BORE HOLE NO: GM-3S
PROJECT: Armco
LOCATION: Middletown, Ohio
REMARKS:

DEPTH (FT.)	SAMPLE NO	BLOW COUNT PER 6 IN.	RECOVERY (FT.)	LITHOLOGIC DESCRIPTION	REMARKS
	7	18-26 -22	1.5	Med SAND (90%); tr med gravel, brown, damp, very well sorted.	
35				Med-cs GRAVEL (30%); fine sand (20%); multi-colored; saturated	Water table at 34'
	8	9-25-25	1.3	Fine-med SAND (70%); silt (20%); tr gravel; light brown-tan, saturated	
40				Med-cs SAND (80%); tr gravel; tr fine; gray-brown, saturated	
	9	16-55	1.0	Fine-med SAND (70%); silt (15%); gravel (15%); brown-tan, saturated	
45				Med-cs SAND (70%); fine sand (15%); silt (15%); gravel (10%); brown, saturated.	
	10	15-23 -34	1.5		
50	11	22-50	1.0	SILT (40%); clay (30%); sand (15%); gravel (15%); gray, wet-saturated, dense, very compact	

GRAPHIC LOG

GERAGHTY & MILLER, INC.
Ground-Water Consultants

WELL CONSTRUCTION LOG



Measuring Point is Top of Well Casing Unless Otherwise Noted.

*Depth Below Land Surface

Project Armco/C1311AR1 Well GM-3S

Town/City Middletown

County Butler State Ohio

Permit No. _____

Land-Surface Elevation

and Datum _____ feet ☐ surveyed

_____ ☐ estimated

Installation Dates(s) 10/15/87 - 10/16/87

Drilling Method Hollow Stem Auger

Drilling Contractor H. C. Nutting

Drilling Fluid 40 gallons city water

Development Techniques(s) and Date(s)

Brainard-Kilman hand pump and surge block (1/14/88)

Fluid Loss During Drilling _____ gallons

Water Removed During Development 190 gallons

Static Depth to Water 37.70 (1/16/88) feet below M.P.

Pumping Depth to Water _____ feet below M.P.

Pumping Duration _____ hours

Yield _____ gpm Date _____

Specific Capacity _____ gpm/ft

Well Purpose Water level monitoring

Remarks Well came up 2' while grouting and pulling augers.

Prepared by George H. Colvin

SWMU 23

MONITORING WELL CONSTRUCTION AND GROUNDWATER ELEVATIONS SUMMARY TABLE

ARCADIS

Table 2. Site Wide Well Construction and Groundwater Elevations, AK Steel Corporation, Middletown, Ohio.

Well ID	Northing	Easting	Ground Suface Elevation	Reference Point Elevation	Reference Point Elevation December 2008	Borehole depth ft. bls	Well diameter inches	Top of Screen		Bottom of Screen		April 2011	
								Depth ft. bls	Elevation ft. bls	Depth ft. bls	Elevation ft. bls	GW Depth	GW Elev.
Intermediate Aquifer Wells													
GM-01S[3]	547650.39	1431327.99	649.71	652.21	--	51.5	2	39.7	610.01	50	599.71	27.10	625.11
GM-02S[3]	548074.70	1431326.55	648.71	651.21	--	51.5	2	39.7	609.01	50	598.71	26.08	625.13
GM-03S[3]	547954.58	1432522.76	645.98	648.48	--	49.5	2	36	609.98	46.3	599.68	24.42	624.06
GM-04S[3]	543254.63	1436321.43	646.81	649.31	--	36.5	2	19.9	626.91	30.2	616.61	8.78	640.53
GM-05S[3]	546414.04	1434033.89	662.43	664.93	--	64.5	2	57.5	604.93	62.8	599.63	42.53	622.40
GM-06S[3]	549800.31	1434180.34	666.76	669.26	--	65.0	2	52.8	613.96	63.1	603.66	50.99	618.27
GM-07S[3]	545829.55	1434030.48	660.71	663.21	--	61.5	2	49.8	610.91	60.1	600.61	41.05	622.16
GM-08S	545722.65	1435435.96	666.25	667.05	--	66.5	2	53.5	612.75	63.8	602.45	48.35	618.70
GM-09S[3]	544676.81	1436211.96	659.42	661.92	--	56.5	2	44.2	615.22	54.2	605.22	19.27	642.65
GM-10S[3]	543356.99	1440627.48	651.15	653.65	--	61.5	2	55.1	596.05	60.4	590.75	33.11	620.54
GM-11S	543190.20	1438531.12	652.74	654.92	653.85	56.5	2	44.7	608.04	55	597.74	35.55	618.30
GM-12SR	544310.25	1438507.61	659.13	--	661.75	97	2	50	609.13	60	599.13	40.65	621.10
GM-14S	545678.11	1438485.31	660.22	662.09	--	66.5	2	52.6	607.62	62.9	597.32	35.45	626.64
GM-15S[3]	545986.45	1437739.90	659.95	662.45	--	56.5	2	44.1	615.85	54.5	605.45	34.45	628.00
GM-16S[3]	545919.77	1436662.39	657.65	660.15	--	61.5	2	49.4	608.25	59.7	597.95	37.50	622.65
GM-17S[1][3]	547823.25	1435116.65	668.86	671.36	--	70	2	58.6	610.26	68.9	599.96	51.45	619.91
GM-18S[3]	548708.88	1435803.78	673.52	676.02	--	71.5	2	58.9	614.62	69.2	604.32	NM	NA
GM-19S[3]	549102.63	1437119.69	671.17	673.67	--	66.5	2	54.3	616.87	64.6	606.57	50.32	623.35
GM-20S[1]	550728.52	1437268.10	670.88	674.26	672.45	71.5	2	59.6	611.28	69.9	600.98	55.93	616.52
GM-21S[3]	551905.67	1436311.09	670.47	672.97	--	81.5	2	69.3	601.17	79.7	590.77	55.60	617.37
GM-22S[3]	550604.85	1436418.87	667.30	669.80	--	71.5	2	59	608.30	69.3	598.00	51.65	618.15
GM-23S	549678.37	1435726.24	667.90	670.12	--	76.5	2	62.9	605.00	73.2	594.70	54.90	615.22
GM-24S[3]	550029.28	1436589.21	672.40	674.90	--	71.5	2	59	613.40	69.3	603.10	56.05	618.85
GM-25S[3]	552721.05	1435522.70	664.49	666.99	--	66.5	2	55.3	609.19	65.6	598.89	47.52	619.47
GM-26S[3]	551064.20	1435473.94	663.64	666.14	--	71.5	2	58.7	604.94	69	594.64	49.17	616.97
GM-27S[3]	552451.49	1434183.04	658.70	661.20	--	71	2	59.83	598.87	70.13	588.57	42.88	618.32
GM-29D[1][3]	539857.72	1434427.84	657.04	659.54	--	65	4	58.4	598.64	63.4	593.64	6.48	653.06
GM-30D[1][3]	541116.28	1438325.62	654.84	657.34	--	51.3	4	46.3	608.54	51.3	603.54	39.28	618.06
GM-35I	539961.05	1436056.11	654.49	--	656.64	56	2	37	617.49	47	607.49	10.18	646.46
GM-37I	542689.65	1443157.34	656.40	--	658.27	61	2	50	606.40	60	596.40	29.48	628.79
GM-39D	542845.28	1445981.47	656.53	658.80	657.81	57	4	49.2	607.33	54.2	602.33	24.93	632.88
GM-41I	547429.32	1442160.71	655.98	--	658.73	177	2	38	617.98	48	607.98	27.64	631.09
GM-43I	545130.17	1441042.26	654.80	--	657.24	58	2	45	609.80	55	599.80	32.52	624.72
GM-46D[1][3]	541994.46	1436170.54	646.66	649.16	--	65	4	54.9	591.76	64.9	581.76	26.18	622.98
GM-48S[3]	549252.51	1430470.99	647.06	649.56	--	52	2	40	607.06	50	597.06	24.78	624.78
GM-49S[3]	547845.18	1429167.14	641.88	644.38	--	47	2	34	607.88	44	597.88	19.12	625.26
GM-57I	540236.76	1442546.39	655.63	657.06	--	55	2	37	618.63	47	608.63	24.43	632.63
GM-58I	540359.68	1443536.44	649.53	651.52	--	46	2	30	619.53	40	609.53	17.65	633.87
GM-60I	541284.71	1441301.36	655.15	658.70	657.55	61.75	2	49.75	605.40	59.75	595.40	30.70	626.85
GM-63I[3]	541884.02	1441430.36	662.71	665.21	--	72	2	60	602.71	70	592.71	38.51	626.70
GM-64I[3]	540673.83	1443161.08	655.83	658.33	--	50	2	35	620.83	45	610.83	25.27	633.06
MW-3I	541111.82	1442597.17	650.99	653.40	--	56	2	41.5	609.49	51.5	599.49	20.93	632.47
MW-5I	539485.66	1443392.95	655.00	657.37	--	60	2	42.5	612.50	52.5	602.50	23.75	633.62
A1I[3]	551160.36	1434400.79	665.20	667.70	--	69	2	58	607.20	68	597.20	53.82	613.88
DMW-1D[3]	544315.09	1436032.89	653.70	656.20	--	44	2	32	621.70	42	611.70	17.15	639.05
DMW-2D[3]	544318.46	1436027.28	654.51	657.01	--	48	2	37.5	617.01	47.5	607.01	15.01	642.00
DMW-4D	544747.84	1436084.76	655.57	656.88	--	48	2	37.5	618.07	47.5	608.07	13.30	643.58
DMW-8[3]	545284.19	1436042.96	660.39	662.89	--	49	2	44	616.39	49	611.39	NM	NA

ATTACHMENT B

QUALITY ASSURANCE PROJECT PLAN

RCRA Facility Investigation
QUALITY ASSURANCE PROJECT PLAN
SWMU 23 and Additional Areas of Potential Concern AA02 – AA09
Middletown, Ohio
Revision 1

Prepared for:

AK Steel Corporation
9227 Centre Pointe Drive
West Chester, OH 45069

Prepared by:

KEMRON Environmental Services, Inc.
2343-A State Route 821
Marietta, OH 45750

May 2013

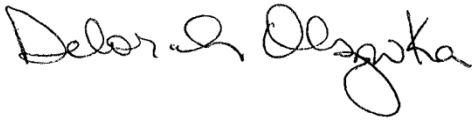
SIGNATURE PAGE

RCRA Facility Investigation
QUALITY ASSURANCE PROJECT PLAN
SWMU 23 and Additional Areas of Potential Concern AA01-AA09
Middletown, Ohio

Prepared by: KEMRON Environmental Services, Inc.
Prepared for: AK Steel Corporation

05/10/2013
KEMRON Project Manager **Date**
Mary Lou Rochotte, C.P.G

05/10/2013
AK Steel Project Manager **Date**
Jim Kemp



05/09/2013
TestAmerica Project Manager **Date**
Debbie Olszowka

U.S. EPA Project Manager **Date**
Michael Mikulka

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Appendix A **Standard Operating Procedures**

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LIST OF ABBREVIATIONS AND ACRONYMS

AA	Additional Areas of Potential Concern
CMS	Corrective Measures Study
COPC	Constituent of Potential Concern
DQA	Data Quality Assessment
DQO	Data Quality Objectives
EDD	electronic data deliverable
ENVIRON	ENVIRON International Corporation
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
IMs	Interim Measures
KEMRON	KEMRON Environmental Services
MDL	Method Detection Limit
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NRDC	Natural Resources Defense Council
Ohio EPA	Ohio Environmental Protection Agency
OSHA	Occupational Health and Safety Administration
PCBs	polychlorinated biphenyls
QAM	Quality Assurance Manual
QAPP	Quality Assurance Project Plan
QA/QC	quality assurance/quality control
QMP	Quality Management Plan
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
RLs	Reporting Limits
RPD	relative percent difference
SDG	sample delivery group
SOP	Standard Operating Procedure
SVOCs	semi-volatile organic compounds
SWMUs	Solid Waste Management Units
TestAmerica	TestAmerica Analytical Testing Corporation
United States et al.	United States, State of Ohio, Sierra Club, and NRDC
USEPA	United States Environmental Protection Agency
VOCs	volatile organic compounds
WAN	wide area network
%R	percent recovery

1.0 PROJECT DESCRIPTION

1.1 Introduction

This Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) Quality Assurance Project Plan (QAPP) addresses the procedures necessary to document all monitoring methods, sampling, field measurements, and sample analyses performed during the RFI being conducted for Solid Waste Management Unit (SWMU) 23 and eight (8) Additional Areas of Potential Concern (AAs), located in Middletown, Ohio (Figure 1). The scope of work for the RFI process is described in Attachment 2 of the Consent Decree. As such, this RFI QAPP applies to the RFI Work Plan, Revision 4 that addresses activities associated with SWMU 23 and the AAs.

The RFI QAPP will ensure that all information, data, and resulting decisions are technically sound, statistically valid, and properly documented. This RFI QAPP includes a brief description of the project; project organization and responsibility; quality assurance objectives; sampling and analytical procedures; and data reduction, validation, and reporting requirements. All quality assurance/quality control (QA/QC) procedures will be structured in accordance with applicable United States Environmental Protection Agency (USEPA) and Ohio Environmental Protection Agency (Ohio EPA) requirements, regulations, guidance, and technical standards (e.g., USEPA, 1998; 2002a).

1.2 Site Description and History

The AK Steel Middletown Works is located in the City of Middletown in Butler County in southwestern Ohio (Figure 1). AK Steel produces flat-rolled carbon, stainless and electrical steel products, as well as carbon and stainless tubular steel products for the automotive, appliance, construction, and manufacturing industries. The AK Steel Middletown Works facility covers approximately 2,600 acres and consists of the Former Coil Paint Area (SWMU 23), the North Plant Area, the Melt Plant Area, the South Plant Area, and the Slag Processing Area.

A portion of Dicks Creek flows between the AK Steel plant and the Slag Processing Area. Dicks Creek is a small, partially channelized stream draining an urbanized and industrialized watershed of approximately 50 square miles (Figure 1). Dicks Creek is located to the south and east of Middletown, Ohio, in Butler and Warren Counties. The creek originates to the east of Middletown and flows 12 miles westward toward its confluence with the Great Miami River. AK Steel owns the land surrounding and underlying those portions of Dicks Creek between river mile 2.5 and 5.6.

SWMU 23 is the former location of two (2) lagoons and one (1) sand bed within in the Coil Paint Area, which were associated with process water treatment. Alternately, some documents reference the sand bed as a third lagoon. SWMU 23 is located approximately ½ mile west of the main plant area. The lagoons and sand bed have been closed since the early 1980's. The SWMU 23 lagoons were designed to receive wastewater from Coil Paint Area production facilities. These wastewaters contained metals, and potentially could have contained VOCs. SWMU 23 is to be investigated to determine if volatile organic compounds (VOCs) or RCRA metal constituents from the lagoons have impacted soil or groundwater.

Additional Areas AA02 through AA09 are off-site properties located adjacent to Dicks Creek and south of the AK Steel Plant. Historical evidence from review of aerial photographs and interviews with local residents and workers associated with the late 1960's channelization of Dicks Creek indicates dredged spoils from the creek may have been placed on one or more of these properties. Dicks Creek sediments are documented as being impacted with PCBs and ongoing Interim Measures (IMs) have been implemented in Dicks Creek adjacent to the Additional Areas to remove contaminated sediments and floodplain soils. The potential exists for PCB-impacted sediments, removed during historical creek excavation activities, to have been deposited on the Additional Areas resulting in a potential release of PCBs to the environment. The eight (8) AAS are to be investigated be investigated to determine if materials containing PCBs have been placed on any AA from historical dredging operations conducted in Dicks Creek prior to the Interim Measures. The AAs are currently either vegetated or have gravel or concrete drive and parking areas, such that the parcels are stable and contain no incision that would expose underlying soils for significant runoff of potential PCB

contaminants. Current use of the various parcels varies including uses such as: residential, open, graded, gravel covered field, or commercial use (such as Fraternal Order of Eagles, Schwanns commercial food distribution center; other businesses). AA 01 has been addressed through IMs implemented during 2012.

1.3 Project Objectives

The project objectives of the RFI of SWMU 23 and AAs AA02 through AA09 will be to:

1. Characterize the nature and extent of contamination on the subject parcels.
2. Gather environmental data necessary to support an assessment of risk to human health and the environment posed by any identified releases of constituents.
3. Determine whether a corrective measures study (CMS) is required.

The RFI Work Plan describes the specific tasks and detailed field methodology necessary for evaluating the nature, extent, and impact of contamination, if any, originating from SWMU 23 or on an AA. This RFI QAPP is designed to assure and control data quality from its inception during sampling and analysis through reduction, validation, technical evaluation and reporting.

Figures 12 – 20 of the RFI Work Plan show the anticipated soil and groundwater sample locations. The constituents of interest with regard to this QAPP are as follows:

- Volatile Organic Compounds (VOCs) in soil and groundwater at SWMU 23;
- Total RCRA Metals in the soil and groundwater at SWMU 23; and
- Polychlorinated Biphenyls (PCBs) in soil at the AAs.

Information on laboratory analytical methods and sample containers is provided in Table 3.

1.4 Project Schedule

A schedule for the implementation of the RCRA Facility Investigation and Corrective Measures Study is included in the RFI Work Plan. The schedule includes the selection of subcontractors, implementation of field work, data analysis, and the preparation of the various reports.

1.5 Data Quality Objectives

As defined by the USEPA (2006), Data Quality Objectives (DQOs) are most commonly used during the planning stages of any study that requires data collection. DQOs define the goals of the investigation and guide data collection. In environmental data collection projects, the ultimate goal of the DQO process is to obtain high quality and valid data to support environmental decisions. This goal can be achieved through systematic planning and strict adherence to quality assurance/quality control (QA/QC) measures. The DQO process is a seven-step iterative process designed to ensure that data collection is resource effective, while meeting the objectives of the study

Data Quality Objectives (DQOs) were developed in accordance with EPA's seven step process to serve as a basis for designing a plan for collecting data of sufficient quality and quantity to support the goals of the RFI (EPA 2006). The seven steps include: (1) State the Problem; (2) Identify the Goals of the Investigation; (3) Identify Information Inputs; (4) Define the Boundaries of the Investigation; (5) Develop a Decision Rule; (6) Specify Performance Criteria; and (7) Develop a Plan for Obtaining Data. The DQOs were developed after the historical data for the site were assessed for usability and evaluated, and data gaps were identified.

The DQO process identified the following problems to be addressed by the goals of the RFI:

1. Assess the concentrations of COPCs in environmental media at SWMU 23;
2. Assess the concentrations of PCBs in soil at each Additional Area (AA02 through AA09);
3. Determine the nature and extent of constituent releases from SWMU 23, if any;
4. If COPC concentrations at SWMU 23 and/or one or more AA are determined to exceed established screening values, assessment of risk to human health and the environment is necessary. Risk assessment will establish whether the site poses an unacceptable risk to potential receptors. Assessment will include potential and actual human health exposures, both short term (acute) and long term (chronic);
5. Determining if a Corrective Measures Study is necessary for SWMU 23 or one or more of the Additional Areas, based on the site specific data and information gathered during the RFI.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

AK Steel has responsibility for all phases of the investigation. AK Steel has selected KEMRON to manage the project, direct the field investigations, manage all incoming data, develop and populate the project database, prepare the report, and perform any subsequent studies, as appropriate. Specific individuals will be designated to each responsibility upon implementation of the project. The KEMRON Project Manager will report to the AK Steel Project Manager and Project Coordinator.

2.1 Management Responsibilities

Management responsibility, including the AK Steel Project Coordinator, AK Steel Project Manager, and the KEMRON Project Manager are described below.

2.1.1 AK Steel Project Coordinator

The primary function of the AK Steel Project Coordinator (Pat Gallo) is to ensure that technical, financial, and scheduling objectives are achieved successfully. The Project Coordinator will approve all external reports (deliverables) before their submission to the United States et al.

2.1.2 AK Steel Project Manager

The AK Steel Project Manager (James Kemp) is responsible for implementing the project and has the authority to commit the resources necessary to meet project objectives and requirements. The AK Steel Project Manager will work directly with the KEMRON Project Manager to monitor and direct daily activities; communicate health and safety information to project personnel during daily safety tailgate meetings; regulate site admittance to personnel; and serve as a liaison among KEMRON, subcontractor(s), and regulatory personnel and the AK Steel Project Coordinator.

2.1.3 KEMRON Project Manager

The KEMRON Project Manager (Mary Lou Rochotte) has responsibility for ensuring that the project meets the objectives of the United States et al. and KEMRON's quality standards. The KEMRON Project Manager will provide assistance to the AK Steel Project Manager and Project Coordinator in terms of writing and distributing the RFI QAPP to all those parties connected with the project (including each laboratory). The KEMRON Project Manager will report directly to the AK Steel Project Coordinator and is responsible for technical quality control and project oversight.

2.1.4 KEMRON Database Manager

The KEMRON database manager (David Pitzer) will be responsible for the following:

- Setting up the project database as specified on a company server;
- Creating and maintaining lists of synonyms and valid values for data fields;
- Controlling access to the database;
- Performing routine maintenance on the database including backup and performance refinement;
- Protecting the usability and integrity of the database;
- After reviewing electronic data deliverables (EDDs), uploading the data into the database and verifying the new data is formatted correctly and complete;

- Overseeing post-import quality control;
- Verifying that all data values are valid;
- Updating data records based on the data validation report, including the documentation of these changes; and
- Creating queries, data exports, and summary tables from the database to be used to produce text, tables, graphs, maps, isopleth plots, models, and other products used to describe the data. Assigning staff to quality control evaluation of summary tables, queries, and database export routines.

2.2 Field Responsibilities

The KEMRON field team leader will support the AK Steel Project Manager. The team leader is responsible for leading and coordinating the day-to-day activities of the various resource specialists under their supervision. Specific field team leader responsibilities include:

- Providing day-to-day coordination with the KEMRON Project Manager on technical issues in specific areas of expertise;
- Developing and implementing field-related work plans, assurance of schedule compliance, and adherence to management-developed study requirements;
- Coordinating and managing field activities, including sampling and drilling, and supervising field staff;
- Implementing QC for technical data provided by the field staff, including field measurement data;
- Adhering to work schedules provided by the AK Steel and/or KEMRON Project Manager;
- Authoring and approving of text and graphics required for field team efforts;
- Coordinating and overseeing technical efforts of subcontractors assisting the field team;
- Identifying problems at the field team level, resolving difficulties in consultation with the AK Steel and KEMRON Project Managers, implementing and documenting corrective action procedures, and providing communication between team and upper management; and
- Participating in preparation of the final report.

Table 1 provides the names and points of contact for KEMRON personnel and subcontractors.

Field Technical Staff

The technical staff for this project will be drawn from KEMRON's pool of corporate resources and, as necessary, subcontractors. The technical staff will be utilized to gather and analyze data and to prepare various task reports and support materials. All of the designated technical team members are experienced professionals who possess the degree of specialization and technical competence to effectively and efficiently perform the required work.

Table 1
Contractor and Subcontractor Key Points of Contact
AK Steel SWMU 23 and AAs

Contractor	Title	Email	Key Point of Contact
Mary Lou Rochotte, CPG, PMP	Project Manager	mrochotte@kemron.com	KEMRON Environmental Services, Inc. 2343-A State Route 821 Marietta, Ohio 45750 T (740) 373-4308 F (740) 376-2536
Lou Ehrhard, PG	QA Manager	lehrhard@kemron.com	KEMRON
Marilyn Zumbro	H&S Officer	mzumbro@kemron.com	KEMRON
Kim Trowbridge	Risk Assessor	ktrowbridge@kemron.com	KEMRON
Subcontractor	**Title**	**Email**	**Key Point of Contact**
Debbie Olszowka	Laboratory Project Manager	Debbie.Olszowka@testamericainc.com	TestAmerica
Becki Strait	Laboratory QA Officer	becki.strait@testamericainc.com	TestAmerica (North Canton, OH)
Michael H. Dunn	QA Manager	mike.dunn@testamericainc.com	TestAmerica (Nashville, TN)
Tim Goodall	Laboratory Director	tggoodall@terracon.com	Terracon
David Westendorf, P.E.	Laboratory QA Officer	dwestendoft@terracon.com	Terracon

2.3 Laboratory Responsibilities

Laboratory chemical analytical services are anticipated to be provided by TestAmerica of North Canton, OH (TestAmerica). This laboratory maintains NELAP certification for all applicable analyses for this RFI. Geotechnical testing services are anticipated to be provided by Terracon Consultants, Inc. (Terracon). H.C. Nutting Company (HCN), headquartered in Cincinnati, Ohio, officially changed its name to Terracon Consultants, Inc. on Jan. 1, 2012. HCN has performed geotechnical analysis for Interim Measures under the Consent Decree. If additional laboratories are required, an addendum or revision to this RFI QAPP will be submitted. The individual laboratories and their responsibilities are listed below. All information below is based on and consistent with the laboratory information included in the AK Steel SWMUs 38 and 39 RFI/CMS Work Plan (ENVIRON, 2012) or previously provided in the Interim Measures QAPP (ENVIRON, 2007), as approved by EPA.

2.3.1 TestAmerica Analytical Testing Corporation

Test America
4101 Shuffle Street NW
North Canton, OH 44720
Phone: (330) 497-9396

Test America
2960 Foster Creighton Drive
Nashville, TN 37204
Phone: (615) 726-0177

TestAmerica is responsible for analyses of groundwater and soil sample VOCs using USEPA Method 8260, PCB-Aroclors using USEPA Method 3520/8082 (fluids, applicable only to QA samples) and 3541 (or 3540)/8082 (solids), total and dissolved RCRA metals using USEPA Method 6010, and total and dissolved mercury using USEPA Method 7470/71.

Laboratory Project Manager

The TestAmerica laboratory Project Manager (Debbie Olszowka) will serve as the liaison to the KEMRON Project Manager and will be responsible for the following:

- Ensuring client specifications are met by communicating project and quality assurance requirements to the laboratory;
- Notifying laboratory personnel of incoming projects and sample delivery schedules;
- Verifying complete chain-of-custody documentation
- Monitoring the status of all projects in-house to ensure timely delivery of reports;
- Informing the client of project-related problems, resolving service issues, and coordinating technical issues with the laboratory staff;
- Coordinating client requests for sample containers and other services;
- Scheduling sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples;
- Coordinating any subcontract work;
- Preparing data packages;
- Reviewing laboratory data reports, quotes, and sample login sheets;
- Reviewing and approving data reports prior to their release to the client; and
- Ensuring client specific reporting and quality control requirements are met.

Laboratory Division Manager

The TestAmerica laboratory division manager (Opal Johnson) provides the resources necessary to implement and maintain an effective and comprehensive quality assurance program and will be responsible for the following:

- Providing technical guidance to the analytical staff. The Division Manager may be the source point for technical help or designate an individual(s) to fill this role. A Division Manager appoints the technical directors for the appropriate fields of testing. The names of the technical director will be included in the national database;
- If a technical director is absent for a period of time exceeding 15 consecutive calendar days, the Division Manager must designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function (this can be one of the other technical directors). If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing;
- Ensuring that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented;
- Ensuring that personnel are free from any commercial, financial, and other undue pressures that might adversely affect the quality of their work;

- Ensuring TestAmerica's human resource policies are adhered to and maintained;
- Ensuring that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the laboratory specific Quality Assurance Manual (QAM) or laboratory standard operating procedures (SOPs) may be suspended by the Division Manager;
- Reviewing and approving all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to;
- Establishing and maintaining a laboratory information system for tracking all samples in the
- laboratory and utilizes the system to ensure all sample holding times are met; and
- Assuming the responsibilities of the QA Officer in the temporary absence of the QA Officer.

Laboratory Operations Manager

The TestAmerica laboratory operations manager (Carolynne Roach) will be responsible for the following:

- Monitoring standards of performance in quality control and quality assurance;
- Monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data;
- Ensuring that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory;
- Providing training direction to laboratory staff;
- Monitoring production efforts of the laboratory. Investigating techniques to enhance production and improve quality through technical advances and improved Laboratory Information Management System utilization; and
- Coordinating and reviewing preparation of all test method SOPs.

Laboratory Quality Assurance Officer

The TestAmerica laboratory QA Officer (Becki Strait) has the overall responsibility for ensuring that systems are maintained to produce data that are technically sound, legally defensible, and of consistent quality in line with the laboratory specific QAM and SOPs. Specifically, the laboratory QA Officer will be responsible for the following:

- Serving as the focal point for QA/QC and overseeing and/or reviewing Quality Control Data;
- Maintaining and updating the laboratory specific QAM. Ensure that the manual accurately reflects the procedures of the laboratory;
- Monitoring and communicating regulatory changes that may affect the laboratory to management, marketing, and the Corporate Director of Quality Assurance;
- Pursuing and maintaining appropriate laboratory certification and contract approvals. Arranging for the analysis of Performance Testing samples necessary to satisfy certification requirements;

- Training and advising the laboratory staff on QA/QC procedures that are pertinent to their daily activities;

- Functioning independent from laboratory operations for which he/she has quality assurance oversight;

- Possessing a general knowledge of the analytical test methods for which data audit/review is performed (and/or have the means of getting this information when needed);

- Arranging for or conducting internal audits; and

- Notifying laboratory management of deficiencies in the quality system and ensuring that corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs must be temporarily suspended or restricted following the procedures outlined in the laboratory specific QAM.

Final responsibility for project quality rests with KEMRON's Project Manager. Independent QA will be provided by the laboratory Project Manager and QA Officer prior to release of all data to KEMRON/AK Steel.

Laboratory Technical Staff

The TestAmerica laboratory technical staff will be responsible for sample analyses and identification of necessary corrective actions. The staff will report directly to the laboratory operations manager.

Some of the SOPs provided by the analytical laboratories have either a CONTROLLED DOCUMENT or UNCONTROLLED DOCUMENT watermark. A controlled document means that analytical laboratory has assigned some type of identifier to the document and the laboratory tracks each person or holder that receives the document. The holder must acknowledge receipt of the document from the laboratory. Anytime a revision is made to a controlled document, all holders of that document will receive a new revised copy of the document. If a holder of a controlled document leaves the company that they work for, they are required to return all copies of the controlled document to the laboratory. If a document has the CONTROLLED DOCUMENT watermark but does not have an identifier on it, it means that the document is available as a controlled document but was not requested as a controlled document.

In general, the UNCONTROLLED DOCUMENT watermark means that a document is not updated once the analytical laboratory releases the document to a holder.

2.3.2 Terracon Consultants, Inc.

Terracon Consultants, Inc.
611 Lunken Park Drive
Cincinnati, OH 45226
Phone: (513) 321 5816

Terracon Consultants, Inc. (Terracon) will be responsible for analyses of geotechnical data. H.C. Nutting Company (HCN), headquartered in Cincinnati, Ohio, officially changed its name to Terracon Consultants, Inc. on Jan. 1, 2012. HCN has performed geotechnical analysis for Interim Measures under the Consent Decree.

Permeability testing of SWMU 23 cap materials may be necessary during implementation of the RFI. Undisturbed soil collected within Shelby tubes from the cap material of SWMU 23 will be analyzed for hydraulic conductivity in accordance with American Society for Testing and Materials [ASTM] D5084.

Liquid limit, plastic limit, and plasticity index will be analyzed in accordance with ASTM D4318. In addition, particle size analysis will be conducted in accordance with ASTM D422.

All relevant Terracon SOPs and ASTM Methods are included in Appendix A. The specific Terracon SOPs to be used are listed below:

SOP GT-002: Processing Bulk Samples

SOP GT-005: ASTM D4318 – Liquid Limit, plastic limit, and Plasticity Index of Soils

SOP GT-007: ASTM D422 – Soil Classification - Grain Size Analysis of Soil

SOP GT-008: ASTM D2216 – Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

SOP GT-012: Preparing Test Specimens from Undisturbed Samples for Permeability Testing

SOP GT-013: ASTM D5084 - Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter

Laboratory Director

The Terracon Laboratory Director (Tim Goodall) will serve as liaison to the KEMRON Project Manager and will report directly to the Terracon Operations Manager for the Cincinnati Division (Jess Schroeder). The Laboratory Director will be responsible for the following:

- Ensuring internal quality control;
- Ensuring that report quality and format are consistent corporate-wide;
- Establishing required competency levels of senior level professionals;
- Providing technical input to the President and Upper Management;
- Evaluating technical aspects of new market potential;
- Coordinating technical resource sharing; and
- Reviewing technical issues as submitted by Division and/or Department Managers for review.

Laboratory Manager

The Terracon Laboratory Manager (George Sanders) will report to the Laboratory Director and will be responsible for the following:

- Coordinating and overseeing laboratory analyses;
- Overseeing preparation of analytical reports; and
- Ensuring internal quality control.

Quality Control Officer

The Terracon Control Officer (David Westendorf, P.E.) has the overall responsibility for ensuring quality control for laboratory data.

Laboratory Technicians

The Terracon laboratory technical staff (Steve Vogelsang, Fred Eichler, and David Roelker) will be responsible for sample analyses and identification of necessary corrective actions. The staff will report directly to the Terracon Laboratory Manager.

2.4 Quality Assurance/Data Validation Responsibilities

2.4.1 Data Validation

The Data Validator will report directly to the KEMRON Project Manager and will be responsible for the following data validation activities:

- Verifying complete chain-of-custody documentation;
- Comparing blank values to sample values (equipment blanks, laboratory blanks, and method blanks);
- Verifying sample analysis dates and preparation dates with respect to sample holding times;
- Evaluating data on matrix spike/matrix spike duplicate (MS/MSD) samples;
- Reviewing field documentation (where necessary);
- Summarizing and assigning validation codes to the sample data; and
- Documenting data that do not meet validation criteria.

Data validation may be conducted by a third-party firm or by a KEMRON chemist not involved with project oversight or the sample collection process.

2.4.2 Data Assessment

The KEMRON Project Manager (Mary Lou Rochotte) is responsible for the assessment of data to ensure the adherence to high scientific and technical standards.

Additionally, KEMRON will assure that at least 5% of analytical data are validated independently by a KEMRON chemist not involved in the sampling effort or by a third-party firm.

2.5 Project Organization Diagram

Figure 10 of the RFI Work Plan presents a diagram illustrating the project organization.

2.6 Special Training Requirements, Drug Testing, and Certifications

All contractor field personnel involved in the RFI field work will have completed the 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) training with annual refresher courses as required by the Occupational Safety and Health Administration (OSHA). In addition, AK Steel Corporation has specific health and safety standards that may be applicable. A Health and Safety Plan (HASP) has

been developed and is included in the RFI Workplan. Laboratory personnel training and qualifications are described in laboratory-specific Quality Management Plans (QMPs), which are available upon request.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

Field and laboratory quality assurance objectives for each analytical method have been specified in Tables 2.1a through 2.3b.

Table 2.1a
QC Guidelines for VOC Soil Samples

Sampling Procedure	Analytical Method	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)
12-SSP-01 SOP for Soil Sampling 12-PID-AKMW SOP for Field Screen of Samples using Photo-Ionization Detector 12-SBC-AKMW SOP for Soil Boring Collection	8260B	Accuracy/Bias /Contamination	<Reporting Limit (RL)	Field Blanks	S
		Precision	≤ 35%	Field Duplicate	S
		Accuracy/Bias /Contamination	<RL exception: common laboratory contaminants as noted in SOP	Method Blank	A
		Accuracy/Bias	Laboratory-derived limits	LCS	A
		Accuracy/Bias	Laboratory-derived limits	MS/MSD	A
		Precision	Laboratory-derived limits (RPD ≤30)	MS/MSD	A
		Precision	Standards (% Rec) Laboratory-derived limits 4-Bromofluorobenzene (52-136) Toluene-D8 (67-125) 1,2-Dichloroethane-D4 (58-123) Dibromofluoromethane (37-132)	Surrogates	A
		Precision	Retention time ±30 seconds of last CC Area changes by a factor of two (-50% to +100%)	Internal Standards	A

[1]If information varies within an analytical group, separate by individual analyte.

Table 2.1b
QC Guidelines for VOC Groundwater Samples

Sampling Procedure	Analytical Method	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)
12-GWS-01 SOP for Groundwater Sampling	8260B	Accuracy/Bias /Contamination	<RL	Field Blanks	S
		Precision	≤ 25%	Field Duplicate	S
		Accuracy/Bias /Contamination	<RL	Method Blank	A
		Accuracy/Bias	Laboratory-derived limits	LCS	A

		Accuracy/Bias	Laboratory-derived limits	MS/MSD	A
		Precision	Laboratory-derived limits (RPD ≤30)	MS/MSD	A
		Precision	Standards (% Rec) Laboratory-derived limits 4-Bromofluorobenzene (66-117) Toluene-D8 (74-115) 1,2-Dichloroethane-D4 (63-129) Dibromofluoromethane (75-121)	Surrogates	A
		Precision	Retention time ±30 seconds of last CC Area changes by a factor of two (-50% to +100%)	Internal Standards	A

[1]If information varies within an analytical group, separate by individual analyte.

Table 2.2a
QC Guidelines for PCB Soil Samples

Sampling Procedure	Analytical Method	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)
12-SSP-01 SOP for Soil Sampling 12-SBC-AKMW SOP for Soil Boring Collection	8082	Accuracy/Bias /Contamination	<RL	Field Blanks	S
		Precision	≤ 35%	Field Duplicate	S
		Accuracy/Bias /Contamination	<RL	Method Blank	A
		Accuracy/Bias	Laboratory-derived limits	LCS	A
		Accuracy/Bias	Laboratory-derived limits	MS/MSD	A
		Precision	Laboratory-derived limits (RPD ≤30)	MS/MSD	A
		Precision	Standards (% Rec) Laboratory-derived limits 2,4,5,6-Tetrachloro-m-Xylene (29-151) Decachlorobiphenyl (PCB-209) (14-163)	Surrogates	A

[1]If information varies within an analytical group, separate by individual analyte.

Table 2-2b
QC Guidelines for PCB Water Samples

Sampling Procedure	Analytical Method	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)
07-DCN-00 SOP for Sampling Equipment and Container Decontamination 07-FQC-00 SOP for Field Quality Control	8082	Accuracy/Bias /Contamination	<RL	Field Blanks	S
		Precision	≤ 25%	Field Duplicate	S
		Accuracy/Bias /Contamination	<RL	Method Blank	A
		Accuracy/Bias	Laboratory-derived limits	LCS	A
		Accuracy/Bias	Laboratory-derived limits	MS/MSD	A
		Precision	RPD ≤30	MS/MSD	A
		Precision	Standards (% Rec) Laboratory-derived limits2,4,5,6-Tetrachloro-m-Xylene (30-150) Decachlorobiphenyl (PCB-209) (40-135)	Surrogates	A

[1]If information varies within an analytical group, separate by individual analyte.

Table 2.3a
QC Guidelines for Metals Soil Samples

Sampling Procedure	Analytical Method	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)
12-SSP-01 SOP for Soil Sampling 12-SBC-AKMW SOP for Soil Boring Collection	6010B/7471A	Accuracy/Bias /Contamination	<RL	Field Blanks	S
		Precision	≤ 35%	Field Duplicate	S
		Accuracy/Bias /Contamination	No target analytes > RL except for common laboratory contaminants as noted in the SOP	Preparation Blank	A
		Accuracy/Bias /Contamination	No target analytes	Calibration Blanks	A
		Accuracy/Bias	Laboratory-derived limits 80-120%	LCS	A
		Accuracy/Bias	Laboratory-derived limits 75-125%	MS	A
		Precision	Laboratory-derived limits RPD ≤20%	MSD	A
		Accuracy/Bias	%D ≤10 or data is flagged	Serial Dilution	A

[1]If information varies within an analytical group, separate by individual analyte.

Table 2.3b QC Guidelines for Metals Groundwater Samples					
Sampling Procedure	**Analytical Method**	**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)**
12-GWS-01 SOP for Groundwater Sampling	6010B/7470A	Accuracy/Bias /Contamination	<RL	Field Blanks	S
		Precision	≤ 25%	Field Duplicate	S
		Accuracy/Bias /Contamination	No target analytes > RL except for common laboratory contaminants as noted in the SOP	Preparation Blank	A
		Accuracy/Bias /Contamination	No target analytes	Calibration Blanks	A
		Accuracy/Bias	Laboratory-derived limits 80-120%	LCS	A
		Accuracy/Bias	Laboratory-derived limits 75-125120%	MS	A
		Precision	Laboratory-derived limits RPD ≤20 %	MSD	A
		Accuracy/Bias	%D ≤10 or data is flagged	Serial Dilution	A

[1]If information varies within an analytical group, separate by individual analyte.

3.1 Precision

3.1.1 Definition

Precision is defined as the degree of mutual agreement of independent measurements as a result of repeated application of a process under specific conditions. Precision is concerned with the closeness of results during repeated analysis of the same sample. Precision is addressed by calculating the relative percent difference (RPD) for each pair of duplicate analyses.

3.1.2 Precision Objectives

Precision objectives for each analytical method in terms of the RPD of duplicate measurements have been specified for laboratory measurements in Tables 2.1a through 2.3b. The following QC samples may be used to evaluate precision in the laboratory:

- Laboratory duplicates;
- Reagent water blank spike duplicates; or
- Matrix spike duplicates.

3.2 Accuracy

3.2.1 Definition

Accuracy is defined as the degree of agreement of a measured value with the true or expected value.

3.2.2 Accuracy Objectives

Accuracy objectives for each analytical method in terms of the percent recovery (%R) from analysis of samples of known analyte concentration are presented in each respective laboratory QAPP. The following QC samples will be analyzed to evaluate accuracy in laboratory measurements:

- Laboratory control samples;
- Matrix spikes; or
- Reagent water blank spikes.

3.3 Completeness

Completeness is defined as a measure of the amount of valid data obtained from a measurement system compared to the amount that is expected under the conditions of the measurement. At least 90 percent of all samples analyzed by the laboratory must be valid to be considered complete.

3.4 Representativeness

Representativeness expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. As such, representativeness describes whether samples collected, or the aliquot(s) selected by the laboratory for analysis, are sufficient in number, type, location, frequency, and size to be characteristic of the substance analyzed.

Representativeness is a qualitative parameter that is dependent upon the proper design of the sampling program and proper laboratory protocol. The project sampling plan was designed to provide data representative of site conditions. Representativeness will be met by ensuring that work specifications are followed, proper sampling techniques are used, and proper analytical procedures are followed.

4.0 SAMPLING PROCEDURES

This section presents the field data collection methods and procedures to be implemented during the planned investigations. The procedures to be used during the investigations were chosen for their ability to provide data representative of site conditions, compatibility with analytical considerations, practicality, and simplicity.

The specific SOPs to be used are listed below:

SOP 07-DOC-00: Field Documentation

SOP 07-FQC-00: Field Quality Control

SOP 07-PAS-00: Sample Packing and Shipping

SOP 07-DCN-00: Sampling Equipment and Container Decontamination

SOP 12-SSP-01: Soil Sampling in the Field

SOP 12-GWS-01: Groundwater Sampling

SOP 12-MWI-AKMW: Groundwater Monitoring Well Installation and Development, Site Specific to AK Steel Middletown Works

SOP 12-PID-AKMW: Field Screen of Samples using Photo-Ionization Detector, Site Specific to AK Steel Middletown Works

SOP 12-SBC-AKMW: Soil Boring Collection, Site Specific to AK Steel Middletown Works

4.1 Sample Containers, Preservatives, and Volume Requirements

The required sample container, preservatives, and volume for all analyses will be collected in accordance with each respective laboratory QAPP. The volume of sample collected must be sufficient to perform the required analyses with an additional amount for QC needs (MS/MSDs, laboratory duplicates, etc.). All sample containers used for the collection of samples will be pre-cleaned by the manufacturer or by the laboratory supplying the sample bottles, as appropriate. Refer to Table 3 for a list of target compounds, laboratory analytical methods, sample containers, and holding times.

4.2 Field Equipment Decontamination

To minimize decontamination requirements and to prevent cross-contamination, disposable equipment (e.g., gloves, core liners) will be used whenever possible. Decontamination procedures will include (1) removing any gross contamination and placing into a container; (2) washing any reusable sampling equipment with a clean scrub brush and a mixture of distilled water and laboratory-grade detergent; (3) rinsing the equipment with distilled water (in a separate bucket); and (4) rinsing the equipment with a final rinse of laboratory-grade, analyte-free water. Decontamination fluids will be collected for proper disposal. Decontaminated utensils will be kept in their appropriate equipment containers while not in use to reduce the potential for contamination.

All sampling equipment decontamination will be performed in the field. When possible, equipment will be decontaminated in batches at a central staging area. All non-disposable equipment will be decontaminated prior to use, following use, between each sampling location, and prior to storage.

4.3 Sample Packaging and Shipping Procedures

Samples will be shipped under proper chain-of-custody, via overnight delivery by common carrier or delivered in person to the laboratory. Samples will be maintained at 4°C (± 2°C) or received with intact ice. All coolers will be sealed with packing tape, and custody seals will be attached to coolers shipped by common courier (e.g., Federal Express, United Parcel Service, etc.). Glass sample bottles will be wrapped in plastic bubble pack for protection from breakage. Other standard packing materials may also be used. Sample containers will be arranged to minimize movement when shipping to prevent contact

between containers. Sample packing and shipping procedures are further discussed in KEMRON SOP 07-PAS-00.

4.4 Sample Identification

Sample labels are necessary to identify the samples. The labels shall be affixed to the sample container (not to the caps) prior to or at the time of sampling. Pertinent information on the labels shall be filled out in indelible ink at the time of collection and will include the following information:

- Sample number/identification code;
- Name (initials) of collector;
- Date and time of collection;
- Project title and/or project number;
- KEMRON;
- Required analyses;
- Analysis method;
- Sample matrix; and
- Preservative.

After collection, all samples will be handled as little as possible. Field personnel will use extreme care to maintain the integrity of the samples. If samples are placed in a cooler with wet ice, personnel will package the ice so that melted ice will not leak into the sample containers or cause cross contamination.

Table 3
Summary of Sample Container and Sample Preservation Requirements
AK Steel SWMU 23 and AAs

Parameter	Sample Container		Preservation Methods	Holding Times
	Quantity	Type		
Solid Samples				
Volatile Organic Compounds	3	5-g EnCore or TerraCore samplers, zero headspace	Cool to 4 ± 2°C	48 hours from collection date/time (must be analyzed within 48 hours or transferred to soil vial within 48 hours)
Aroclors	2	250-mL wide-mouth glass container, Teflon®-lined cap	Cool to 4 ± 2°C	Extraction: 14 days from collection date Analysis: 40 days from extraction date
Metals*	1	200-g polyethylene or glass container, Teflon®-lined cap	Cool to 4 ± 2°C	6 months
Mercury*	1	200-g polyethylene or glass container, Teflon®-lined cap	Cool to 4 ± 2°C	28 days
Aqueous Samples				
Volatile Organic Compounds	3	40-mL glass vials, Teflon®-lined septum cap	HCl to pH <2, Cool to 4 ± 2°C	14 days
Aroclors	2	1-liter narrow mouth amber glass, Teflon®-lined cap	Cool to 4 ± 2°C	Extraction: 7 days from collection date Analysis: 40 days from extraction date
Metals	1	500 mL polyethylene container	HNO_3 to pH <2, Cool to 4 ± 2°C	6 months
Mercury	1	500 mL polyethylene container	HNO_3 to pH <2, Cool to 4 ± 2°C	28 days

Notes:
*= Metals and mercury sample for soil will be combined into one 500-mL bottle. Metals and mercury sample for water will be combined into one 500-mL bottle.
**= PCB aqueous samples limited to QC samples. Two 1-liter containers are sufficient for each sample PCB analyses.
VOC = Volatile Organic Compound
mL = milliliter
°C= Celsius
g = gram
HCL = Hydrochloric Acid
HNO_3 = Nitric Acid

5.0 SAMPLE IDENTIFICATION

Custody is a vital aspect of sampling procedures. The data and samples must be traceable from the time of sample collection, through sample analyses, data generation, and report preparation.

5.1 Field Records

All field activities will be recorded at the time of the activity in appropriate log sheets and/or field notebooks. Several different types of records may be created during the sampling event. All records will be prepared in indelible ink, dated and signed by the author, and any errors will be corrected by drawing a single line through the error and initialing and dating the error. No pages should be removed from the field notebook. Records of photo documentation and sample location description (i.e., distance measurement, latitude and longitude coordinates) will be maintained. All field data records, including sampling logs, equipment calibration specifications, laboratory task orders (if needed), and chain-of-custody forms, will be reviewed for accuracy and completeness. All records will be maintained in the project file as a permanent record.

5.2 Sample Container Custody

All containers will be shipped from the supplier to the KEMRON office or a location designated by the KEMRON Project Manager by common carrier in sealed packages or picked up in person from the laboratory. The supplier will include a shipping form or laboratory chain-of-custody listing all containers shipped and the purpose of each container. It is KEMRON's policy that all containers are considered to be in the custody of the supplier until the sample containers are received by KEMRON. Upon receipt, sample containers will be checked to determine if any breakage occurred during shipment. The containers will be maintained in the custody of the receiver in a clean, secure area until they are used for sample collection. Once samples have been collected, they will be maintained in the custody of the sampling personnel. The procedure for maintaining and documenting chain-of-custody in the field is described in KEMRON SOP 07-DOC-00 and will be as follows:

1. The sample label that is affixed to the sample container will be inspected to confirm that all of the required information has been provided (See Section 4).

2. The sample container will be sealed in a self-sealing (e.g., zip-lock®) plastic bag or wrapped in bubble pack, and packed in a cooler chest in a manner that will minimize movement. All samples will be cooled with double-bagged wet ice.

3. For each cooler chest that is sent to the laboratory, a Chain-of-Custody Record will be completed. All information on the Chain-of-Custody Record and the sample container labels will be checked against field records and the samples will be recounted.

4. If a commercial courier service (e.g., Federal Express) is being used to transport the samples to the laboratory, the Chain-of-Custody Record will be signed by a member of the field team, and a copy of the Record will be retained by the field team. The remaining copies of the Record will be sealed in a self-sealing plastic bag and placed in the cooler chest with the samples. The cooler chest will be sealed with packaging tape and two custody seals that have been signed and dated by a member of the field team. Because commercial couriers will not sign chain-of-custody forms, the package routing documentation maintained by the courier service during shipping serves as chain-of-custody documentation for the samples.

All laboratory analysts and technicians must follow chain-of-custody protocols described in the respective laboratory's QAPP, which will be provided upon request.

5.3 Data Archiving and Project File

A project file will be developed for data generated during this investigation and will include the following items: all reports, field notes, laboratory reports, signed chain-of-custody forms, sampling procedures, and any other pertinent documents. These items will be stored in the KEMRON office responsible for the project with access limited to select project personnel. The file custodian will be the KEMRON Project Manager or her designee. All documents used in decision making will be maintained at Middletown Works. The project file will be maintained for a period of at least five (5) years after termination of the Consent Decree.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

This section describes procedures for calibrating all field and laboratory instruments used for conducting field measurements and laboratory analyses.

6.1 Field Instrument Calibration

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications. Equipment to be used during the field sampling will be examined prior to conducting the field investigation to certify that it is in operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to ensure that all maintenance requirements are being observed.

Calibration of field instruments is governed by the manufacturers' specific SOP for the applicable field analysis method. Calibration and calibration checks of field instruments will be in accordance with the procedures described in the appropriate SOPs. Calibration will be performed at the intervals specified by the manufacturer, or more frequently if conditions dictate. In the event that an internally calibrated field instrument fails to meet calibration/check-out procedures, it will be returned to the manufacturer for service and an alternate back-up instrument will be made available.

6.2 Laboratory Instrument Calibration

The calibration procedures and calibration frequency employed by the laboratory will be in accordance with the analytical methods specified in the respective analytical method SOPs as supplied by the laboratories and located in Appendix A. Records of instrument calibration, repairs, and replacement activities will be maintained. These records will be filed at the location where the work is performed and will be subject to QA audit at any time. Project requirements are provided in Tables 4.1 through 4.3.

Table 4-1
Calibration Requirements and Corrective Actions for Method 8260B
AK Steel SWMU 23 and AAs

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Where calibration criteria and/or corrective actions are inconsistent with the laboratory's most current SOP, provisions in the SOP will apply.						
Initial Calibration 5-pt curve	Prior to sample analysis Set-up, major maintenance, and quarterly	RRF > 0.01 (Advisory), RRF > 0.10 for SPCCs; except chlorobenzene and 1,1,2,2-TCA > 0.30. RSD ± 15%; RSD ± 30% for CCCs response factors. Lab may use first or higher order regression fit (r ≥ 0.995) if %RSD > 15%.	If RSD of the average RRF for calibration check compounds > 30%, the initial calibration must be repeated. Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Lab Representative	Linear response	RRF > 0.01 (Advisory), RRF > 0.10 for SPCCs; except chlorobenzene and 1,1,2,2-TCA > 0.30. RSD ±15%; RSD ± 30% for CCCs response factors. Lab may use first or higher order regression fit (r ≥ 0.995) if %RSD > 15%.
Continuing calibration check	Daily before sample analysis, and every 12 hours	%Difference for RF of CCCs of continuing calibration compounds ±20% from initial calibration. RRF > 0.01 (Advisory), RRF > 0.10 for SPCCs; except chlorobenzene and 1,1,2,2-TCA > 0.30.	Samples cannot begin until this criterion is met. Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Lab Representative	System control	%Difference for RF of CCCs of continuing calibration compounds ±20% from initial calibration. RRF > 0.01 (Advisory), RRF > 0.10 for SPCCs; except chlorobenzene and 1,1,2,2-TCA > 0.30.

Table 4-1
Calibration Requirements and Corrective Actions for Method 8260B
AK Steel SWMU 23 and AAs

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 blanks	Every 12 hours	No analytes detected ≥ RL		Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank. Document source of contamination.	Lab Representative	System contamination	No analytes detected ≥ RL
Tuning BFB	Prior to calibration	Must meet tuning criteria.		Re-tune, re-calibrated.	Lab Representative	Ion control	Must meet tuning criteria.
LCS	Every batch	QC limits specified in Tables 2.1a and 2.1b.		Qualify associated data biased high or biased low as appropriate.	Lab Representative	Compound recovery	Specified QC limits
Internal Standards	Every sample	Standards Fluorobenzene Chlorobenzene-D5 1,4-Dichlorobenzene-D	Retention time ±30 seconds of last CC Area changes by a factor of two (-50% to +100%)	Inspect for malfunction. Demonstrate that system is functioning properly. Reanalyze samples with	Lab Representative	System response	Retention time ±30 seconds of last CC Area changes by a factor of two (-50% to +100%)

Table 4-1
Calibration Requirements and Corrective Actions for Method 8260B
AK Steel SWMU 23 and AAs

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits			Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
					standards outside criteria.			
Surrogate	Every Sample	Standards Dibromofluoromethane Toluene-D8 4-Bromofluorobenzene 1,2-DCA-D4	Solid 37-132 67-125 52-136 58-123	Aqueous 75-121 74-115 66-117 63-129	If any surrogate compounds do not meet criteria, there should be a re-analysis to confirm that the non-compliance is due to the sample matrix effects rather than laboratory deficiencies.	Lab Representative	Recovery	Solid 37-132 67-125 52-136 58-123 Aqueous 75-121 74-115 66-117 63-129
Matrix Spike and Duplicate	1 per 20 per matrix	QC limits specified in Tables 2.1a and 2.1b.			If MS/MSD results do not meet criteria, the reviewer should review the data in conjunction with other QC results to determine if the problem is specific to the	Lab Representative	Matrix specific recovery	Specified QC limits

Table 4-1
Calibration Requirements and Corrective Actions for Method 8260B
AK Steel SWMU 23 and AAs

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			QC samples or systematic.			
Duplicate Sample	10% of field samples per matrix	RPD ≤ 25% aqueous; ≤ 35% solid	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Precision	To ensure precision in sample homogeneity during collection and analysis
Rinse Blank	1 per 20 samples or 1 per day	No target analytes	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Accuracy, Representativeness	To ensure the decontamination of sampling equipment has been adequately performed; to assess cross contamination and/or incidental contamination to the sample container.
Temperature Blank	1 temperature blank per cooler.	Temperatures inside cooler are within acceptable range upon arrival at the laboratory.	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader		To verify sample cooler temperature during transport.
Trip Blank	1 trip blank per cooler of VOA	No target analytes, volatiles in water.	Data reviewer should review	Project Manager/Field	Accuracy, Representativ	To determine if cross contamination occurs

Table 4-1
Calibration Requirements and Corrective Actions for Method 8260B
AK Steel SWMU 23 and AAs

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
	samples		and judge all of the target compounds against the acceptance criteria.	Team Leader	eness	during shipment or storage with volatile organic analyses (VOA) samples.
Source Water	1 per source	No target analytes	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Representativ eness	To characterize decontamination water source; not applicable if potable water is used in decontamination or laboratory grade deionized water is used for decontamination rinse.

Table 4-2
Corrective Actions for Method 8082
AK Steel SWMU 23 and AAs

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Where calibration criteria and/or corrective actions are inconsistent with the laboratory's current SOP, provisions in the SOP will apply.						
Initial calibration curve 5-pt curve	Prior to sample analysis Set-up, major maintenance	linear - mean RSD for all analytes ≤20% with no individual analyte RSD >30%	Must meet criteria prior to sample analysis	Lab Representative	Linear response	linear - mean RSD for all analytes ≤20% with no individual analyte RSD >30%
Continuing calibration (calibration check)	Daily before sample analysis, and After every 10 samples and at the end of the analysis sequence	%D recovery ± 15% of the response factor from the initial curve.	If criteria are not met, reanalyze the daily standard. If the daily standard fails a second time, initial calibration must be repeated.	Lab Representative	System control	%D recovery ± 15% of the response factor from the initial curve.
Independent reference standard (LCS)	1 per batch	QC limits specified in Tables 2.2a and 2.2b.	Qualify associated data biased high or biased low as appropriate.	Lab Representative	Recovery	Specified QC Limits
Method blanks	1 per batch	No target analytes detected ≥ Reporting Limits	Correct problem then reprep and analyze	Lab Representative	System contamination	No target analytes detected ≥ Reporting

Table 4-2
Corrective Actions for Method 8082
AK Steel SWMU 23 and AAs

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 and all samples processed with the contaminated blank. Document source of contamination.			Limits
Surrogate spikes	Every sample	Standards DCBP TCMX	Aqueous %R 10-130% 23-136%	Solid %R 14-163% 29-151%	If any surrogate compounds do not meet criteria, there should be a re-analysis to confirm that the non-compliance is due to the sample matrix effects rather than laboratory deficiencies.	Lab Representative	Recovery	Aqueous %R 10-130% 23-136% Solid %R 14-163% 29-151%
Matrix spike and matrix spike duplicate (1016/1260 mix)	1 per 20 samples per matrix	Standards PCB-1016 PCB-1260	Aqueous %R 67-120 30–120 RPD	Solid %R 22-157 13-161 RPD	Data reviewer may use the MS and MSD results in conjunction with other QC sample results	Lab Representative	Matrix specific recovery	QC Limits

Table 4-2
Corrective Actions for Method 8082
AK Steel SWMU 23 and AAs

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits			Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			≤ 30 for all	≤30 for all	to determine the need for some qualification of the data.			
Duplicate Sample	10% of field samples per matrix	RPD ≤ 25% aqueous; ≤ 35% solid			Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Precision	To ensure precision in sample homogeneity during collection and analysis
Rinse Blank	1 per 20 samples or 1 per day	No target analytes			Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Accuracy, Representativeness	To ensure the decontamination of sampling equipment has been adequately performed; to assess cross contamination and/or incidental contamination to the sample container.
Temperature Blank	1 temperature blank per cooler.	Temperatures inside cooler are within acceptable range upon arrival at the			Data reviewer should review	Project Manager/Field		To verify sample cooler

Table 4-2
Corrective Actions for Method 8082
AK Steel SWMU 23 and AAs

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
		laboratory.	and judge all of the target compounds against the acceptance criteria.	Team Leader		temperature during transport.
Source Water	1 per source	No target analytes	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Representativeness	To characterize decontamination water source; not applicable if potable water is used in decontamination or laboratory grade deionized water is used for decontamination rinse.

Table 4-3
Corrective Actions for Methods 6010B and 7470A/7471A
AK Steel SWMU 23 and AAs

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Where calibration criteria and/or corrective actions are inconsistent with the laboratory's current SOP, provisions in the SOP will apply.						
Initial calibration curve (3-pt curve Hg) (1-pt curve ICP)	Daily or major maintenance, instrument modification, replacement of the torch, replacement of the mirror	r > 0.995 for all elements r: linear correlation coefficient	If r < 0.995 for any element, the standards for that element must be prepared again and/or the lower/upper range standard must be used.	Lab Representative	Linear response	r > 0.995 for all elements r: linear correlation coefficient
Continuing calibration verification (CCV)	Every 10 samples or 2 per 8 hr and end of run.	Recovery ±10% of true value for ICP Recovery ±20% of true value for Hg	Reanalyze CCV. If the CCV fails second time, the terminate analysis, correct problem, re-calibrate instrument, and re-verify calibration prior to continuing sample analyses.	Lab Representative	System control	Recovery ±10% of true value for ICP Recovery ±20% of true value for Hg
Interference check	Beginning of each sample analytical run.	Recovery ±20% of true value.	Terminate the analysis, correct the	Lab Representative	System control	Recovery ±20% of true

Table 4-3
Corrective Actions for Methods 6010B and 7470A/7471A
AK Steel SWMU 23 and AAs

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			problem, re-calibrate, re-verify the calibration, and reanalyze the samples.			value.
Continuing calibration blank (CCB)	Every 10 samples, end of analytical run	Concentration < 3 X the background mean (ICP). No target analytes.	If the average in not within criteria, terminate the analysis, correct the problem, re-calibrate, and reanalyze all samples analyzed since the last acceptable CCB.	Lab Representative	System contaminat ion	Concentration < 3 X s of the background mean (ICP). No target analytes.
Serial Dilution (ICP)	1 per 20 samples per matrix for samples > 50x Method Detection Limit (MDL)	Difference between diluted and undiluted sample ≤10%.	Chemical or physical interference should be suspected. Investigate to determine cause.	Lab Representative		Difference between diluted and undiluted sample ≤10%.

Table 4-3
Corrective Actions for Methods 6010B and 7470A/7471A
AK Steel SWMU 23 and AAs

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Preparation blank	1 per batch per matrix	No target analytes > RL except for common laboratory contaminants as noted in the SOP .	Documented source of contamination.	Lab Representative	System contamination	No target analytes.
Laboratory Control Sample	1 per analytical batch	80%≤%Rec.≤120%	Qualify associated data biased high or biased low as appropriate.	Lab Representative	Recovery	80%≤%Rec.≤ 120%
Matrix spike and matrix spike duplicate	1 per analytical batch	75%≤%Rec.≤125%; %RPD<20%; If spike(s) outside of limits, analyze PDS.	If matrix spike recovery does not meet criteria (except Ag), a post digestion spike is required for all methods except GFAA. Qualify results in accordance with Regional criteria.	Lab Representative	Matrix specific recovery	75%≤%Rec.≤ 125%; %RPD<20%; If spike(s) outside of limits, analyze PDS.
Duplicate Sample	10% of field samples per matrix	RPD ≤ 25% aqueous; ≤ 35% solid	Data reviewer should review and judge all of the target compounds against the	Project Manager/Field Team Leader	Precision	To ensure precision in sample homogeneity during collection and

Table 4-3
Corrective Actions for Methods 6010B and 7470A/7471A
AK Steel SWMU 23 and AAs

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			acceptance criteria.			analysis
Rinse Blank	1 per 20 samples or 1 per day	No target analytes	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Accuracy, Represent ativeness	To ensure the decontaminati on of sampling equipment has been adequately performed; to assess cross contamination and/or incidental contamination to the sample container.
Temperature Blank	1 temperature blank per cooler.	Temperatures inside cooler are within acceptable range upon arrival at the laboratory.	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader		To verify sample cooler temperature during transport.

Table 4-3
Corrective Actions for Methods 6010B and 7470A/7471A
AK Steel SWMU 23 and AAs

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Source Water	1 per source	No target analytes	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Represent ativeness	To characterize decontaminati on water source; not applicable if potable water is used in decontaminati on or laboratory grade deionized water is used for decontaminati on rinse.

7.0 ANALYTICAL PROCEDURES

The laboratory designated to perform sample analyses is presented (including the laboratory address and phone number) in Section 2.3 of this QAPP. TestAmerica is responsible for groundwater and soil sample analyses of VOCs using USEPA Method 8260B, PCB-Aroclors using USEPA Method 3520/8082 (fluids) and 3541 (or 3540)/8082 (solids), total and dissolved metals using USEPA Method 3005A/6010B (fluids) and 3050B/6010B (solids), and total and dissolved mercury using USEPA Method 7470/71.

The analytical methods and laboratory-specific SOPs are presented in Appendix A. Laboratory-determined reporting limits are presented in Table 5.1a.

Table 5.1a
Summary of Reporting Limits and Data Quality Levels for VOCs
Soil Samples
AK Steel SWMU 23 and AAs

Analyte	Analytical Method	USEPA RSLs[2]		USEPA Region 5 ESLs (ug/kg)	Achievable Laboratory Limits[1]	
		Residential Soil (ug/kg)	Industrial Soil (ug/kg)		MDLs (ug/kg)	RLs (ug/kg)
Acetone	8260B	61,000	630,000,000	2,500	6.3	20
Benzene	8260B	1,000	5,400	255	0.23	5
Bromobenzene	8260B	300,000	1,800,000	None Available	0.33	5
Bromochloromethane	8260B	160,000	680,000	None Available	0.71	5
Bromodichloromethane	8260B	270	1,400	540	0.28	5
Bromoform	8260B	62,000	220,000	15,900	0.33	5
Bromomethane	8260B	7,300	32,000		0.54	5
2-Butanone (MEK)	8260B	28,000,000	200,000,000	89,600	1.4	20
Tert-Butylbenzene	8260B	None Available	None Available	None Available	0.29	5
Sec-Butylbenzene	8260B	None Available	None Available	None Available	0.18	5
n-Butylbenzene	8260B	3,900,000	51,000,000	None Available	0.23	5
Carbon Disulfide	8260B	820,000	3,700,000	94.1	0.44	5
Carbon Tetrachloride	8260B	610	3,000	2,980	0.37	5
Chlorobenzene	8260B	290,000	1,400,000	13,100	0.33	5
Chloroethane	8260B	None Available	None Available	None Available	0.86	5
Chloroform	8260B	290	1,500	1,190	0.29	5
Chloromethane (Methylene Chloride)	8260B	56,000	960,000	4,050	0.41	5

Table 5.1a
Summary of Reporting Limits and Data Quality Levels for VOCs
Soil Samples
AK Steel SWMU 23 and AAs

Analyte	Analytical Method	USEPA RSLs[2]		USEPA Region 5 ESLs (ug/kg)	Achievable Laboratory Limits[1]	
		Residential Soil (ug/kg)	Industrial Soil (ug/kg)		MDLs (ug/kg)	RLs (ug/kg)
4-Chlorotoluene	8260B	1,600,000	20,000,000	None Available	0.51	5
2-Chlorotoluene	8260B	1,600,000	20,000,000	None Available	0.4	5
Dibromochloromethane	8260B	680	3,300	2,050	0.55	5
Dibromomethane	8260B	25,000	110,000	None Available	0.63	5
1,2-Dichlorobenzene	8260B	1,900,000	9,800,000	2,960	0.36	5
1,4-Dichlorobenzene	8260B	2,400	12,000	546	0.66	5
1,3-Dichlorobenzene	8260B	None Available	None Available	37,700	0.35	5
Dichlorodifluoromethane	8260B	94,000	400,000	39,500	0.5	5
1,1-Dichloroethane	8260B	3,300	17,000	20,100	0.36	5
1,2-Dichloroethane	8260B	430	2,200	21,200	0.34	5
Cis-1,2-Dichloroethene	8260B	160,000	2,000,000	None Available	0.36	5
Trans-1,2-Dichloroethene	8260B	150,000	690,000	784	0.41	5
1,1-Dichloroethene	8260B	240,000	1,100,000	8,280	0.52	5
1,3-Dichloropropane	8260B	1,600,000	20,000,000	None Available	0.34	5
2,2-Dichloropropane	8260B	None Available	None Available	None Available	0.94	5
1,2-Dichloropropane	8260B	940	4,700	32,700	0.69	5
1,1-Dichloropropene	8260B	None Available	None Available	None Available	0.3	5
Cis-1,3-Dichloropropene	8260B	1,700	8,300	398	0.34	5
Trans-1,3-Dichloropropene	8260B	1,700	8,300	398	0.54	5
Ethylbenzene	8260B	5,400	27,000	5,160	0.26	5
Hexachlorobutadiene	8260B	6,200	22,000	39.8	1.2	5
n-Hexane	8260B	570,000	2,600,000	None Available	1.2	5
Isopropylbenzene	8260B	2,100,000	11,000,000	None	0.16	5

Table 5.1a
Summary of Reporting Limits and Data Quality Levels for VOCs
Soil Samples
AK Steel SWMU 23 and AAs

Analyte	Analytical Method	USEPA RSLs[2]		USEPA Region 5 ESLs (ug/kg)	Achievable Laboratory Limits[1]	
		Residential Soil (ug/kg)	Industrial Soil (ug/kg)		MDLs (ug/kg)	RLs (ug/kg)
(Cumene)				Available		
p-Isopropyltoluene	8260B	None Available	None Available	None Available	0.21	5
Methyl butylketone (2-Hexanone)	8260B	None Available	None Available	None Available	0.63	20
Methyl tert-butyl ether	8260B	43,000	220,000	None Available	0.43	20
4-Methyl-2-pentanone (MIBK)	8260B	5,300,000	53,000,000	None Available	0.54	20
Naphthalene	8260B	3,600	18,000	99.4	0.19	5
N-Propylbenzene	8260B	3,400,000	21,000,000	None Available	0.4	5
Styrene	8260B	6,300,000	36,000,000	4,690	0.15	5
1,1,1,2-Tetrachloroethane	8260B	1,900	9,300	22,500	0.62	5
1,1,2,2-Tetrachloroethane	8260B	560	2,800	127	0.34	5
Tetrachloroethene	8260B	22,000	110,000	9,920	0.52	5
Toluene	8260B	5,000,000	45,000,000	5,450	0.27	5
1,2,4-Trichlorobenzene	8260B	22,000	99,000	11,100	0.27	5
1,1,1-Trichloroethane	8260B	8,700,000	38,000,000	29,800	0.56	5
1,1,2-Trichloroethane	8260B	1,100	5,300	28,600	0.39	5
Trichloroethene	8260B	910	6,400	12,400	0.42	5
Trichlorofluoromethane	8260B	790,000	3,400,000	16,400	0.34	5
1,2,4-Trimethylbenzene	8260B	62,000	260,000	None Available	0.65	5
1,3,5-Trimethylbenzene	8260B	780,000	10,000,000	None Available	0.25	5
Vinyl Acetate	8260B	970,000	4,100,000	12,700	0.25	10
Vinyl Chloride	8260B	60	1,700	646	0.39	5
Xylenes, Total	8260B	630,000	2,700,000	10,000	1.8	15

[1]Achievable MDLs and RLs are limits that an individual laboratory can achieve when performing a specific analytical method. Laboratory MDLs are statistically derived limits and may be subject to change over the course of a project. Reporting limits presented are based upon wet weight. The reporting limits presented by the laboratory in the analytical report will be calculated based upon dry weight and will be higher and vary slightly from sample to sample.
[2]Detectable concentrations will be screened against the USEPA Regional Screening Levels from the November 2012 Summary Table.

Table 5.1b Summary of Reporting Limits and Data Quality Levels for VOCs Groundwater Samples AK Steel SWMU 23 and AAs						
Analyte	Analytical Method	USEPA RSLs[2]		USEPA Region 5 ESLs (ug/L)	Achievable Laboratory Limits[1]	
		MCL (ug/L)	TapWater (ug/L)		MDLs (ug/L)	RLs (ug/L)
Acetone	8260B	None Available	12,000	1,700	1.1	10
Benzene	8260B	5.0	0.39	114	0.13	1
Bromobenzene	8260B	None Available	54	None Available	0.13	1
Bromochloromethane	8260B	None Available	83	None Available	0.29	1
Bromodichloromethane	8260B	80 (TTHMs)	0.12	None Available	0.15	1
Bromoform	8260B	80 (TTHMs)	7.9	230	0.64	1
Bromomethane	8260B	None Available	7.0	None Available	0.41	1
2-Butanone (MEK)	8260B	None Available	4,900	2,200	0.57	10
Tert-Butylbenzene	8260B	None Available	None	None Available	0.13	1
Sec-Butylbenzene	8260B	None Available	None	None Available	0.13	1
n-Butylbenzene	8260B	None Available	780	None Available	0.12	1
Carbon Disulfide	8260B	None Available	720	15	0.13	1
Carbon Tetrachloride	8260B	5.0	0.39	240	0.13	1
Chlorobenzene	8260B	100	72	47	0.15	1
Chloroethane	8260B	None Available	None	None Available	0.29	1
Chloroform	8260B	80 (TTHMs)	0.19	140	0.16	1
Chloromethane (Methylene Chloride)	8260B	5.0	9.9	940	0.3	1
4-Chlorotoluene	8260B	None Available	190	None Available	0.18	1
2-Chlorotoluene	8260B	None Available	180	None Available	0.11	1
Dibromochloromethane	8260B	80 (TTHMs)	0.15	None Available	0.18	1
Dibromomethane	8260B	None Available	7.9	None Available	0.28	1

Table 5.1b
Summary of Reporting Limits and Data Quality Levels for VOCs
Groundwater Samples
AK Steel SWMU 23 and AAs

Analyte	Analytical Method	USEPA RSLs[2]		USEPA Region 5 ESLs (ug/L)	Achievable Laboratory Limits[1]	
		MCL (ug/L)	TapWater (ug/L)		MDLs (ug/L)	RLs (ug/L)
1,2-Dichlorobenzene	8260B	600	280	14	0.13	1
1,4-Dichlorobenzene	8260B	75	0.42	9.4	0.13	1
1,3-Dichlorobenzene	8260B	None Available	None Available	38	0.14	1
Dichlorodifluoromethane	8260B	None Available	190	None Available	0.31	1
1,1-Dichloroethane	8260B	None Available	2.4	47	0.15	1
1,2-Dichloroethane	8260B	5.0	0.15	910	0.22	1
Cis-1,2-Dichloroethene	8260B	70	28	None Available	0.17	1
Trans-1,2-Dichloroethene	8260B	100	86	970	0.19	1
1,1-Dichloroethene	8260B	7.0	260	65	0.19	1
1,3-Dichloropropane	8260B	None Available	290	None Available	0.16	1
2,2-Dichloropropane	8260B	None Available	None Available	None Available	0.13	1
1,2-Dichloropropane	8260B	5.0	0.38	360	0.18	1
1,1-Dichloropropene	8260B	None Available	None Available	None Available	0.13	1
Cis-1,3-Dichloropropene	8260B	None Available	0.41	None Available	0.14	1
Trans-1,3-Dichloropropene	8260B	None Available	0.41	None Available	0.19	1
Ethylbenzene	8260B	700	1.3	14	0.17	1
Hexachlorobutadiene	8260B	None Available	0.26	0.053	0.3	1
n-Hexane	8260B	None Available	250	None Available	0.26	1
Isopropylbenzene (Cumene)	8260B	None Available	390	None Available	0.13	1
p-Isopropyltoluene	8260B	None Available	None Available	None Available	0.12	1
Methyl butylketone (2-Hexanone)	8260B	None Available	None Available	None Available	0.41	10
Methyl tert-butyl ether	8260B	None Available	12	None	0.17	5

Table 5.1b
Summary of Reporting Limits and Data Quality Levels for VOCs
Groundwater Samples
AK Steel SWMU 23 and AAs

Analyte	Analytical Method	USEPA RSLs[2]		USEPA Region 5 ESLs (ug/L)	Achievable Laboratory Limits[1]	
		MCL (ug/L)	TapWater (ug/L)		MDLs (ug/L)	RLs (ug/L)
				Available		
4-Methyl-2-pentanone (MIBK)	8260B	None Available	1,000	None Available	0.32	10
Naphthalene	8260B	None Available	0.14	13	0.24	1
N-Propylbenzene	8260B	None Available	520	None Available	0.14	1
Styrene	8260B	100	1,100	32	0.11	1
1,1,1,2-Tetrachloroethane	8260B	None Available	0.5	None Available	0.23	1
1,1,2,2-Tetrachloroethane	8260B	None Available	0.066	380	0.18	1
Tetrachloroethene	8260B	5.0	9.7	45	0.29	1
Toluene	8260B	1,000	860	253	0.13	1
1,2,4-Trichlorobenzene	8260B	70	0.99	30	0.15	1
1,1,1-Trichloroethane	8260B	200	7,500	76	0.22	1
1,1,2-Trichloroethane	8260B	5.0	0.24	500	0.27	1
Trichloroethene	8260B	5.0	0.44	47	0.17	1
Trichlorofluoromethane	8260B	None Available	1,100	None Available	0.21	1
1,2,4-Trimethylbenzene	8260B	None Available	15	None Available	0.12	1
1,3,5-Trimethylbenzene	8260B	None Available	87	None Available	0.096	1
Vinyl Acetate	8260B	None Available	410	248	0.19	2
Vinyl Chloride	8260B	2.0	0.015	930	0.22	1
Xylenes, Total	8260B	10,000	190	27	0.34	3

[1]Achievable MDLs and RLs are limits that an individual laboratory can achieve when performing a specific analytical method. Laboratory MDLs are statistically derived limits and may be subject to change over the course of a project.

[2]Detectable concentrations will be screening against the USEPA Regional Screening Levels from the November 2012 Summary Table.

Table 5.2a
Summary of Reporting Limits and Data Quality Levels for PCBs
Soil Samples
AK Steel SWMU 23 and AAs

Analyte	Analytical Method	USEPA RSLs[2]		USEPA Region 5 ESLs	Achievable Laboratory Limits[1]	
		Residential Soil (mg/kg)	Industrial Soil (mg/kg)	(mg/kg)	MDLs (mg/kg)	RLs (mg/kg)
Aroclor-1016	8082	3.9	21	12 Total PCBs	0.021	0.033
Aroclor-1221	8082	0.14	0.54	12 Total PCBs	0.016	0.033
Aroclor-1232	8082	0.14	0.54	12 Total PCBs	0.014	0.033
Aroclor-1242	8082	0.22	0.74	12 Total PCBs	0.013	0.033
Aroclor-1248	8082	0.22	0.74	12 Total PCBs	0.017	0.033
Aroclor-1254	8082	0.22	0.74	12 Total PCBs	0.017	0.033
Aroclor-1260	8082	0.22	0.74	12 Total PCBs	0.017	0.033

[1]Achievable MDLs and RLs are limits that an individual laboratory can achieve when performing a specific analytical method. Laboratory MDLs are statistically derived limits and may be subject to change over the course of a project. Reporting limits presented are based upon wet weight. The reporting limits presented by the laboratory in the analytical report will be calculated based upon dry weight and will be higher and vary slightly from sample to sample.

[2]Detectable concentrations will be screening against the USEPA Regional Screening Levels from the November 2012 Summary Table.

Table 5.2b
Summary of Reporting Limits and Data Quality Levels for PCBs
Water Samples
AK Steel SWMU 23 and AAs

Analyte	Analytical Method	Achievable Laboratory Limits[1]	
		MDLs (ug/L)	RLs (ug/L)
Aroclor-1016	8082	0.17	1.0
Aroclor-1221	8082	0.13	1.0
Aroclor-1232	8082	0.16	1.0
Aroclor-1242	8082	0.22	1.0
Aroclor-1248	8082	0.1	1.0
Aroclor-1254	8082	0.16	1.0
Aroclor-1260	8082	0.17	1.0

[1]Achievable MDLs and RLs are limits that an individual laboratory can achieve when performing a specific analytical method. Laboratory MDLs are statistically derived limits and may be subject to change over the course of a project.

NOTE: Aqueous PCB analysis only applicable to QC samples. Screening criteria, other than noting an analyte detection, are not applicable for this matrix.

Table 5.3a Summary of Reporting Limits and Data Quality Levels for Metals Soil Samples AK Steel SWMU 23 and AAs						
Analyte	Analytical Method	USEPA RSLs[2]		USEPA Region 5 ESLs (mg/kg)	Achievable Laboratory Limits[1]	
		Residential Soil (mg/kg)	Industrial Soil (mg/kg)		MDLs (mg/kg)	RLs (mg/kg)
Aluminum	6010B	77,000	990,000	None Available	20	9.5
Arsenic	6010B	0.39	1.6	5.7	0.3	1.0
Barium	6010B	15,000	190,000	1.04	0.071	20
Cadmium	6010B	70	800	0.00222	0.036	0.2
Chromium	6010B	0.29 (Cr VI)	5.6	0.400	0.2	0.5
Lead	6010B	400	800	0.0537	0.19	0.3
Selenium	6010B	390	5,100	0.0276	0.45	0.5
Silver	6010B	390	5,100	4.04	0.1	0.5
Zinc	6010B	23,000	310,000	6.62	2	1.0
Mercury	7471A	10	43	0.100	0.015	0.1

[1]Achievable MDLs and RLs are limits that an individual laboratory can achieve when performing a specific analytical method. Laboratory MDLs are statistically derived limits and may be subject to change over the course of a project. Reporting limits presented are based upon wet weight. The reporting limits presented by the laboratory in the analytical report will be calculated based upon dry weight and will be higher and vary slightly from sample to sample.

[2]Detectable concentrations will be screening against the USEPA Regional Screening Levels from the November 2012 Summary Table.

Table 5.3b Summary of Reporting Limits and Data Quality Levels for Metals Groundwater Samples AK Steel SWMU 23 and AAs						
Analyte	Analytical Method	USEPA RSLs[2]		USEPA Region 5 ESLs (mg/L)	Achievable Laboratory Limits[1]	
		MCL (mg/L)	Tapwater (mg/L)		MDLs (mg/L)	RLs (mg/L)
Aluminum	6010B	None Available	16	None Available	0.2	0.097
Arsenic	6010B	0.010	0.000045	0.148	0.0032	0.010
Barium	6010B	2.0	2.9	0.220	0.00067	0.200
Cadmium	6010B	0.005	0.0069	0.00015	0.00066	0.002

Table 5.3b
Summary of Reporting Limits and Data Quality Levels for Metals
Groundwater Samples
AK Steel SWMU 23 and AAs

Analyte	Analytical Method	USEPA RSLs[2]		USEPA Region 5 ESLs (mg/L)	Achievable Laboratory Limits[1]	
		MCL (mg/L)	Tapwater (mg/L)		MDLs (mg/L)	RLs (mg/L)
Chromium	6010B	0.1	None Available	0.042	0.0022	0.005
Lead	6010B	0.015	None Available	0.00117	0.0019	0.003
Selenium	6010B	0.050	0.078	0.005	0.0041	0.005
Silver	6010B	None Available	0.071	0.00012	0.0022	0.005
Zinc	6010B	None Available	4.7	0.066	1.0	0.005
Mercury	7470A	0.002	0.00063	0.0000013	0.00012	0.0002

[1]Achievable MDLs and RLs are limits that an individual laboratory can achieve when performing a specific analytical method. Laboratory MDLs are statistically derived limits and may be subject to change over the course of a project.

[2]Detectable concentrations will be screening against the USEPA Regional Screening Levels from the November 2012 Summary Table.

8.0 INTERNAL QUALITY CONTROL CHECKS

Internal quality control checks are a systematic procedure designed to monitor field and laboratory procedures in order to assure quality methods with legitimate results. These checks are in place to make certain that neither the field sampling techniques, nor the laboratory practices will influence the integrity of data.

8.1 Field Quality Control Samples

Field QC samples (refer to Table 6.1) are necessary to monitor both field and laboratory performance. Field QC samples provide a means of checking the validity of the sample results. These data will be periodically examined to determine if any problems are evident with specific types of media samples or with the procedures used by the subcontracted laboratories. Field QC samples to be collected during sampling activities include equipment rinseate blanks and field duplicate samples. One equipment rinseate blank will be collected per 10 samples, not to exceed more than one equipment rinseate blank each day during the field investigation. One field duplicate will be collected per 20 investigative samples per sample matrix.

The effectiveness of personnel sample-handling techniques may be monitored by submitting preserved equipment rinseate blank samples for laboratory analysis. These blank samples will be prepared by field personnel and treated in the same manner as actual samples. Rinseate blanks will be collected by pouring laboratory-grade, analyte-free water over a decontaminated piece of sampling equipment and catching the rinseate in a laboratory container. That collected water is preserved in the same manner as the field samples and analyzed for the parameters/analytes of investigation. Appropriate sample containers for each analyte group must be used. These blanks will be appropriately labeled and documented in field records. These blanks will be stored, transported, and analyzed with the investigative samples.

The precision of sample collection techniques may be monitored through the collection and analyses of field duplicate samples. This type of sample also may serve as a rough estimate of the laboratory analytical precision based on the sample homogeneity. Duplicate samples are collected by equally dividing the sample removed from a sample source, then placing the aliquots into separate, but identical, sample containers. This process will be repeated until each sample container is properly filled, thus resulting in two samples. The collected duplicate samples will be preserved in the same manner as the investigative samples and analyzed for the same parameters/analytes of investigation. These duplicate samples will be appropriately labeled and documented in field records. These samples will be stored, transported, and analyzed with the investigative samples.

Table 6.1
Types of Field Quality Control Samples
AK Steel SWMU 23 and AAs

Type of Control	Purpose of Sample	Collection Frequency
Duplicate Sample	To ensure precision in sample homogeneity during collection and analysis	10% of field samples per matrix
Rinse Blank	To ensure the decontamination of sampling equipment has been adequately performed; to assess cross contamination and/or incidental contamination to the sample container	One per 20 samples or one per day
Temperature Blank	To verify sample cooler temperature during transport	One temperature blank per cooler
Trip Blank	To evaluate if cross contamination occurs during shipment or storage with volatile organic analyses (VOA) samples	One trip blank per cooler of VOA samples
Source Water	To characterize decontaminated water	One per source

Table 6.2 Field QC Elements of a QA Program AK Steel SWMU 23 and AAs				
Item	**DQO**	**Parameter**	**Frequency of Association**	**Criteria Requirement**
Source Water	R	Target list of parameters	Per project	Less than laboratory reporting limit or if detected approved by AK Steel
Field Duplicates	P	Target list of parameters	One per ten samples	RPD ≤25% aqueous; ≤35% solid
Trip Blank	A,R	Volatiles in water	One per cooler with volatiles	No target analytes
Rinse Blank	A,R	Target list of parameters	One per 20 samples per matrix per equipment type	No target analytes
Field Logbook	A,C	Target list of parameters	Daily	Filled out and representative of investigation activities
Chain of Custody	R	Target list of parameters	Every sample	Filled out correctly to include signatures; no missing or incorrect info
Chemical Parameter Forms	R	Target list of parameters	Every sample	Filled out correctly to include analytical parameters; and applicable coding info
Field Instrument Calibration Logs	A	Target list of parameters	Every measurement	Measurements must have associated calibration reference

Legend:
A = Accuracy
C = Completeness
P = Precision
R = Representativeness

8.2 Laboratory Quality Control

Laboratories are required to demonstrate their ability to produce acceptable results using the methods selected by the generation of acceptable QC data. Analytical data is evaluated by the laboratory prior to submittal based on internal reviews of the QC data. Internal laboratory QC procedures used to assess laboratory performance are specified in the respective laboratory SOPs (Appendix A). Internal QC checks performed during this project to assess sampling procedures and laboratory performance on every QC batch of no more than 20 field samples (i.e., a sample delivery group [SDG]) include laboratory duplicate samples, MS/MSD samples, laboratory method blanks, and laboratory control samples.

Analytical QC checks are performed in each laboratory. These procedures are based upon USEPA analytical methods and generally accepted standards of good laboratory practice. Key components of the laboratories' QA/QC programs are discussed in the respective laboratory QAPPs.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

The following sections provide a brief summary of data reduction, validation, and reporting.

9.1 Data Reduction

Data reduction is the process of converting analytical data from electronic form or instrument form into digital form and correcting for all sample weight, dilution factors, and percent solids calculations that may be applicable. This process is performed in the field and in the laboratory.

9.1.1 Field Data Reduction Procedures

Raw data from field measurements and sample collection activities will be recorded in the field records and on applicable field log sheets. Instrument readings will be recorded directly into the field records. Field personnel will submit all field notes and field logs to the Project Manager at the end of each work week. These documents include daily logs, soil boring and sampling logs, and laboratory chain-of-custody documents. Errors will be corrected by drawing a single line through the error and initialing and dating the error. Recorded field data will be reviewed for accuracy and completeness by the KEMRON Project Manager or designee.

9.1.2 Laboratory Data Reduction Procedures

Raw data from analytical laboratory measurements may or may not be generated by digital readout instruments. In addition, due to dilution and concentration factors often developed during the analysis of the sample, careful data reduction is required to convert raw data to useable digital analytical data. All data reductions will be performed according to procedures presented in each laboratory's QAPP.

9.2 Data Validation

Data validation is a systematic process for reviewing a body of assembled analytical results against a set of criteria to determine if the data are adequate for the intended use. Data validation involves: (1) data editing; (2) data screening and checking; (3) data auditing and verification; (4) data certification; and (5) final data review and informed decision-making whereby data are accepted, qualified, or rejected.

All analytical data developed from this investigation will be validated according to criteria specified in the functional guidelines for data validation (USEPA, 2008 and 2010) and the guidance on data verification and data validation (USEPA, 2002b). The data validation procedures employed for this project will include an evaluation of the field data package, an evaluation of the laboratory's analytical data packages, and documentation of data that do not meet validation criteria.

An independent 3rd party will complete a Level II data validation checklist to verify that the laboratories have performed in accordance with requirements specified in this QAPP, Guidance for Data Quality Assessment (USEPA, 2000), Guidance of Environmental Data Verification and Data Validation (USEPA, 2002b), and the respective laboratories' SOPs (Appendix A). Therefore, the laboratories are required to submit data that are supported by sufficient QC backup information to allow the reviewers to conclusively evaluate the quality of the data. Level II validation will include a systematic review of the data for compliance with the established QC criteria for the methods used, which may be modified, as appropriate, by the requirements specified in the current USEPA Functional Guidelines for Organics Analyses (USEPA, 2008) and National Functional Guidelines for Inorganic Data Review (USEPA, 2010). The data will also be reviewed to determine compliance with the criteria set forth in USEPA Guidance for Quality Assurance Project Plans (USEPA, 2002a) and Final Guidance for Data Usability in Risk Assessment (USEPA, 1992).

Analytical data will be submitted to KEMRON in both hard copy and in a computerized format, organized to facilitate data management. The laboratory-provided data flags may include such items as estimated concentration; below required reporting limit, and concentration of chemicals also found in blanks. All data

will then be processed through a Data Validation Checklist for Level II reporting requirements (Appendix B). This checklist includes:

- Laboratory report information;
- Field data package documentation including: sampling notes, sample locations and depths, collection techniques, field preparation, container type and preservation method, chain-of-custody form completion, analyses requested, completed sampling logs, number and type of field QC samples collected, field equipment calibration and decontamination, sample shipping, and laboratory task order; and
- All QA reporting levels including: sample results, parameters analyzed, method of analysis, reporting limits of analysis, master tracking list, sample collection date, laboratory sample received date, sample preparation and extraction date, sample analysis date, chain-of-custody signed by lab custodian, and a narrative summary of QA or sample problems.

Level II requirements include: holding times, reporting limits, method blanks, equipment rinseate blanks, matrix spike, percent recovery, MS/MSD precision, laboratory control sample, surrogate spike recoveries, and duplicate comparison.

The data reviewer comments will indicate that the data are (1) usable as a quantitative concentration (no data qualifiers present); (2) usable, with caution, as an estimated concentration ("J" or "UJ" data qualifiers present); or (3) unusable due to unacceptable QC results ("R" data qualifiers present). The complete data set will be incorporated into the project file.

9.3 Laboratory Reporting Requirements

For analytical data quality to be adequately assessed, it is imperative that expected reporting levels are communicated to the laboratories. The laboratories must submit, in addition to the analytical results, all supporting QC data required for effective validation of the analytical data. If increased defensibility of the laboratories' data is required, additional documentation of analytical QC data should be available upon request to the laboratories to support validation conclusions and data usability determinations.

Laboratory data reports will consist of sample results plus the QA/QC data specified below. The following are general requirements for each sample analyzed by the laboratory:

- The results of each analysis;
- The list of the parameters of investigation;
- The method of analysis and the reporting limit for each analyte;
- Dates of sample collection, receipt, preparation/extraction, and analysis;
- Copy of the chain-of-custody forms signed by the laboratory's sample custodian;
- A narrative summarizing any QA/QC deviances and the corrective action taken; and
- A master list relating laboratories ID to sample ID.

The list below describes the information to be provided for all the organic analyses performed on a gas chromatograph (where applicable):

- Results of method blanks and trip blanks, water blanks and/or extraction blanks;

- Results of MS/MSD, control limits, expected value, RPD, and %R; and
- Results of surrogates spikes, the expected value, control limits, and %R.

Level II hard copy data deliverables will be generated at the time of analyses.

9.4 Data Acquisition Requirements

Data may be acquired from previously collected sources (e.g., literature, databases, and spreadsheets). Acceptance criteria for previously compiled data will be such that qualifying standards will allow for the combination of new and old data sets. Observations and transformation equations of the previous project(s) will be evaluated to assure that the assumptions of the previous source(s) are consistent with the current project.

9.5 Data Management

Data management will be conducted as described in the RFI Data Management Plan (KEMRON, 2012).

9.5.1 Data Analysis

Project data will be processed in order to create data products for use in decision-making and reporting. Analyses may include statistical interpretation of results, risk assessments, and other approaches. Analysts may compile, separate, or summarize data in their analysis, so as to best utilize field measurements, laboratory measurements, and geospatial data. This, in turn, will be used to produce text, tables, graphs, maps, isopleth plots, models, and other products used to describe the data. Results of such analyses may lead to other analyses.

9.5.2 Processed Data Tracking

Processed data will be tracked from receipt of the QA field and analytical data to the creation of the final summary report. All data processing tracking records will be stored in the project file.

9.5.3 Data Security/Storage

Data security measures help ensure that critical data are not lost, destroyed, or altered without supporting documentation. Measures include preserving both electronic and hard copy forms of all original data and chain-of-custody forms, maintaining the project database with routine backups, and limiting access to the project file to select personnel.

Within KEMRON, the project database will be maintained on a company server. Interconnected personal workstations and laptops allow access to the database through a wide area network (WAN). Use of a server allows for central data storage and management of data set access. Access is limited to select KEMRON project personnel. All hard copy data generated through the current project will be stored in a project file maintained at KEMRON. Decision making data and documents will be retained by AK Steel as well. (see Section 5.3).

10.0 PERFORMANCE AND SYSTEM AUDITS

Performance and systems audits for sampling and analysis operations consist of on-site review of field and laboratory QA systems and on-site review of equipment for sampling, calibration, and measurement techniques. Performance and systems audits evaluate and verify that organization, operations, and procedures are in compliance with protocols specified in this QAPP and work plans. Performance and systems field audits will be conducted at the beginning of the project and additional audits will be conducted at the discretion of the KEMRON and/or AK Steel Project Managers. Field oversight and laboratory audits may also be performed at the discretion of the USEPA and Ohio EPA. Recent laboratory audits and/or certifications for the analyses performed under this QAPP will be provided to USEPA for review upon request.

11.0 PREVENTIVE MAINTENANCE

11.1 Field Instrument Routine Maintenance Activities

The field instrument routine preventive maintenance procedures for each instrument are specified in the appropriate operation manual. All equipment preventive maintenance performed during the project will be noted in field records or on applicable field forms.

11.2 Laboratory Instrument Routine Maintenance Activities

The contracted laboratories maintain an equipment maintenance program designed to track routine maintenance, calibration, and repair of laboratory equipment. Maintenance of equipment is in accordance with manufacturer's recommendations and with normal laboratory practices. Maintenance records will be maintained in permanent records or equipment logs.

11.3 Contingency Plans

The performance of all field instruments is pre-tested prior to beginning any fieldwork to determine if instruments are in good working order. To allow for contingencies if an instrument is found to be in need of repair, equipment is also available through other KEMRON offices or rental companies that may be picked up locally or delivered on short notice by commercial courier service (e.g., Federal Express).

11.4 Inspection/Acceptance Requirements for Supplies and Consumables

All non-disposable field sampling equipment will undergo routine decontamination procedures as described in KEMRON SOP 07-DCN-00 – Sampling Equipment and Container Decontamination. These methods will be validated through the use of rinseate blanks to assure acceptance requirements. All disposable sampling equipment will be considered contaminant-free prior to use. Laboratory-grade, analyte-free water will be certified by the laboratory. Sample jars will arrive from the laboratories certified as pre-cleaned.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO EVALUATE DATA PRECISION, ACCURACY, AND COMPLETENESS

12.1 Precision

Precision of laboratory analysis is addressed by calculating the RPDs for each pair of duplicate analyses using the following equation:

$$RPD = \frac{[S - D]}{(S + D)/2} \times 100\%$$

where, RPD = relative percent difference
S = sample value (original)
D = duplicate sample value

12.2 Accuracy

Accuracy of laboratory analysis is assessed by using the following equations:

$$\%R = \frac{[A - B]}{C} \times 100\%$$

where, A = spiked sample result
B = sample result
C = spike added

And

$$\%D = \frac{[M - T]}{T} \times 100\%$$

where, %D = percent difference
M = measured value
T = true value (sample value for serial dilution)

12.3 Completeness

Data completeness is assessed using the following equation:

$$C = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100\%$$

where, C = Percent complete
Valid Data Obtained = The number of environmental samples obtained or to be obtained for a specific purpose, or in order to satisfy a particular project objective, that are not qualified to use or invalid.
Total Data Planned = The number of environmental samples obtained or to be obtained.

12.4 Assessment of Data

Data will be assessed in order to determine if the data collected and produced is of the desired type, quality, and quantity to satisfy its intended use. All project data will be assessed in accordance with the Region 5 Data Quality Assessment (DQA) Policy, under direction from the *Guidance for Data Quality Assessment - Practical Methods for Data Analysis* (USEPA, 2000).

13.0 CORRECTIVE ACTION

Corrective actions may be required for two classes of problems: (1) analytical and equipment problems and (2) noncompliance problems. Analytical and equipment problems may occur during sampling, sample handling, sample preparation, laboratory instrumental analysis, and data review. Noncompliance problems occur when activities are conducted outside the requirements of the QAPP. For both types of problems, a formal corrective action program will be implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the KEMRON Project Manager who will then contact the AK Steel Project Manager, if appropriate. The KEMRON Project Manager is responsible for implementing corrective action at the field, laboratory, and data assessment stages of the project. If the problem is analytical in nature, information on these problems will be promptly communicated to the appropriate laboratory Project Manager. Corrective actions will be documented in writing and the USEPA Project Manager will be notified via telephone, letter correspondence, or later in data reports depending on the significance of the corrective action.

Responsibilities for corrective actions are provided in Table 7.

Table 7
Assessment Type and Corrective Action Responses
AK Steel SWMU 23 and AAs

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation[1]	Individual(s) Receiving Corrective Action Response (Name, Title, Organization)	Timeframe for Response
Readiness Review	Written Summary of Findings	FS, QA Manager, PM, KEMRON	Within 24 hours after review	Memorandum	FS, QA Manager, PM, KEMRON	Within 24 hours after notification of deficiency requiring CA
Field Sampling Technical Systems Audit	Written Audit Report	FS, QA Manager, PM, KEMRON	5 business days after audit	Memorandum	FS, QA Manager, PM, KEMRON	Within 24 hours after notification of significant deficiencies
On-site Analytical TSA	Written Audit Report	FS, laboratory	72 hours after audit report receipt by laboratory	Letter or Memorandum	FS, QA Manager, PM, KEMRON	24 hours after notification of deficiency(ies) requiring CA
Data Review and Validation	Data Validation Report and Usability Assessment	KEMRON Data Validator, or subcontractor	Within 30 business days from receipt of analytical report	CA Report from Analytical Laboratory	Data Validator; FS, QA Manager, KEMRON	3 business days from identification of deficiency affecting data

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The laboratory and/or field project records will contain a description of the following QA items (if applicable):

- Changes in this QAPP;
- Results of performance evaluation audits;
- Significant QA/QC problems, recommended solutions, and results of corrective actions;
- Data quality assessment in terms of precision, accuracy, representativeness, completeness, comparability, and conformance with reporting limits and/or estimated quantitation limits;
- Indication of whether the QA objectives were met; and
- Limitations on use of the measurement data.

The KEMRON Project Manager will review all aspects of the implementation of this QAPP on a regular basis. Reviews will be performed at the completion of each field activity, and a summary report will be completed at that time. The above items, as applicable, will be discussed in the RFI report.

15.0 REFERENCES

ENVIRON. 2007. Interim Measures Quality Assurance Project Plan, Dicks Creek, Middletown, Ohio, Revision 1. June 2007.

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USEPA. 2002a. Guidance for Quality Assurance Project Plans. EPA/240/R-02/009. U.S. Environmental Protection Agency. December.

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APPENDIX A

STANDARD OPERATING PROCEDURES

Field SOPs – KEMRON:

SOP 07-DCN-00	Standard Operating Procedure for Sampling Equipment and Container Decontamination
SOP 07-DOC-00	Standard Operating Procedure for Field Documentation
SOP 07-FQC-00	Standard Operating Procedure for Field Quality Control
SOP 07-PAS-00	Standard Operating Procedure for Sample Packaging and Shipping
SOP 12-GWS-01	Standard Operating Procedure for Groundwater Sampling
SOP 12-SSP-01	Standard Operating Procedure for Soil Sampling in the Field
SOP 12-MWI-AKMW	Standard Operating Procedure for Groundwater Monitoring Well Installation and Development, Site Specific to AK Steel Middletown Works
SOP 12-PID-AKMW	Standard Operating Procedure for Field Screen of Samples Using Photo-Ionization Detector, Site Specific to AK Steel Middletown Works
SOP 12-SBC-AKMW	Standard Operating Procedure for Soil Boring Collection, Site Specific to AK Steel Middletown Works

Laboratory SOPs -TestAmerica North Canton and Nashville:

SOP NC-GC-038	Gas Chromatographic Analysis
SOP NC-IP-010	Acid Digestion for Solid Samples
SOP NC-IP-011	Acid Digestion for Aqueous Samples
SOP NC-MS-019	Determination of Volatile Organics by GC/MS
SOP NC-MT-012	Inductively Couples Plasma – Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses
SOP NC-MT-014	Preparation an Analysis of Mercury in Aqueous and Solid Samples by Cold Vapor Atomic
SOP NC-OP-037	Continuous Liquid/ Liquid Extraction of Organic Compounds from Waters
SOP NV-03-231	Preparation of Soil/Sediment by Automated Soxhlet for Semivolatile Organic Compounds SW-846 Method 3541

SOP NC-OP-40	Soxhlet Extraction of Organic Compounds

Laboratory SOPs-Terracon Consultants, Inc.:

SOP GT-002:	Processing Bulk Samples
SOP GT-005:	ASTM D4318 – Liquid Limit, plastic limit, and Plasticity Index of Soils
SOP GT-007:	ASTM D422 – Soil Classification - Grain Size Analysis of Soil
SOP GT-008:	ASTM D2216 – Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass
SOP GT-012:	Preparing Test Specimens from Undisturbed Samples for Permeability Testing
SOP GT-013:	ASTM D5084 - Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter

FIELD SOPs

Document Control #: 07-DCN-00

STANDARD OPERATING PROCEDURE FOR SAMPLING EQUIPMENT AND CONTAINER DECONTAMINATION

Issue Date: November 8, 2004
Revision 00

Last Review/Implementation Date: November 2004

KEMRON Environmental Services

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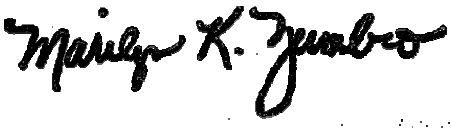

11/8/04

Marilyn Zumbro, Regional Manager — Date

11/8/04

John Dwyer, Vice President — Date

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Kelly Clemons, Corporate Quality Assurance Manager — Date

The following persons have read and understand this SOP:

Signature	Date

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1. PURPOSE

By complying with procedures specified in this SOP, KEMRON will ensure the removal of contaminants of concern from sampling, drilling and other field equipment.

2. SCOPE

This procedure provides technical guidance on decontamination of sampling equipment and sample containers. The following two categories of cleaning are covered in this procedure:

- Equipment cleaning procedures
- Emergency field sample container cleaning

3. RESPONSIBILITIES

3.1 Project Manager Responsibilities

The KEMRON project manager ensures that any project specific deviations from this procedure will be provided to the Field Staff.

3.2 Field Staff Responsibilities

The KEMRON field staff follow procedures specified in this SOP unless written site-specific protocol is provided by the Project Manager.

4. DEFINITIONS / SPECIFICATIONS

4.1 Emergency Field Sample Container Cleaning Procedures

Emergency field sample container cleaning procedures are restricted procedures to be used when events occur outside the scope of the sampling plan.

4.2 Soap

Soap is a standard brand of phosphate-free laboratory detergent such as Liquinox®. Use of other detergent is justified and documented in the field logbooks and inspection or investigative reports.

4.3 Solvent

Solvent is pesticide-grade isopropanol. Other solvents may be substituted for a particular purpose if required. Pesticide-grade acetone or methanol are acceptable.

4.4 Tap water

Tap water may be used from any municipal water treatment system (spigot at the office). Use of an untreated potable water supply is not an acceptable substitute for tap water.

4.5 Deionized water

Deionized water should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits).

4.6 Disinfectant soap

Disinfectant soap is a standard brand of disinfectant cleaner.

4.7 Nitric acid solution (10%)

Nitric acid solution (10%) is made from reagent-grade nitric acid and deionized water.

4.8 Containment

Containment includes securing all waste materials generated during site operations. Routine practice should include all efforts to keep used decontamination solutions at the site. This is especially the case for equipment used to collect samples of hazardous materials or toxic wastes or materials from hazardous waste sites or RCRA facilities.

5. HANDLING OF AND CONTAINERS FOR CLEANING SOLUTIONS

Improperly handled cleaning solutions may easily become contaminated. Storage and application containers must be constructed of the proper materials to ensure their integrity. The following are acceptable materials used for containing the specified cleaning solutions:

5.1 Standard Use Solutions

5.1.1 Soap is kept in clean plastic, metal, or glass containers until used. It should be poured directly from the container during use.

5.1.2 Tap water may be kept in clean tanks, hand pressure sprayers, squeeze bottles, or applied directly from a hose. All secondary containers are labeled as to the contents.

5.1.3 Deionized water is stored in clean containers that can be closed prior to use. It can be applied from plastic squeeze bottles. All secondary containers are labeled as to the contents.

5.2 Non-standard Use Solutions

5.2.1 Disinfectant soap should be kept in clean containers until use. It should be poured directly from the container.

5.2.2 Solvent is stored in the unopened original containers until used. They may be applied using the low pressure nitrogen system fitted with a Teflon® nozzle, or using Teflon® squeeze bottles. All secondary containers are labeled as to the contents.

5.2.3 Nitric acid should be kept in the glass container it is received in, and placed in squeeze bottles prior to application. All secondary containers are labeled as to the contents.

Note: Hand pump sprayers generally are not acceptable storage or application containers for the above materials (with the exception of tap water). This also applies to stainless steel sprayers. All hand sprayers have internal oil coated gaskets and black rubber seals that may contaminate the solutions.

6. EQUIPMENT CLEANING PROCEDURES

All equipment taken to the field is assumed to be clean. The following procedures are completed during a sampling event for multiple uses of the equipment, at the conclusion of equipment use at the site and if visual evidence indicates a need at the onset of equipment use.

6.1 Specifications for Decontamination Pads

The size of the project will dictate the method of containing decontamination fluids. Examples of containment include the use of drums, buckets, decon pads, etc. For additional information see Appendix C.

6.2 Sampling Equipment Cleaning Procedures

The following procedures are used for all sampling equipment (well casings, well screens, or split spoon samplers, etc.), unless otherwise specified in the project documents or unless it is observed that additional cleaning is necessary. For additional guidance on decontamination and cleaning of equipment, see Appendix A.

1) Clean with tap water and soap using a brush if necessary to remove particulate matter and surface films. Equipment may be steam cleaned (soap and high pressure hot water) as an alternative to brushing; however, PVC or plastic items should not be steam cleaned.
2) Rinse thoroughly with tap water.
3) Rinse thoroughly with deionized water.
4) If equipment is to be used during the next field sampling operations, remove from the decontamination area and cover with plastic. If equipment cleaning is a final project cleaning or will be used during a later sampling phase, wrap equipment in one layer of aluminum foil. Seal the foil wrapped equipment in plastic and label.
5) For information related to contaminant of decontamination solutions, see Appendix F.

After collecting samples that contain oil, grease, or other hard to remove materials, it may be necessary to rinse the equipment several times with pesticide-grade acetone, hexane or petroleum either to remove the materials before proceeding with the first step of cleaning; or in extreme cases, it may be necessary to steam clean the field equipment before the first step of cleaning; or discarding the equipment may be necessary. Special considerations need to be addressed with the Project Manager before deciding to use solvent rinses since waste handling and disposal are regulated activities.

6.3 Cleaning Procedures for Specialized Equipment

For specialized field equipment the manufacturer's manual should state the cleaning procedures KEMRON should use. For additional information see Appendix A.

6.4 Handling of Cleaned Equipment

After field cleaning, equipment should be handled only by personnel wearing clean gloves to prevent re-contamination. In addition, the equipment should be moved away (preferably upwind) from the cleaning area to prevent recontamination. If the equipment is not to be immediately re-used it should be covered with plastic sheeting or wrapped in

aluminum foil to prevent re-contamination. The area where the equipment is kept prior to re-use must be free of contaminants.

7. SUBCONTRACTOR EQUIPMENT CLEANING PROCEDURES

Heavy equipment rental and operation is usually subcontracted and the subcontractor is responsible for equipment decontamination. KEMRON personnel are responsible to direct any additional cleaning should there be a need. For additional information see Appendix D.

8. EQUIPMENT CLEANING PROCEDURES AT THE OFFICE

Additional cleaning and decontamination procedures maybe warranted when equipment or parts of equipment arrival back at the office (field or home). See Appendix B for examples of cleaning procedures and Appendix G for disposal of office cleaning solutions.

9. PROCEDURES FOR DISPOSABLE SAMPLE CONTAINER CLEANING AND USE: IN EMERGENCY FIELD SITUATIONS

The following container use and cleaning procedures are restricted for use in emergency situations only which maybe necessary if sampling occurs outside planned events; or in the case of an ER mobilization where additional containers may be needed in a pinch. In such emergencies, new one-pint or one-quart mason jars may be used to collect samples for analyses of organic compounds and metals in waste and soil samples. These containers would also be acceptable on an emergency basis for the collection of water samples for extractable organic compounds, pesticides, and metals analyses. These jars cannot be used for the collection of water samples for volatile organic compound analyses. The rubber sealing ring should not be in contact with the jar and aluminum foil should be used, if possible, between the jar and the sealing ring. If possible, the jar and aluminum foil should be rinsed with pesticide-grade isopropanol and allowed to air dry before use. Several empty bottles and lids maybe submitted to the laboratory as blanks for quality control purposes.

10. PREPARATION OF DISPOSABLE SAMPLE CONTAINERS

Routine sampling procedures call for precleaned containers supplied by the laboratory. However, if disruption of the supply of pre-cleaned, certified sample bottles occurs, sampling may have to continue regardless. In such situations, the laboratory is contacted immediately for the shipping of additional containers and approval of any field cleaning procedures of sample containers in the interim. For examples of cleaning disposable sample containers under such conditions, see Appendix E.

11. REFERENCE

1) Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, US EPA Region 4, November 2001

APPENDIX A: ADDITIONAL FIELD CLEANING PROCEDURES

Well Sounders, Tapes, or Interface Probes

The following cleaning procedures are used for well sounders or tapes:

1) Wash with soap and tap water.
2) Rinse with tap water.
3) Rinse with deionized water.

Pumps

Cleaning of pumps not specified in this section will be cleaned in a similar fashion.

1) Goulds® Pump Cleaning Procedure
   CAUTION - During cleaning always disconnect the pump from the generator. The Goulds© pump should be cleaned prior to use and between each monitoring well. The following cleaning procedure is used:
   a) Using a brush, scrub the exterior of the contaminated hose and pump with soap and tap water.
   b) Rinse the soap from the outside of the pump and hose with tap water.
   c) Rinse the tap water residue from the outside of pump and hose with deionized water.
   d) Place the pump and hose in a clean plastic bag.

2) Redi-Flo2® Pump
   CAUTION - Make sure the pump is not plugged in. The Redi-Flo2® pump should be cleaned prior to use and between each monitoring well. The following cleaning procedure is used:
   a) Using a brush, scrub the exterior of the pump, electrical cord and garden hose with soap and tap water. Do not wet the electrical plug.
   b) Rinse with tap water.
   c) Rinse with deionized water.
   d) Place the equipment in a clean plastic bag.

3) Redi-Flo2® Ball Check Valve
   The following procedure is required to clean the Redi-Flo2® ball check valve:
   a) Completely dismantle ball check valve. Check for wear and/or corrosion, and replace as needed.
   b) Using a brush, scrub all components with soap and tap water.
   c) Rinse with deionized water.
   d) Reassemble and re-attach the ball check valve to the Redi-Flo2® pump head.

Automatic Sampler Tubing

Whenever possible use new or dedicated tubing. In cases where new or dedicated tubing can not be used, Silastic® and Tygon® tubing previously used in the automatic samplers may be field cleaned as follows:

a) Flush tubing with tap water and soap.
b) Rinse tubing thoroughly with tap water.
c) Rinse tubing with deionized water.

Full-face, ½-face, and SCBA Face-masks

When there is a potential for exposure, face masks are used. This being the case, cleaning of face masks always follows use.

a) Wash face-mask thoroughly inside and out with hot tap water and disinfectant soap. Use only soft brushes. Do not use scouring pads of any type.
b) Rinse face-mask thoroughly inside and out with tap water.
c) Hang face-mask up until completely dry.
d) Place face-mask in plastic bag and return to SCBA case.

APRs are completely dismantled prior to cleaning. Then steps as outlined above for SCBA face masks are used. When completely dry, the APR is reassembled and placed in a plastic bag.

APPENDIX B: PROCEDURES FOR OFFICE CLEANING

See the web at the following web address for additional cleaning procedures (see reference section): http://www.epa.gov/region4/sesd/eisopqam/eisopqam.pdf

APPENDIX C: EXAMPLE OF CONSTRUCTION & USE OF A DECONTAMINATION PAD

1) The pad should be constructed in an area known or believed to be free of surface contamination.
2) The pad should not leak excessively.
3) If possible, the pad should be constructed on a level, paved surface and should facilitate the removal of wastewater. This may be accomplished by either constructing the pad with one corner lower than the rest, or by creating a sump or pit in one corner or along one side. Any sump or pit should also be lined.
4) Sawhorses or racks constructed to hold equipment while being cleaned should be high enough above ground to prevent equipment from being splashed.
5) Water should be removed from the decontamination pad frequently.
6) A temporary pad should be lined with a water impermeable material with no seams within the pad. This material should be either easily replaced (disposable) or repairable.
7) At the completion of site activities, the decontamination pad should be deactivated. The pit or sump should be backfilled with the appropriate material designated by the site project leader, but only after all waste/rinse water has been pumped into containers for disposal. No solvent rinsates will be placed in the pit.
8) Solvent rinsates should be collected in separate containers for proper disposal. If the decontamination pad has leaked excessively, soil sampling may be required.

APPENDIX D: SUBCONTRACTOR EQUIPMENT CLEANING PROCEDURES

This section is provided as a guide to KEMRON employees to ensure that the subcontractor equipment brought on-site and cleaned in the field has been cleaned to a level equivalent to that stated here. In addition, the procedures stated provide an example of text that should be included in the SOW the subcontractor is to follow where it is specified that such steps are to be completed either prior to arrival at the site or between each drilling event, as applicable.

These procedures are used for drilling activities involving the collection of soil samples for trace organic and inorganic constituent analyses, and for the construction of monitoring wells to be used for the collection of groundwater samples for trace organic and inorganic constituent analyses.

Preliminary Equipment Cleaning (prior to site arrival)

The subcontractor should bring drilling and related equipment pre-cleaned and ready for use. The following outlines the equivalent procedures that should be used by the subcontractor:

1) All downhole augering, drilling, and sampling equipment should be sandblasted before use if painted, and/or there is a buildup of rust, hard or caked matter, etc., that cannot be removed by steam cleaning (soap and high pressure hot water), or wire brushing. Sandblasting should be performed prior to arrival on site, or well away from the decontamination pad and areas to be sampled.
2) Any portion of the drill rig, backhoe, etc., that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (soap and high pressure hot water) and wire brushed (as needed) to remove all rust, soil, and other material which may have come from other hazardous waste sites before being brought on site.
3) Printing and/or writing on well casing, tremie tubing, etc., should be removed before use. Emery cloth or sand paper can be used to remove the printing and/or writing. Most well material suppliers can supply materials without the printing and/or writing if specified when ordered.

Drill Rig Field Cleaning Procedure for Multiple Boreholes

Any portion of the drill rig, backhoe, etc., that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (soap and high pressure hot water) between boreholes.

Field Cleaning Procedure for Drilling Equipment

The following is a standard procedure for subcontractor field cleaning of augers, drill stems, rods, tools, and associated equipment.

1) Clean with tap water and soap, using a brush if necessary, to remove particulate matter and surface films. Steam cleaning (high pressure hot water with soap) may be necessary to remove matter that is difficult to remove with the brush. Drilling equipment that is steam cleaned should be placed on racks or saw horses at least two feet above the floor of the decontamination pad. Hollow-stem augers, drill rods, etc., that are hollow or have holes that transmit water or drilling fluids, should be cleaned on the inside with vigorous brushing.
2) Rinse thoroughly with tap water.
3) Remove from the decontamination pad and cover with clean, unused plastic. If stored overnight, the plastic should be secured to ensure that it stays in place.

APPENDIX E: PREPARATION OF DISPOSABLE SAMPLE CONTAINERS

1. Introduction

   No disposable sample container (with the exception of the glass and plastic compositing containers) may be reused. All disposable sample containers will be stored in their original packing containers. When packages of uncapped sample containers are opened, they will be placed in new plastic garbage bags and sealed to prevent contamination during storage. Specific pre-cleaning instructions for disposable sample containers are given in the following sections.

2. Plastic Containers used for "Classical" Parameters

   Plastic containers used for oxygen demand, nutrients, classical inorganics, and sulfides have no precleaning requirement. However, only new containers may be used.

3. Glass Bottles for Semi-Volatile GC/MS Analytes

   These procedures are to be used only if the supply of pre-cleaned, certified sample bottles is disrupted. Laboratory approval is necessary prior to implementation of these procedures. If desired, pesticide-grade methylene chloride may be substituted for pesticide-grade isopropanol. In addition, 1:1 nitric acid may be substituted for the 10% nitric acid solution.

   a) Wash bottles and jars, Teflon® liners, and caps in hot tap water and soap.
   b) Rinse three times with tap water.
   c) Rinse with 10% nitric acid solution.
   d) Rinse three times with deionized water.
   e) Rinse bottles, jars, and liners (not caps) with solvent.
   f) Oven dry bottles, jars, and liners at 125EC. Allow to cool.
   g) Place liners in caps and close containers.
   h) Store in contaminant-free area.

4. Glass Bottles for Volatile GC/MS and TOX Analyses

   These procedures are to be used only if the supply of pre-cleaned, certified sample bottles is disrupted. Prior QA approval is necessary prior to implementation of these procedures.
   a) Wash vials, bottles and jars, Teflon liners and septa, and caps in hot tap water and laboratory detergent.
   b) Rinse all items with deionized water.
   c) Oven dry at 125oC and allow to cool.
   d) Seal vials, bottles, and jars with liners or septa as appropriate and cap.
   e) Store in a contaminant free area.

5. Plastic Bottles for ICP Analytes

   These procedures are to be used only if the supply of pre-cleaned, certified sample bottles is disrupted. Prior QA approval is necessary prior to implementation of these procedures.
   a) Wash bottles and caps in hot tap water with soap.
   b) Rinse both with 10% nitric acid solution.
   c) Rinse three times with deionized water.
   d) Invert bottles and dry in contaminant free environment.
   e) Cap bottles.
   f) Store in contaminant free area.

APPENDIX F: DISPOSAL OF FIELD CLEANING SOLUTIONS

Disposal of Field Cleaning Solutions

Procedures for the safe handling and disposition of investigation derived waste (IDW), including used wash water, rinse water, and spent solvents are specified in the project plan. The following are provided as general guidelines:

1) Minimum Requirements for Non-hazardous Disposal of IDW
   a) Liquid and soil/sediment IDW is containerized and analyzed before disposal.
   b) The collection, handling, and proposed disposal method is specified in the approved project plan.

2) Minimum Requirements for Management of Hazardous IDW
   a) Spent solvents should remain in the field for proper disposal or recycling.
   b) All hazardous IDW is containerized. Proper handling and disposal should be arranged prior to commencement of field activities.

DISPOSAL of IDW

Type	Hazardous	Non-Hazardous
Spent Solvents	Containerize in original containers. Clearly identify contents. Leave on-site with permission of site operator, otherwise return to field office for proper disposal.	N/A
Decontamination Water	Containerize in 55-gallon drum with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with site manager for testing and disposal.	Containerize in 55-gallon drum with tight fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with site manager for testing and disposal.

APPENDIX G: DISPOSAL OF OFFICE (FIELD OR HOME) CLEANING SOLUTIONS

Since equipment is cleaned in the field at the conclusion of field activities, any additional cleaning completed at the office will have differing waste disposal procedures for cleaning solutions than those in the field. Procedures for safe handling and disposition of such spent cleaning solutions, including washwater, rinse water, spent acid solutions, and spent solvents are as follows:

1) Washwater
   Since equipment is decontaminated before its return to the office, the washwater may be disposed in the sanitary drain in the washroom. When large equipment (vehicles, augers, etc.) is washed outside, it may wash onto the ground without recovery of the washwater.

2) Rinsewater
   Since equipment is decontaminated before its return to the office, the rinsewater may be disposed in the sanitary drain in the washroom. When large equipment (vehicles, augers, etc.) is rinsed outside, it may go onto the ground without recovery.

3) Nitric Acid
   Nitric acid cleaning solutions are to be diluted to a pH greater than 2.0, and flushed down the sanitary drain in the washroom. If used outdoors, this material should be captured and diluted to a pH greater than 2.0, and flushed down the sanitary drain in the washroom.

4) Solvent
   All solvents used should be captured, properly labeled, and stored on the premises of the office until arrangements for proper disposal are made. Used solvents can be classified as either "solvent for recovery" or "solvent for disposal". Solvent for recovery is that which was used in the standard field cleaning or office cleaning of equipment. Solvent used for cleaning badly contaminated equipment (e.g., tar removal, etc.) should be designated for disposal. The two groups should be labeled "For Recovery" or "For Disposal" and stored separately at the office.

Document Control #: 07-DOC-00

STANDARD WORK INSTRUCTION FOR FIELD DOCUMENTATION

Issue Date: November 8, 2004
Revision 00

Last Review/Implementation Date: November 2004

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John Dwyer, Vice President — Date

11/8/04

Kelly Clemons, Corporate Quality Assurance Manager — Date

The following persons have read and understand this SOP:

Signature	Date

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1. POLICY

All field activities are documented. The extent of the documentation is such that all activities can be recreated either through real time note taking or through reference to other documents and/or logbooks. All logbooks have the KEMRON home office address and phone number, toll free if available written on the inside of the front cover.

2. PURPOSE

This procedure establishes the field documentation at KEMRON.

3. SCOPE

The technical documentation addressed in this procedure applies to field activities that KEMRON directly participates in or where project over-ste is provided by KEMRON.

4. DEFINITIONS AND ACRONYMS

Not Applicable

5. RESPONSIBILITIES

5.1 Project Manager

To ensure that the Field documentation procedures are followed.

5.2 Field Staff

At a minimum, to complete field documentation as stated in this SOP.

6. Field Documentation

The two types of field documentation routinely completed at KEMRON include bound notebooks and field data sheets. Specific types of field data sheets may be required by the client which are used in place of any generic field sheets used by KEMRON.

6.1 Bound Field Logbooks

Bound field logbooks are assigned to the job or job tasks for larger/multiphase work. The front of each logbook will state “KEMRON”, book number, project name, project number, and start/end dates for the logbook. The spine of the logbook will contain the following minimally: project number and start and end dates. Field notes recorded in bound notebooks will be recorded with a ball point pen, on all- weather bound environmental field notebooks with consecutively numbered pages. See section 7 for information to be recorded. Upon completion of the data entry and entry review (see section 6.3) the logbooks are immediately placed in a central project location for retrival by other KEMRON personnel.

6.2 Field Data Sheets

When field data sheets are used a reference to the frequency of site visits is established by either of the following: (1) client/work agreement (i.e., take data every month); or (2) the data sheets are referenced in a bound logbook. When referenced in a bound notebook, the date, time, number of pages and description of the content of the field data sheets is included. All recorded notes are made with a ball point pen. If multiple field data sheets are used for any given site visit, the pages are numbered using the following format: “page 1 of 3, page 2 of 3, page 3 of 3” in a conspicuous location on each data sheet.

6.3 QA/QC of Field Documentation

6.3.1 Making Corrections

If an entry is in need of a correction, the erroneous information is crossed out with a single strike mark and initialed and dated, where the correction is recorded as near to the error as possible.

6.3.2 Field Staff

The field staff who collected the data signs and dates the daily entry in the bound notebook or field data sheet(s). This is completed the day the data is taken.

6.3.3 Independent Reviewer

Each set of collected data is reviewed and signed by an independent reviewer. The review is completed as soon as possible following data collection. At a minimum, documentation and validity of the following items should be verified (will be dependent on type of field activity): Correct study area designation and sample numbers; date and time (24-hour system recordings); and ensure that any unused portion of the data sheet and/or bound logbook is lined out, initialed and dated.

7. Types of Field Documentation and Procedures

7.1 Documentation for Sampling Events

7.1.1 **General Entries: Logbooks and Field Data Sheets**

a. Page heading to include location, date, project/client (for easy reference when copying);
b. Time onsite and offsite;
c. Parties involved (list all individuals and companies);
d. Vehicle (type, function, etc.);
e. Weather and site conditions (to include temperature, humidity, barameter,wind dir/speed, rainfall);
f. Health and Safety topics discussed;
g. Detailed descriptions of sample locations that identify the area of concern (make references to maps, GPS location, redlines, manmade objects, terrain, site history, etc.);
h. Sampling equipment used, including reference to the calibration/maintenance logbook;
i. Any equipment decontamination and maintenance completed in the field and reference to who completed the decontamination and field maintenance if other than the field staff and when it was completed;
j. All collected field measurements (PID /OVA, pH, temperature, etc.);
k. Sample number, samplers signature, fractions sampled, date sampled, time (military format) sampled and preservation method for each sample;
l. Identification and types of QC samples collected;
m. Photographic information as specified in section 7.3;
n. Reference to and summary of information on any field data sheets;

o. Fed-ex number, if applicable;
p. Cross through any remaining empty portions of page(s) at conclusion of entries with initials in crossed area;
q. Number pages and sign;

7.1.2 Specific Entries

See the specific technical SOPs for specific items to be recorded for the specific application. Project specific plans may include documentation specifications as well.

7.1.3 Survey Documentation

For surveying well heads, the following are examples of what can be recorded:

a. The benchmark can be a true known elevation or an assumed elevation of 100.0 labeled as TBM (Temporary Benchmark).
b. The Back Sight (BS) is the rod reading taken from the known benchmark or the arbitrary benchmark established as 100.0'.
c. The Back Sight rod reading is simply added to the benchmark elevation to establish the Height of Instrument.
d. The Fore Sight (FS) rod readings are those rod readings taken from unknown elevation points.
e. Establish the elevations of previously unknown points by subtracting the FS rod readings from the HI.
f. A Turning Point (TP) is a newly surveyed point that becomes a new benchmark once the instrument is moved.
g. The above example shows MW-3 becoming a Turning Point (TP 1) as the instrument needed moved to get a line of site on the final two points.
h. Once the instrument is moved a new HI is established by simply taking a rod reading (BS) on your TP.

The above information can be recorded in columns in the logbook with the following column headers: Station ID, BS (+) Rod Reading, HI (height of Instrument), FS (-) Rod Reading, and Elevation.

STATION ID	BS (+) Rod Reading	HI (Height of Instrument)	FS (-) Rod Reading	ELEVATION
Benchmark (BM)	4.5	673.15		668.65
MW-1			4.70	668.45
MW-2			5.25	667.90
MW-3			3.50	669.65
Turning Point (TP 1)	6.5	676.15		669.65
MW-4			5.65	670.50
MW-5			5.50	670.65

7.1.4 Chain of Custody

Cross-reference the Chain of Custody information in the field logbook. The Chain of Custody is completed when samples are taken. Whenever possible, a secondary review is completed. Additional details are recorded in the applicable SOPs or project documents.

7.1.5 Sample Labels

Cross-reference the sample labels information in the field logbook. The sample labels are completed when samples are taken. Sample labels contain information such as sample ID, sampler's signature or first initial and last name, date and time (military format) of sampling, preservative, fraction, etc. Whenever possible, a secondary review is completed.

7.2 Equipment Maintenance & Calibration Logbooks

Each piece of equipment has an assigned logbook which remains at the central facility (regional office or site office). The equipment serial number cross-references the equipment with the logbook. All maintenance and calibration information is included in the logbook(s). Any maintenance and/or calibration that is completed in the field is recorded first in the field log and then transcribed to the equipment maintenance & calibration logbook upon returning to the office / site office.

7.3 Photographic Records

Photographs may be digital or film photographs. Digital photographs will be maintained in digital files and/or a photo record book in the project file. Film photographs will be maintained in the project file. The number and subject of all photographs (digital and film) will be recorded in the field logbook(s) and, if necessary, cross-referenced to the photographic logbook, which maybe used for larger projects. The entries identify the photographs with the following information:

1. The project#, date, time, and location of the photograph, including direction facing;
2. The name of the photographer, and;
3. The signature of the photographer.

Document Control #: 07-FQC-00

STANDARD OPERATING PROCEDURE FOR FIELD QUALITY CONTROL

Issue Date: November 8, 2004
Revision 00

Last Review/Implementation Date: November 2004

KEMRON Environmental Services

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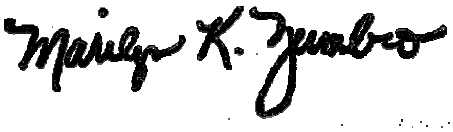

11/8/04

Marilyn Zumbro, Regional Manager — Date

11/8/04

John Dwyer, Vice President — Date

11/8/04

Kelly Clemons, Corporate Quality Assurance Manager — Date

The following persons have read and understand this SOP:

Signature	Date

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1. PURPOSE

This procedure is followed in conjunction with both project specific guidance to determine which quality control samples are necessary and field procedures for technical collection procedures. Following this procedure will ensure that the quality control samples collected will be collected in a consistent manner.

2. SCOPE

This SOP provides basic procedures for collecting various types of quality control samples.

3. RESPONSIBILITIES

3.1 Project Manager Responsibilities

The KEMRON Project Manager ensures that any project specific deviations from this procedure will be provided to the Field Staff.

3.2 Field Staff Responsibilities

The KEMRON field staff follow procedures specified in this SOP unless written site-specific protocol is provided by the Project Manager.

4. DEFINITIONS

4.1 Quality Control Samples

Quality control (QC) samples are collected during field studies for various purposes which include the isolation of site effects (control samples), define background conditions (background sample), evaluate field/laboratory variability (spikes and blanks, trip blanks, duplicate, split samples).

4.2 Control Sample

Control samples are typically a discrete grab sample collected to isolate a source of contamination. Isolation of a source could require the collection of both an upstream sample at a location where the medium being studied is unaffected by the site being studied, as well as a downstream control which could be affected by contaminants contributed from the site under study.

4.3 Background Sample

A background sample is a sample (usually a grab sample) collected from an area, water body, or site similar to the one being studied, but located in an area known or thought to be free from pollutants of concern.

4.4 Split Sample

A split sample is a sample which has been portioned into two or more containers from a single sample container or sample mixing container. The primary purpose of a split sample is to measure sample handling variability. A split sample will also measure inter- or intra-laboratory precision.

4.5 Duplicate Sample

Duplicate samples are two or more samples collected and representative of a given population. The purpose of a duplicate sample is to estimate the variability of a given characteristic or contaminant associated with a population.

4.6 Trip Blanks

A trip blank is prepared at the time of the sample kit preparation (by lab or field office) and remains unopened until analysis. Trip blanks indicate if volatile contamination in the environment of container / sample handling is affecting the associated volatile samples.

4.7 Spikes (also known as proficiency test (pt) samples)

Spikes are a sample with known concentrations of contaminants. Spike samples are often packaged for shipment with other samples and sent for analysis. At no time after their preparation are the sample containers opened before they reach the laboratory. Spiked samples are used to measure bias due to sample handling or analytical procedures.

4.8 Equipment Field Blanks

An equipment field blank is used to determine if contaminants have been introduced by contact of the sample medium with sampling equipment. Equipment field blanks are often associated with collecting rinse blanks of equipment that has been decontaminated.

4.9 Temperature Blanks

A temperature blank is a container of water shipped with each cooler of samples requiring preservation by cooling to $4^{o}C$ (ice). The temperature of the blanks are measured at the time of sample receipt by the laboratory.

4.10 Preservative Blanks

A preservation blank is a sample that is prepared in the field and used to determine if the preservative used during field operations was contaminated, thereby causing a positive bias in the contaminant concentration.

4.11 Field Blanks

A field blank is a sample that is prepared in the field to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected (for example air-borne dust or organic vapors which could contaminate a soil sample).

4.12 Material Blanks

A material blank is a sample of sampling materials (e.g., material used to collect wipe samples, etc.), construction materials (e.g., well construction materials), or reagents (e.g., deionized water generated in the field, water from local water supplies used to mix well grout, etc.) collected to measure any positive bias from sample handling variability. Commonly collected material blanks are listed below:

4.12.1 Wipe Sample Blanks

Wipe sample blanks are a sample of the material used for collecting wipe samples.

4.12.2 Grout Blanks

Grout blanks are a sample of the material used to make seals around the annular space in monitoring wells.

4.12.3 Filter Pack Blanks

Filter pack blanks are a sample of the material used to create an interface around the screened interval of a monitoring well.

4.12.4 Construction Water Blanks

Construction water blanks are a sample of the water used to mix or hydrate construction materials such as monitoring well grout.

4.12.5 Deionized Water Blanks

A deionized water blank is a sample collected from a field deionized water generating system. The purpose of the deionized water blank is to measure positive bias from sample handling variability due to possible localized contamination of the deionized water generating system or contamination introduced to the sample containers during storage at the site.

5. PREPARATION OF FIELD QUALITY CONTROL (QC) SAMPLES

The following procedures are basic procedures, should be used in conjunction with the field SOP and may not apply to a particular project. Project documents will specify what types of QC are necessary.

5.1 Control Sample

5.1.1 Collect the control sample in the same way a study sample is collected, i.e., composite, grab, etc.

5.1.2 Handle the control sample in the same way a study sample is collected, i.e., container, labeling, preservation.

5.2 Background Sample

5.2.1 Collect the background sample in the same way a study sample is collected, i.e., composite, grab, etc. and in the area prescribed by the project documents.

5.2.2 Handle the background sample in the same way a study sample is collected, i.e., container, labeling, preservation.

5.3 Split Sample

5.3.1 Collect a study sample which is approximately double in size than what is normally used.

5.3.2 Homogenize the sample.

5.3.3 Split the sample into two portions.

5.3.4 Aliquot each portion in the appropriate sample containers with the appropriate preservation.

5.3.5 Ship one portion of the split sample with the batch of samples to the laboratory prescribed in the project documents and ship the second portion to a second laboratory as prescribed in the project documents.

Note: Split samples are not collected for volatiles.

5.4 Duplicate Sample

5.4.1 Collect a second study sample from the same location / area / bucket in the same manner as that of the study sample.

5.4.2 Handle the control sample in the same way a study sample is collected, i.e., container, labeling, preservation.

5.5 Trip Blank

Trip blanks are prepared at the time of sample kit preparation which may occur at the laboratory or the field office.

5.5.1 Fill a VOA vial full with deionized water, cap and ensure that there are no air bubbles.

5.5.2 Transport the unopened trip blank with the sample kit to the field.

5.5.3 Transport the unopened trip blank with the samples to the laboratory for analysis

Note: The trip blank is analyzed for volatile organic compounds.

5.6 Spikes

Spikes are generally prepared at the laboratory prior to analysis; however, there are occasions where the project may require field prepared spike samples.

5.6.1 Purchase or prepare a spike or proficiency test sample as stated in the project documents. Prepare a spike sample by fortifying a background sample with the target analytes of interest. Prepare an unfortified background sample also.

5.6.2 Submit the spike sample, and background sample as necessary, to the laboratory for analysis as a “blind” sample.

5.6.3 Evaluation occurs upon receipt of the sample results.

5.7 Equipment Blanks

5.7.1 Bring extra deionized water to the field.

5.7.2 Pour deionized water over the sampling equipment following equipment cleaning and collect in an appropriate sample container.

5.7.3 Handle the sample container in the same fashion as an aqueous sample is handled, i.e. container, preservation, etc.

5.7.4 Collect enough equipment blanks to analyze for all the target analytes of interest or as stated in the project documents.

5.7.5 Transport the sample to the laboratory for analysis with the samples.

5.8 Temperature Blanks

5.8.1 Fill a small container with water. (Sample kits should already contain a temperature blank).

5.8.2 Transport the temperature blank with the samples to the laboratory.

5.8.3 Provide a temperature blank with each cooler or sample transport container.

5.9 Preservative Blanks

5.9.1 Prepare a preservative blank at the appropriate stage and in the frequency as stated in the project documents.
5.9.2 Bring extra deionized water to the field.
5.9.3 Fill the same type bottle used for sample collection with deionized water
5.9.4 Add the appropriate type and amount of chemical preservative.
5.9.5 Transport the sample to the laboratory with the samples.

5.10 Field Blanks

5.10.1 Bring extra deionized water to the field.
5.10.2 Pour the water into the appropriate sample containers at pre-designated locations at the site, according to project documents.
5.10.3 Collect in dusty environments and/or from areas where volatile organic contamination is present in the atmosphere and originating from a source other than the source being sampled.
5.10.4 Add the appropriate chemical preservative according to the analysis.
5.10.5 Transport the sample to the laboratory with the samples.

5.11 Wipe Sample Blanks

5.11.1 Bring extra wipe samples to the site.
5.11.2 Handle, package and transport unused wipe material as that for wipes used for sampling.

5.12 Grout Blanks

5.12.1 Take a sample of the grout used to seal the monitoring well.
5.12.2 Place in a suitable container and transport to the laboratory.

5.13 Filter Pack Blanks

5.13.1 Take a sample of the filter pack used in assembling of the monitoring well.
5.13.2 Place in a suitable container and transport to the laboratory.

5.14 Construction Water Blanks

5.14.1 Take a sample of the water used in preparing construction materials.
5.14.2 Place in a suitable container and transport to the laboratory.

5.15 Deionized Water Blanks

5.15.1 Take a sample of the water generated from the field deionized water source at the end of sampling activities or in intervals as stated in the project documents.
5.15.2 Take the sample in clean environment so external conditions are not affecting the sample.
5.15.3 Place in a suitable container and transport to the laboratory.

6. REFERENCES

Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, EPA Region IV, November 2001

Document Control #: 07-PAS-00

STANDARD OPERATING PROCEDURE FOR SAMPLE PACKAGING AND SHIPPING

Issue Date: November 8, 2004
Revision 00

Last Review/Implementation Date: November 2004

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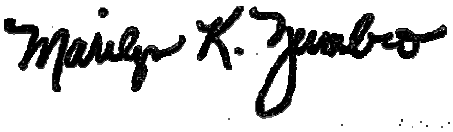

11/8/04

Marilyn Zumbro, Regional Manager — Date

11/8/04

John Dwyer, Vice President — Date

11/8/04

Kelly Clemons, Corporate Quality Assurance Manager — Date

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The following persons have read and understand this SOP:

Signature	Date

1. PURPOSE

The purpose of this procedure is to ensure consistent sample packaging and shipping at KEMRON.

2. SCOPE

This procedure addresses the packaging of samples and proper shipping protocol.

3. RESPONSIBILITIES

3.1 Project Manager

To ensure the field staff follows the procedure as stated and/or to provide written deviations to the SOP.

3.2 Field staff

To follow SOP as stated and any written deviations provided by the Project Manager.

4. POLICY

Samples collected during field investigations or in response to a hazardous materials incident are classified prior to shipment, as either environmental or hazardous materials samples. See Attachment B for the KEMRON policy on shipping, entitled “Shipping Guidelines”.

5. DEFINITIONS

5.1 Hazardous Materials

Materials which meet specific definitions under US DOT regulations which are subject to restrictive shipping requirements including employee training, packaging, labeling, and documentation. See Attachment A

5.2 Environmental Sample

A sample of solid waste, water, soil or air which is collected for the sole purpose of testing to determine characteristics or composition and are not subject to 40 CFR Part 262 through 268 (including use of hazardous waste manifests), if certain conditions of shipment are met.

(Refer to Appendix D of the Sample Shipping Procedures, Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, EPA Region 4, November 2001, (http://www.epa.gov/region4/sesd/eisopqam/eisopqam.html), and "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), US-EPA, April 13, 1981.

5.3 Dangerous Goods

US DOT classified Hazardous Materials may be classified as Dangerous Goods when shipped by air and be subject to further regulatory requirements and/or shipping restrictions than that stated for Hazardous Materials.

According to the International Air Transportation Association (IATA), Dangerous goods are articles or substances which are capable of posing a risk to health, safety, property or the environment and which are shown in the list of dangerous goods in these Regulations or which are classified according to these Regulations.

6. SHIPMENT OF DANGEROUS GOODS (BY AIR)

The Project Manager is responsible for determining if samples collected during a specific field investigation meet the definitions for hazardous materials and/or dangerous goods. When shipping dangerous goods by air, all samples are packaged by a trained and certified employee according to Attachment B. Check with the air shipping carrier to ensure all carrier shipping requirements are met. For additional information, see the IATA Manual.

7. SHIPMENT OF ENVIRONMENTAL SAMPLES

Samples determined by the Project Manager to be environmental samples are shipped using the following protocol, developed jointly between US-EPA, OSHA, and DOT. This procedure is documented in the "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Environmental Laboratory Samples" (3).

Untreated wastewater and sludge from POTW's are considered to be "diagnostic specimens" (not environmental laboratory samples). However, because they are not considered to be etiologic agents (infectious) they are not restricted and may be shipped using the procedures outlined below.

7.1 Shipping Environmental Samples by Commercial Carrier (Air or Ground)

Environmental samples should be packed prior to shipment using the following procedures and in compliance with all carrier requirements:

7.1.1 Allow a small amount of headspace in all bottles (except VOC containers with a septum seal) to compensate for any pressure and temperature changes.

7.1.2 Be sure the lids on all bottles are tight (will not leak). Lids maybe taped or sealed with custody seals as added protection or as required.

7.1.3 Place bottles in separate and appropriately sized polyethylene bags and seal the bags with tape.

7.1.4 Include one temperature blank per cooler.

7.1.5 Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape.

7.1.6 Line the cooler with a large heavy duty plastic bag.

7.1.7 Place an appropriate amount of bubble wrap in the bottom of the cooler and then place the bottles in the cooler with sufficient space to allow for the addition of bubble wrap between the bottles and cans.

7.1.8 Put "double bagged" ice (in heavy duty polyethylene bags and properly sealed) on top of and/or between the samples. Fill all remaining space between the bottles or cans with bubble wrap.

7.1.9 Securely fasten the top of the large garbage bag with tape (preferably plastic electrical tape).

7.1.10 Place the Chain-of-Custody Record into a plastic bag, and tape the bag to the inner side of the cooler lid.

7.1.11 Close the cooler and securely tape (preferably with fiber tape) the top of the cooler shut.

7.1.12 Chain-of-custody seals should be affixed to the top and sides of the cooler within the securing tape so that the cooler cannot be opened without breaking the seal.

7.1.13 Shipping containers must be marked "THIS END UP", and arrow labels which indicate the proper upward position of the container should be affixed to the container. A label containing the name and address of the shipper should be placed on the outside of the container. Labels used in the shipment of hazardous materials (e.g., Cargo Only Air Craft, Flammable Solids, etc.) are not permitted to be on the outside of containers used to transport environmental samples.

7.2 **Hand Delivery of Environmental Samples (by employee or courier)**

7.2.1 Bottles (required for VOC containers with a septum seal) can be filled completely with sample.

7.2.2 Be sure the lids on all bottles are tight (will not leak).

7.2.3 Place bottles in a sturdy cooler with sufficient space to allow for the addition of bubble wrap between the bottles.

7.2.4 Include one temperature blank per cooler.

7.2.5 Put "double bagged" ice (in heavy duty polyethylene bags and properly sealed) on top of and/or between the samples.

7.2.6 Place COC in plastic bag and tape to the inside of the cooler lid, close lid, seal with custody seals and transfer the courier. Alternatively, when delivering by hand, close the cooler and hand deliver with the chain of custody.

8. ASBESTOS SHIPPING

See Figure 1

8.1 Place each asbestos sample in a small Ziploc bag.

8.2 Place the bags of asbestos samples in a large Ziploc bag, as needed.

8.3 Place in a sturdy box for shipping (i.e., FEDEX box)

8.4 Use bubble wrap to prevent the sample(s) from moving around in the box.

8.5 Insert COC in the box.

8.6 Seal box and place “Danger” stickers on the box.

8.7 Affix chain-of-custody seals to the top and sides of the box within any securing tape so that the box cannot be opened without breaking the seal.

9. AIR QUALITY SAMPLE SHIPPING

9.1 Place air samples in a sturdy box suitable for the type of sampling media (i.e., tenax tubes, tedlar bags, summa canisters)

9.2 Use bubble wrap to prevent the sample(s) from moving around in the box.

9.3 Insert COC in the box

9.4 Seal box

9.5 Affix chain-of-custody seals to the top and sides of the box within any securing tape so that the box cannot be opened without breaking the seal.

10. REFERENCES

10.1 Dangerous Goods Regulations, International Air Transport Authority (IATA). Current Edition.

10.2 EPA Order 1000.18, February 16, 1979.

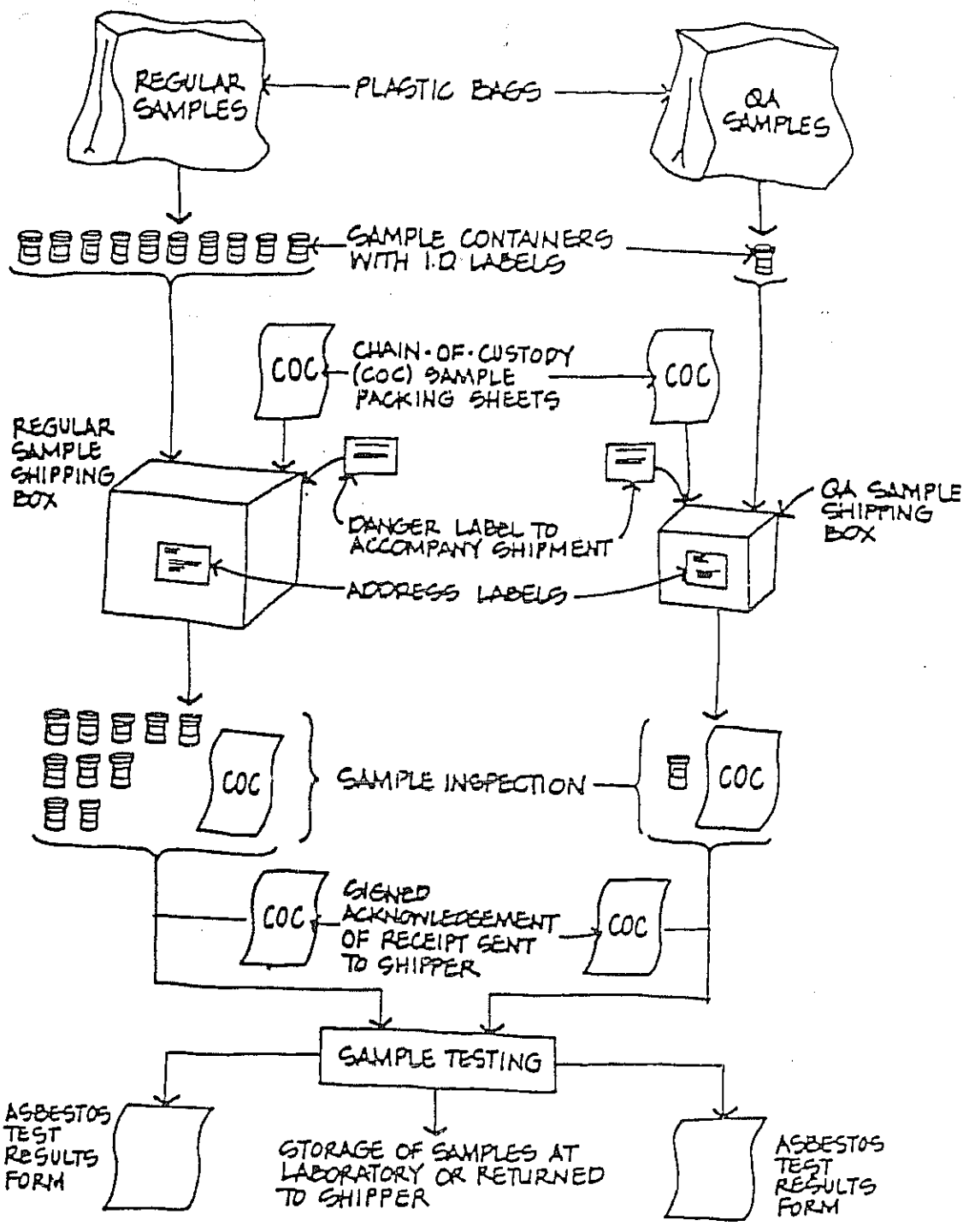
10.3 "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), US-EPA, April 13, 1981.

10.4 40 CFR 136.3. July 1, 2001. See Table 11, Footnote 3.

10.5 Environmental Investigation Standard Operating Procedures and Quality Assurance Manual, USEPA, Region 4, 11/2001

FIGURE 1 – ASBESTOS SAMPLE PACKAGING PROCEDURES

EXHIBIT K-2
SAMPLE HANDLING PROCEDURES



ATTACHMENT A – DEFINITION OF HAZARDOUS MATERIALS

HAZARDOUS MATERIAL DEFINITION

DOT Definition of Hazardous Material:

A hazardous material is defined as any substance or material could adversely affect the safety of the public, handlers or carriers during transportation. All DOT hazardous materials are listed in the DOT's Hazardous Material Table.

There are nine classes of hazardous materials:

Hazard Class 1: Explosives	1.1 mass explosion hazard 1.2 projectile hazard 1.3 minor blast/projectile/fire 1.4 minor blast 1.5 insensitive explosives 1.6 very insensitive explosives
Hazard Class 2: Compressed Gases	2.1 flammable gases 2.2 non flammable compressed 2.3 poisonous
Hazard Class 3: Flammable Liquids	Flammable (flash point below 141°) Combustible (flash point 141°-200°)
Hazard Class 4: Flammable Solids	4.1 flammable solids 4.2 spontaneously combustible 4.3 dangerous when wet
Hazard Class 5: Oxidizers and Organic Peroxides	5.1 Oxidizer 5.2 Organic Peroxide
Hazard Class 6: Toxic Materials	6.1 Material that is poisonous 6.2 Infectious Agents
Hazard Class 7: Radioactive Material	Radioactive I Radioactive II Radioactive III
Hazard Class 8: Corrosive Material	Destruction of the human skin Corrode steel at a rate of 0.25 inches per year
Hazard Class 9: Miscellaneous	A material that presents a hazard during shipment but does not meet the definition of the other classes

ATTACHMENT B – SHIPPING GUIDELINES

KEMRON
ENVIRONMENTAL SERVICES

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MEMO

TO: Lisa Larson, Heather Moorehead, Okey Tucker, Jeannette Hamm, Doug Barnett, Victoria Anderson

FROM: Marilyn Zumbro MKZ

CC: Matt Davey, Chris Amick, Tom O'Hara, Marsha Robinson, Pat Bolger, John Dwyer, Kelly Clemons

DATE: May 13, 2004

RE: **SHIPPING GUIDELINES**

The purpose of this memo is to provide information and additional instruction regarding procedures for the classification and shipment of materials by KEMRON personnel. During the course of normal project activities, KEMRON personnel may be required to ship materials including samples, wastes, chemicals, supplies, calibration solutions/gases, etc. Certain materials which meet specific definitions under US Department of Transportation (DOT) regulations are considered "hazardous materials" and are subject to restrictive shipping requirements including employee training, packaging, labeling, and documentation. Such materials, when shipped by air, may be classified as "dangerous goods' and be subject to further regulatory requirements and/or shipping restrictions.

The following guidelines will be used by KEMRON personnel in determining if materials being offered for shipment are classified as hazardous under DOT regulations. All shipments of hazardous materials by KEMRON must conform to DOT requirements and be prepared and offered for shipment ONLY by personnel who have documented training in accordance with regulatory requirements. ALL shipments of hazardous materials must be approved/authorized by Program/Regional Management.

The following materials are considered hazardous by DOT and must be shipped in accordance with DOT HMT requirements:

- Materials designated as hazardous by listing in the Hazardous Materials Table (49 CFR 172.101);
- Hazardous wastes requiring manifesting;
- Hazardous substances listed in Appendix A to 49 CFR Part 172.101, and shipped in a quantity in one package in excess of the listed RQ;
- Mixtures or solutions of hazardous substances exceeding concentration limits corresponding to RQs as listed in table in 49 CFR Part 171.8;
- Substances or samples of substances meeting the defining criteria of any DOT hazard class or division;
- Other specifically listed DOT regulated hazardous materials such as marine pollutants and elevated temperature materials.

It is important to remember that a hazardous waste, for purposes of DOT, is a material which is subject to the Hazardous Waste Manifest Requirements under USEPA regulations contained in 40 CFR Part 262. Per 40 CFR Part 261.4, samples of solid waste, water, soil, or air which are collected for the sole purpose of testing to determine characteristics or composition, are not subject to 40 CFR Part 262 through 268 (including use of hazardous waste manifests), if certain conditions of shipment are met. Many samples shipped by KEMRON will meet the RCRA criteria for exemption from

provisions of 40 CFR Parts 262 through 268, including hazardous waste manifesting, and will therefore, not meet the DOT definition of a hazardous waste.

This does not mean that all environmental samples are exempt from DOT requirements. Your shipment must pass all the definitional tests in order be excluded by definition. Ask the following questions about the shipment.

- Is it designated in the HM table (49 CFR 172.101)?
- Is it a hazardous waste requiring manifesting?
- Is it a hazardous substance (listed in Appendix A to 49 CFR Part 172.101) in a quantity in one package which exceeds the RQ?
- If it is a hazardous substance mixture or solution, is it in a concentration by weight that exceeds the corresponding RQ of the material as listed in the table found in 49 CFR Part 171.8 *Hazardous Substance* (3)(ii)?
- Does it meet the defining criteria of any DOT hazard class or division?
- Is it specifically listed as a marine pollutant?
- Does it meet the definition of an elevated temperature material?

If the answer to any of these questions is yes, the shipment is regulated by DOT as a hazardous material and subject to specific shipping requirements. Consult with your management to assure that properly trained personnel prepare and offer the shipment and that the shipment meets all regulatory requirements.

ATTACHMENT C – CHAIN OF CUSTODY

COC No. A 39472

109 Starlite Park
Marietta, OH 45750

KEMRON
ENVIRONMENTAL SERVICES

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name:

Project Contact:	Contact Phone #:
Turn Around Requirements:	Location:
Project #:	Project Name:
Sampler (print):	Signature:

Program
- ☐ NPDES
- ☐ AFCEE
- ☐ RCRA
- ☐ USAGE
- ☐ Other ______

Sample I.D. No.	Comp*	Grab	Date	Time	Protocol CWA	Protocol SW846	NUMBER OF CONTAINERS	Hold																	ADDITIONAL REQUIREMENTS

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Relinquished by: (Signature)	Date	Time	Received by: (Signature)
Relinquished by: (Signature)	Date	Time	Received for Laboratory by: (Signature)	Date	Time	Cooler Temp in °C	Remarks:

*Homogenize all composite samples prior to analysis

Page ______ of ______

Document Control #: 12-GWS-01

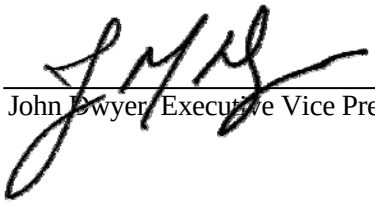
STANDARD OPERATING PROCEDURE FOR GROUNDWATER SAMPLING

Issue Date: November 8, 2004

Revision 01
Last Review/Implementation Date: October 11, 2010

Approved by:

Lou Ehrhard, Corporate Quality Assurance Manager — 10/11/2010 Date



John Dwyer, Executive Vice President — 10/11/2010 Date

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1. PURPOSE

The objectives of this procedure are to ensure valid and representative samples of groundwater are collected from monitoring wells that meet project requirements and to prevent deterioration and contamination of samples before laboratory analysis. By complying with procedures specified in this Standard Operating Procedure (SOP), KEMRON will ensure that the following are successfully completed:

- Proper well purging
- Proper sampling
- Prevention of contamination or cross-contamination of groundwater samples during collection, containerization, transit and storage
- Obtain and record accurate and complete sampling data
- Preparation of complete and correct sample labels
- Preparation and filing of sample chain of custody
- Communication of adequate instruction to the laboratory

2. SCOPE

This SOP provides technical guidance on groundwater sampling including decontamination procedures, types, and uses of sampling equipment. This SOP applies to KEMRON personnel and subcontractors collecting groundwater samples for KEMRON projects. Client, federal, or state requirements, or project-specific requirements may dictate specific types of equipment or procedures to be used when applying this SOP to a particular project. Deviations from this SOP to accommodate site-specific requirements shall be documented in project planning documents and approved by the KEMRON Project Manager and Project Quality Assurance (QA) Manager, if applicable, prior to the performance of work.

3. RESPONSIBILITIES

Project Manager Responsibilities
The KEMRON Project Manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project-specific work plan. The KEMRON Project Manager ensures all field staff have reviewed and signed off on this SOP prior to beginning field work. A sign-off sheet covering all SOPs related to the project will be prepared and filed with project documents.

Field Staff Responsibilities
The KEMRON field staff prepares, organizes, and inspects all equipment required for the sampling event prior to the initiation of sampling activities. The field sampler and shipper are responsible for initiating, maintaining, and transferring chain-of-custody. Upon completion of the sampling event, the field staff returns all equipment to the designated storage area ensuring that the equipment is decontaminated and in a condition ready for reuse. KEMRON field staff

are required to follow procedures specified in this SOP, or approved project-specific SOP, prior to initiating the sampling event. Field staff are required to sign off that they have read and understand this SOP prior to beginning field work.

4. DEFINITIONS

Bailer: A bailer is used to sample wells. A bailer may be of stainless steel, Teflon, polyethylene or other appropriate construction and may be composed of a reel system equipped with a Teflon-coated stainless steel suspension cord, a disposable monofilament line or disposable braided filament line. A bailer can be dedicated or non-dedicated to a specific well and disposable or reusable.

Chain-of-Custody: An unbroken trail of accountability that ensures the physical security of samples, data, and records. The chain-of-custody documentation shows times, dates, and names of the individuals relinquishing and receiving the samples identified on the record.

Custody: Physical control of an object, in this case of an environmental sample.

Dedicated: Equipment (bailer or pump) dedicated to a particular well which remains associated only with the assigned well until the work/project is completed.

Depth to Water: The depth to water is the depth from the top of the well casing to the surface of the groundwater. An established point (typically the surveyed top of the well casing) is utilized as a reference point for measuring the depth to water.

Evacuated Well: Removal of standing volume of water in a well by pumping or bailing.

Field Blank: A field blank sample undergoes the full handling and shipping process of an actual sample. It is designed to detect sample contamination that can occur during field operation or during shipment. Field blanks are prepares using certified clean water preserved in the same manner as the other collected samples, and then submitted to the laboratory for analysis.

Flow Rate: A measurement taken of the water flowing from the well when a pump is used. The flow rate is in units of volume per time.

Low-Flow Purge: Per EPA guidance, low-flow purging is purging at rate of 200-500 mL/min to have minimal drawdown of the water table and minimize disruption of the water column. Low-flow purging is accomplished using low-flow purge equipment.

Non-Dedicated: Equipment (bailer or pump) not dedicated to a particular well. Non-dedicated equipment is decontaminated between use or if for one-time use (bailers) then properly disposed of after use.

Purging: The process of removing stagnant water from a monitoring well (water in the screen, well casing, and saturated annulus) immediately prior to sampling, causing its replacement by groundwater from the adjacent formation, which is representative of actual aquifer conditions.

Recharged Well: A well is considered recharged when the water level recovers to at least 80% of the static water level.

Slow Recovery Wells: Slow recovery wells are wells that require more than 6 hours to recharge after purging.

Stabilized Well: Well stabilization is achieved following well purging when water level and field measurements are observed to be consistent between measurements and within project work plan requirements.

Trip Blank: Unlike field blanks, the trip blanks are used for VOCs analysis only. In addition, trip blanks are prepared prior to going into the field by filling containers (VOC vials) with clean water. The samples are kept closed, handled, transported to the field, and then returned to the laboratory in the same manner as the other samples. Trip blanks are used to evaluate error associated with shipping and handling, and analytical procedures. They are used in conjuncture with field blanks to isolate sources of sample contamination already noted in previous field blanks.

Unrecoverable Wells: Wells that have not sufficiently recovered within 24 hours.

Water Column Height: Well depth minus the depth to water.

Water Level Measurement Reference: The point on the well head where water levels are measured (typically the high point of the casing). This point is marked and surveyed as soon as practical after well installation to ensure consistent measurement.

Water Quality Measurements: Groundwater chemistry measurements used to establish the quality of groundwater. Measurements may include: temperature, pH, conductivity, dissolved oxygen (DO), oxidation reduction potential (ORP), and turbidity.

Well Stability: Stability is achieved as specified in the project work plan. The following are provided as a reference.

- Change in groundwater chemistry measurements is within a specified range (indicating stability) by the end of the third well volume purged; or
- Stabilization is considered complete after five well volumes are purged even if field parameters have not met specified range (range established to indicate stability); or
- Stabilization is complete when well is fully evacuated. Using these criteria, measurements are taken to verify the evacuation. Allow well to recharge prior to sampling.

Well Volume Calculation: The standing water volume for typical well diameters is below:

1" Casing = .041 gal/ft
2" Casing = .163 gal/ft
4" Casing = .653 gal/ft
6" Casing = 1.469 gal/ft

or can be calculated using the following formula:

$$Volume\,(gallons) = (3.14)\,r^2\,h\,x\,(7.48\,\frac{gal}{ft^3})$$

r = inside well casing radius (in feet)
h = height (in feet) of the water column in the well

5. PLANNING

The Project Manager provides written instructions of any project-specific information for the sampling crew. The written instructions can be as simple as a sampling requirements report or as detailed as a Sampling and Analysis Plan (SAP). Items in written sampling instructions may include the following:

a) Purging method;
b) Criteria for a complete purge;
c) Criteria for stabilized well;
d) Wells to sample;
e) Method of sampling;
f) Analytical parameters;
g) Applicable regulations for handling purged water;
h) Volume to purge;
i) Sample containers to be used for each analysis;
j) Preservative for each analysis; and
k) Instructions for samples to be field filtered or lab filtered for dissolved metals, if appropriate.

Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific SAP/Quality Assurance Project Plan (QAPP). Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements typically suggest the collection of a sufficient quantity of quality control (QC) samples such as field duplicate, equipment and/or field blank, and matrix spike/matrix spike duplicate (MS/MSD) samples. These requirements should be outlined in the SAP/QAPP.

Information regarding the sample containers, preservation techniques and holding times for groundwater samples will be described in the project specific SAP/QAPP.

Groundwater sampling may involve chemical hazards associated with the materials being sampled. Adequate health and safety measures must be taken to protect project sampling personnel from potential chemical exposures or other hazards. These measures must be addressed in the project Health and Safety Plan (HASP). This plan must be approved by the project health and safety officer before work commences, must be distributed to all personnel performing sampling, and must be adhered to as field activities are performed.

6. SAMPLE COLLECTION EQUIPMENT AND PROCEDURES

The tables in this section provide a description of equipment and sampling procedures for different groundwater sampling methods. The project work plan will specify the method and any site-specific requirements. Once the groundwater is sampled, the sample will be aliquoted in the following sequence or as stated in the project-specific planning documents: Field Parameters, Volatile Organic Compounds (VOCs); Semi-volatile Organic Compounds (SVOCs), Pesticides, Herbicides, PolyChlorinated Biphenyls (PCBs), Metals, and Wet chemistry parameters.

Groundwater sampling objectives may vary significantly between projects. Project objectives should be defined within the project-specific planning documents. The list of required materials below identifies the types of equipment which may be used for a range of groundwater sampling applications. The various types of sampling equipment which may be used include:

Well Purging Equipment

- Bailers
- Bladder pumps
- Submersible pumps
- Peristaltic pumps
- Centrifugal Pumps
- Waterra™ pumps

Field Instruments

- Individual or multi-parameter meter(s) to measure temperature, pH, specific conductance, DO, ORP, and/or turbidity
- Flow through cell
- Water level measuring device
- Interface probe or product detection paste

Sampling Equipment

- Reusable or disposable bailers
- Bladder Pump
- Electric Submersible Pump
- Suction Lift Pumps (Peristaltic)

Sample Preparation Equipment

- Filtration equipment
- Intermediate containers
- Sample kit (i.e., bottles, labels, preservatives, custody records, cooler)

General Equipment

- Well keys
- Project-specific sampling plans (SAP, QAPP, HASP) including map of wells to be sampled
- Sample collection records
- Field notebook/pen
- Waterproof marker pens
- Deionized water dispenser bottler
- Sample cup
- Buckets
- Coolers, or sample shuttles
- Instrument calibration solutions
- Power source (e.g. generator and/or battery)
- Equipment decontamination supplies
- Health and safety supplies (PPE, etc)
- First-Aid kit
- Tool box (e.g. bolt cutter, turkey baster, etc.)
- 55-gallon drum for collecting purge water
- Graduated cylinder (determine flow)
- Flow controller for submersible pump

Expendable Materials

- Deionized water supply
- Disposable bailer string (nylon or polypropylene)
- 0.45 micron filters
- Paper towels
- Plastic sheeting
- Ice/blue ice for sample preservation
- Disposable latex powder-free glove liners
- Disposable nitrile gloves
- Plastic trash bags
- Ziploc® bags

This equipment list was developed to aid in field organization and should be used in preparation for each sampling event. Depending on the site-specific sampling plan, additional material and equipment may be necessary and should be determined before the scheduled sampling event. Similarly, not all of the items shown in this list may be necessary for any one sampling event.

TABLE 1: Purging and Sampling by Bailer

Purging & Sampling Equipment:	The Bailer system can be dedicated or non-dedicated. a) Bailer: pre-cleaned closed-top Teflon® or stainless steel bailer or a disposable polyethylene bailer. b) Either of the following: (1) New braided nylon or polypropylene cords, one per well (disposable). (2) Reusable lanyards (monofilament, stainless steel, or Teflon-coated) for bailer systems.
Sampling Procedure:	Prior to sampling, obtain sample containers from laboratory (or laboratories). Ensure presence of preservatives in bottles as appropriate. Before opening the well, a clean working surface shall be set up around the well head using a clean plastic sheet. Prior to opening the well, the required health and safety gear (as specified in the HASP) shall be donned. This, at a minimum, usually means wearing gloves to limit the potential for exposure to contaminants as well as reduce the potential for handling-induced contamination of sampling equipment. ***Preliminary Measurements:*** Measure the depth to water at the pre-marked well casing reference point (notch or other visible mark). To obtain a water level measurement, lower the probe of a water level measuring device into the well until the audible sound of the unit is detected or the light on an electronic sounder illuminates. At this time the precise measurement should be determined (to nearest 0.01 feet) by repeatedly raising and lowering the tape to converge on the exact measurement. Obtain the reading from the top of casing (TOC) measuring point. The water level measurement should be entered on the Groundwater Sample Collection Record or in the field log book. The measurement device shall be decontaminated immediately after use with a non-phosphatic detergent and rinsed with distilled water. Generally, only that portion of the tape which enters the water table should be cleaned. It is important that the measuring tape is never placed directly on the ground surface or allowed to become kinked. Measuring devices, including interface probes, which come into contact with free product, will likely require more thorough decontamination. Calculate the water level (water column height) and volume of water in casing. Note: Following water-level measurement, if free phase product is suspected, measure the product thickness with an interface probe as specified in the work plan. If measurable quantities of product are encountered do not sample and notify the Project Manager immediately. Wells containing free product are generally not used for groundwater sampling, since the concentration of contaminants present in the free product can adversely affect the quality of the water sample, lending to a non-representative water sample. ***Purge Well:*** Note: If non-dedicated equipment is used for purging, at least one equipment blank (or as specified in the work plan) will be collected from the decontaminated / cleaned purge equipment. a) Slowly and gently, lower the bailer into the top of the standing water column. Keeping the bailer at the top of the water column is crucial throughout purging especially at the end of purging. b) Allow bailer to fill and remove from the well. Bailer rope should never touch the ground surface at any time during the purge routine. c) Collect purged water in a graduated bucket, etc. to determine the number of well volumes purged. d) Following each well volume purged, take water level and field parameter measurements. e) Containerize the purged water unless specified otherwise in the project work plan.

Purging a well to dryness may occur under some low-yield conditions. When the well recovers, a cascading effect may occur within the screened zone which can volatilize some organic compounds. This may be considered inappropriate by regulatory agencies when VOCs are the target analyte of interest. Purging a well to dryness, then sampling after it has recovered may be acceptable for other target analytes, however. Under low yield conditions, low-flow sampling pumps such as bladder pumps may be required for VOC sample collection.

Stabilize well: Measure and record water level until 80% static water level is achieved (well fully recharged). Groundwater chemistry stability is achieved as specified in the project work plan.

Sampling

a) Sample wells:
   - Within six hours of purging unless it is a slow recovery well or
   - As soon as sufficient recharge water is available or up to 24 hours after purging for slow-recovery wells or wells that purge completely dry.

b) Use one pre-cleaned or disposable bailer per well.
c) Attach new nylon rope or other appropriate cord to the bailer.
d) Slowly immerse the bailer into the top of the water column to reduce agitation and splashing.
e) Fill bailer and promptly remove from well.
f) Record sample observations in field log book.

Aliquot Sample: Aliquot the sample into the containers necessary for both laboratory analysis and any further field measurements. For laboratory analysis, include initials, date and time of sample collection, sample ID, preservative and analysis required on sample container label. Place in cooler and cool to 4° Celsius (C) with wet ice.

Groundwater samples collected for total dissolved metals analyses will be field filtered prior to being placed in sample containers and properly preserved. Groundwater filtration will be performed using a peristaltic pump and a 0.45-micron in-line water filter. Disposable filters are commonly available in 0.45-micron size. Low-capacity or high-capacity cartridges are available and may be selectively used based on sample turbidity.

The filtration of groundwater samples shall be performed either directly from the pump discharge line or from laboratory-supplied intermediate containers. In either case, well purging shall be performed first. Fresh groundwater shall then be filtered directly into sample containers.

Post-Sampling Activities: Several activities need to be completed and documented once ground-water sampling has been completed. These activities include, but are not limited to:

- Ensuring that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible.
- Processing all sample paperwork, including copies provided to laboratory.
- Compiling all field data for site records.
- Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

TABLE 2: Pump Assisted Purging and Bailer Sampling

Purge Equipment:	Motor-driven centrifugal lift pump
Sampling Equipment:	The bailer system can be dedicated or non-dedicated. a) Bailer: pre-cleaned closed-top Teflon® or stainless steel bailer or a disposable polyethylene bailer. b) Either of the following: (1) New braided nylon or polypropylene cords, one per well (disposable); (2) Reusable lanyards (monofilament, stainless steel, or Teflon-coated) for dedicated bailer systems.
Sampling Procedure:	Before opening the well, a clean working surface shall be set up around the well head using a clean plastic sheet. Prior to opening the well, the required health and safety gear (as specified in the HASP) shall be donned. This, at a minimum, usually means wearing gloves to limit the potential for exposure to contaminants as well as reduce the potential for handling-induced contamination of sampling equipment. ***Preliminary Measurements:*** Measure the depth to water at the pre-marked well casing reference point (notch or other visible mark) and calculate the water level (water column height) and volume of water in casing. Note: Following water-level measurement, if free phase product is suspected, measure the product thickness with an interface as specified in the work plan. If measurable quantities of product are encountered do not sample and notify the Project Manager immediately. Ensure that water level meter / interface probes are probes are decontaminated after each use. If multiple wells are to be sampled, work from cleanest to most contaminated based on analytical data and/or field observations made during soil boring / well installation. ***Purge well:*** Note: If non-dedicated equipment is used for purging, at least one equipment blank (or as specified in work plan) will be collected from the decontaminated / cleaned purge equipment. Operate all petroleum fueled power sources or related equipment downwind from the sampling location. The well should not be pumped dry during low-flow purging. 1. Lower the pump / hose assembly into the top of the standing water column. Keep assembly towards top of water column so that the purging will evacuate water from the formation into the screened area of the well and up through the casing to remove the entire static volume. 2. Begin purging the water and calculate flow rate. 3. Determine the time necessary to purge one-half the well screen volume or that specified in the work plan. Take field parameters, depth to water, and flow-rate measurements every five to ten minutes (or more frequently depending on the time necessary to purge one-half the well screen volume). 4. Stop purging when necessary well volume has been removed and parameters have stabilized. 5. Containerize the purged water unless specified otherwise in the project work plan. ***Stabilize well:*** Measure and record water level until 80% static water level is achieved (well fully recharged). Measure and record field parameters until groundwater chemistry stability is achieved as specified in the project work plan. See Appendix A for reference information. ***Sampling*** 1. Sample wells: a) Within six hours of purging unless it is a slow recovery well or b) As soon as sufficient recharge water is available or up to 24 hours after purging for slow-recovery wells or wells that purge completely dry.

	2. Use one pre-cleaned or disposable bailer per well. 3. Attach new nylon rope or other appropriate cord to the bailer. 4. Slowly immerse the bailer into the top of the water column to reduce agitation and splashing. 5. Fill bailer and promptly remove from well. 6. Record sample observations in field log book. ***Aliquot Sample:*** Aliquot the sample into the containers necessary for both laboratory analysis (see Tables 4, 5, 6 and 7) and for any further field measurements. For laboratory analysis, include initials, date and time of sample collection, sample ID, preservative and analysis required on sample container label. Place in cooler and cool to 4° Celsius (C) with wet ice. ***Decontamination:*** After sampling a well, decontaminate all reusable equipment and samplers prior to future use. See Section 7 for field measurements.

TABLE 3: Low Flow Purge and Sample Collection

Purging and Sampling Strategy:	Low-Flow Purge Collection Method with pump
Purge and Sampling Equipment:	Dedicated or non-dedicated equipment may be used. Low-Flow Purge System includes submersible or non-submersible pump, tubing, and suspension cable.
Sampling Procedure:	***Pump Installation and Set-up***: (1) Open the well and allow water level to equilibrate before taking a water level measurement. (2) Measure the depth to water at the pre-marked well casing reference point (notch or other visible mark) and calculate the water level (water column height) and volume of water in casing. (3) Set submersible pump or pump tubing within the screen interval or slightly above screened interval of the well. (4) Place the pump inlet or submersible tubing just below the surface of the water column. (5) Set-up associated equipment (generator, compressor, tubing, etc.) ***Preliminary Measurements:*** Ensure well is fully recharged after pump / tubing installation by taking a water level measurement. Note: Following water-level measurement, if free phase product is suspected, measure the product thickness with an interface probe if and as specified in the work plan. If measurable quantities of product are encountered do not sample and notify the Project Manager immediately. ***Purge and Stabilize Well:*** Note: If non-dedicated equipment is used for purging, at least one equipment blank (or as specified in work plan) will be collected from the decontaminated / cleaned purge equipment. Operate all petroleum fueled power sources or related equipment downwind from the sampling location. The well should not be pumped dry during low-flow purging. The intent of low flow purging is to minimize drawdown. 1. Lower the pump inlet as the well is purged. 2. Calculate Flow Rate of purged groundwater (range 200 - 500 ml per minute). 3. Flow rate may be adjusted at this point but flow-rate should not exceed the recharge rate of the aquifer. Monitor by measuring the top of the water column with a water level recorder or similar device while purging. Continue to measure the water level flow-rate until stability is achieved. 4. A maximum of a 0.3 foot draw down produced by the pump is allowed. 5. Take field parameters, depth to water, and flow-rate measurements every 5–10 minutes (or more frequently depending on the time necessary to purge one-half the well screen volume). 6. Containerize the purged water unless specified otherwise in the project work plan. 7. Sampling occurs after the water level and groundwater chemistry parameters are stable as specified in this SOP or in project work plans. ***Sampling:*** 1. Reduce the flow to a low level to minimize sample disturbance, if necessary, and collect sample directly from the discharge tubing connected to the pump. Discharge tubing should not contact the inside of the sample containers. 2. Aliquot samples according to the number and analysis type as specified in the work plan and include initials, date and time of sample collection, sample ID, preservative and analysis required on sample container label. 3. Place in cooler and cool to 4° Celsius (C) with wet ice. 4. Record sample observations in field log book.

	Decontamination: After sampling, decontaminate the low-flow purge equipment prior to further use. See Section 7 for field measurements.

7. FIELD MEASUREMENTS

Unless specified otherwise in the project documents, the following measurements will be taken and evaluated for acceptability:

Parameter	**Acceptable Range for establishing stability***
Temperature	10% of reading
pH	±0.2 standard units
Conductivity	±3% of readings
Dissolved oxygen	±10% of reading
Redox potential(ORP or Eh)	±20 mV
Turbidity*	≤5 NTU or ±10% reading, whichever is greater

* Three consecutive acceptable measurements typically establish water quality.

8. FIELD RECORDS

8.1 Field Logbook

See the Field Documentation SOP, 07-DOC-00, for generic documentation items. Examples of specific groundwater sampling documentation include the following:

a. Facility name;
b. Date and time of arrival;
c. Weather conditions, including ambient air temperature and/or miscellaneous observations;
d. Location;
e. Well designation;
f. Well sequence number;
g. Static water level;
h. Total depth of well;
i. Depth to water;
j. Casing diameter;
k. Well volume or one-half well screen volume;
l. Method of purging;
m. Start time of purging;
n. Total volume of water evacuated from well;
o. Evacuation completion time;
p. Sampling strategy (bailer manual, bailer automated, low-flow);
q. Flow rate recorded (if applicable);
r. Field equipment calibration check;

s. Water quality measurements;
t. Water level at time of sample collection;
u. Date and time sample collected;
v. Sample sequence number;
w. Sample Equipment ID;
x. Other pertinent observations of water samples (color, turbidity, odor, etc.);
y. Fractions sampled and preservation method;
z. Signature of sampler(s) and date; and
aa. Miscellaneous information, i.e. well was purged dry prior to taking 3 evacuation volumes.

8.2 Chain-of-Custody

Completed chain-of-custody (COC) forms are maintained for all samples. A sample COC form is included as Attachment A. The original form is sent to the laboratory with the samples and signed by the lab upon receipt. Original completed COC forms are returned to KEMRON with the analytical report. A copy of the COC will be retained by the field crew and kept with field records.

9. DECONTAMINATION

All equipment taken to the field will be clean. Decontamination of equipment is performed:

- During a sampling event for multiple uses of equipment,
- At the conclusion of equipment use at the site, or
- If visual evidence indicates a need for decontamination at the onset of equipment use. This does not apply for dedicated equipment.

The following procedures, or those specified in the site-specific documents, are completed for reusable non-dedicated equipment:

1. Hand wash, or flush / pump through automatic samplers/equipment, with a non-phosphate laboratory detergent dissolved in tap water;
2. Rinse with tap water;
3. Rinse thoroughly with deionized water;
4. Allow to air dry;
5. Wrap in aluminum foil and a plastic bag, if needed, and store in a clean, dry area;

Decontamination solvent such as isopropanol, acetone or hexane may be used for equipment exposed to high levels of organics (e.g., NAPL). Use of specific decontamination solvents should be addressed in project-specific planning documents and the waste solvents managed and disposed in accordance with applicable regulations.

10. SHIPPING

Equipment decontamination chemicals may need to be shipped to the site and samples may need to be shipped to the laboratory. All shipping is completed in accordance with the current federal and international regulations (DOT, EPA, IATA). Shipping stipulations are to be included in the

project documents, but should you have any questions regarding shipping of any materials to and from the site, contact the authorized shipping representative in your home office.

11. ASSOCIATED SOPs

Field Log Book 07-DOC-00
Well Installation 12-MWC-00

12. REFERENCES

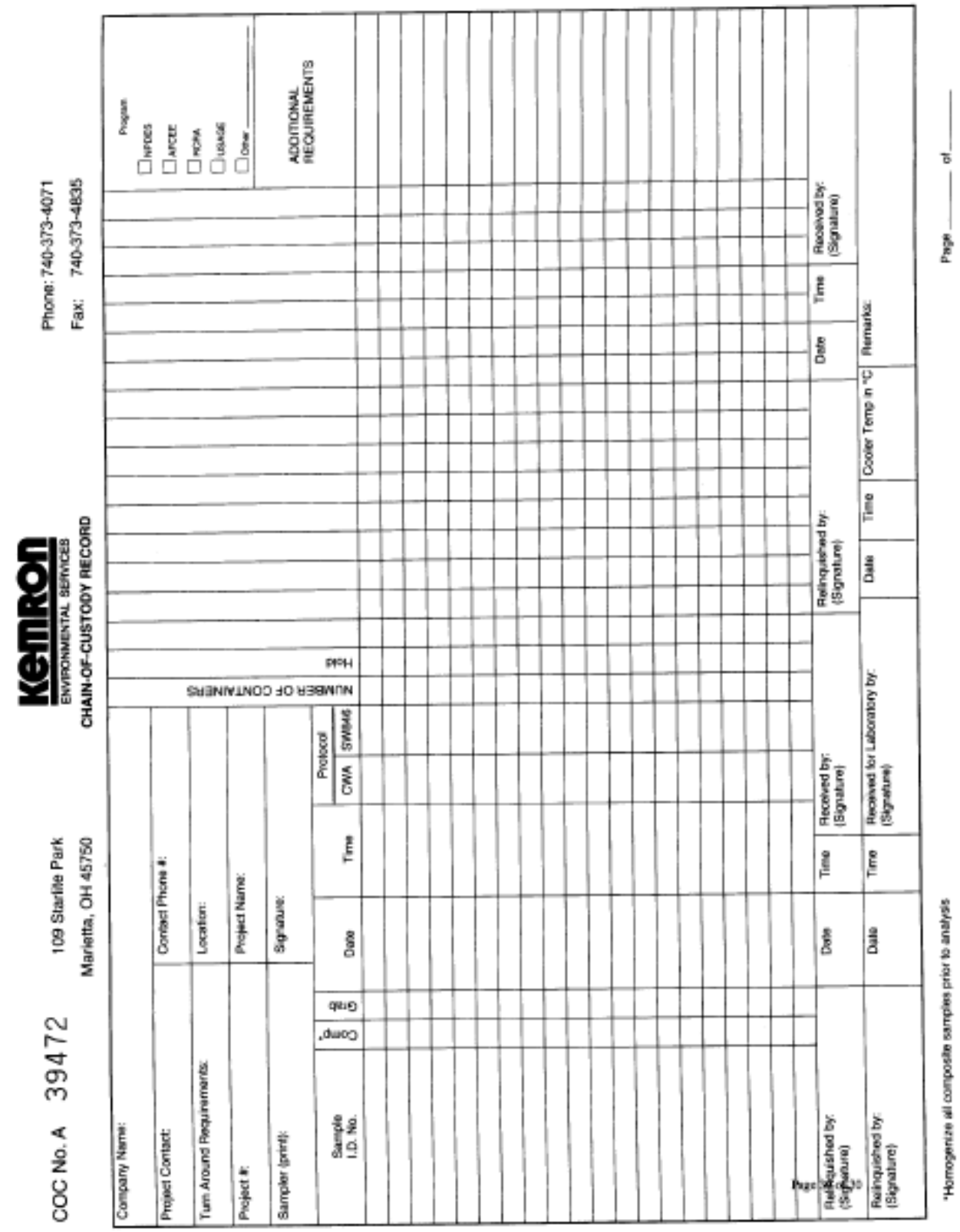
USEPA, Ground-Water Sampling Guidelines for Superfund and RCRA Project Managers, EPA 542-S-02-001, May 2002.

USEPA, Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedure, EPA 540-S-95-504, April 1996.

USEPA, RCRA Groundwater Monitoring, Draft Technical Guidance, November 1992.

USEPA, Potential Source of Error in Groundwater Sampling at Hazardous Waste Sites, EPA 540-S-92-019, August 1992.

ATTACHMENT A – EXAMPLE CHAIN-OF-CUSTODY



KEMRON ENVIRONMENTAL SERVICES
CHAIN-OF-CUSTODY RECORD
COC No. A 39472
109 Starlite Park
Marietta, OH 45750
Phone: 740-373-4071
Fax: 740-373-4835

Company Name:
Project Contact:
Contact Phone #:
Turn Around Requirements:
Location:
Project #:
Project Name:
Sampler (print):
Signature:

Program: NPDES, AFCEE, RCRA, USACE, Other

Sample I.D. No. | Comp.* | Grab | Date | Time | Protocol: CWA, SW846 | NUMBER OF CONTAINERS: HNO3

ADDITIONAL REQUIREMENTS

Relinquished by: (Signature) | Date | Time | Received by: (Signature)
Relinquished by: (Signature) | Date | Time | Received for Laboratory by: (Signature) | Date | Time | Cooler Temp in °C | Remarks:

*Homogenize all composite samples prior to analysis

Page ____ of ____

ATTACHMENT B – EXAMPLE GROUNDWATER SAMPLE COLLECTION FORM

Well Number _____ Project Name:
Sample No. _____ Project Number:
Date _____ Collectors:

Well Information

Monument Condition ☐Good ☐Needs Repair_____
Well Cap Condition ☐Good ☐Locked ☐Replaced ☐Needs Replacement
Elevation Mark ☐ Yes ☐ Added ☐other
Well Diameter ☐ 2-inch ☐4-inch ☐6-inch ☐Other
☐Odor Comments _____

Purge Data

Total Well Depth _____ ft ☐ Clean Bottom ☐Muddy Bottom ☐Not Measured
Depth to Water _____ ft
Casing Volume _____ ft (H20) X _____ gpf = _____ X 3 = _____ gallons
3/4 " = 0.02 gpf 1"= 0.04 2" = 0.16 gpf 4"=0.65 gpf 6"=1.47 gpf

Purge Method

Pump Type: Tubing: Total volume purged _____
Sample Intake Depth _____ Purge Rate _____
Purge Start Time _____ Purge Stop Time _____ Sample Rate _____

Field Parameters

Time	Gallons	pH	Turbidity (NTU)	Temperature (C)	Conductivity (mS/cm)	D.O. (mg/L)	Comments

Sampling Device

Filter Type: Size:

Sample Containers Collection Time_____

Tag No.	Type		Preservative	Analytical Method	QA Remarks
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			

Document Control #: 12-SSP-01

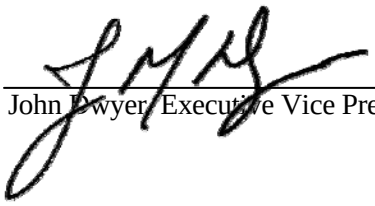
STANDARD OPERATING PROCEDURE FOR SOIL SAMPLING IN THE FIELD

Issue Date: November 8, 2004

Revision 01
Last Review/Implementation Date: October 11, 2010

Approved by:

Lou Ehrhard, Corporate Quality Assurance Manager — 10/11/2010 Date



John Dwyer, Executive Vice President — 10/11/2010 Date

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1. PURPOSE

The objectives of soil sampling are to ensure that valid and representative samples are collected that meet the requirements of the project, and to prevent deterioration and contamination of the samples before laboratory analysis. By complying with procedures specified in this Standard Operating Procedure (SOP), KEMRON will ensure that the following are successfully completed:

- Proper excavation.
- Proper sampling.
- Prevention of contamination or cross-contamination of soil samples during collection. containerization, transit and storage.
- Obtain and record accurate and complete sampling data.
- Preparation of complete and correct sample labels.
- Preparation and maintenance of sample chain-of-custody.

2. SCOPE

This procedure provides technical guidance on soil sampling including decontamination procedures, types, and uses of sampling equipment. This SOP applies to KEMRON personnel and subcontractors collecting soil samples for KEMRON projects. Client, federal, or state requirements, or project-specific requirements may dictate specific types of equipment or procedures to be used when applying this SOP to a particular project. Deviations from this SOP to accommodate site-specific requirements shall be documented in project planning documents and approved by the KEMRON Project Manager and Project Quality Assurance (QA) Manager, if applicable, prior to the performance of work.

3. RESPONSIBILITIES

Project Manager Responsibilities
The KEMRON Project Manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project-specific work plan. The KEMRON Project Manager ensures all field staff have reviewed and signed off on this SOP prior to beginning field work. A sign-off sheet covering all SOPs related to the project will be prepared and filed with project documents.

Field Staff Responsibilities
The KEMRON field staff prepares, organizes, and inspects all equipment required for the sampling event prior to the initiation of sampling activities. The field sampler and shipper are responsible for initiating, maintaining, and transferring chain-of-custody. Upon completion of the sampling event, the field staff returns all equipment to the designated storage area ensuring that the equipment is decontaminated and in a condition ready for reuse. KEMRON field staff are required to follow procedures specified in this SOP, or approved project-specific SOP, prior to

initiating the sampling event. Field staff are required to sign off that they have read and understand this SOP prior to beginning field work.

4. DEFINITIONS

bgs: - below ground surface

Dressing Soil Surfaces - Dressing soil surfaces includes removing the exposed soil surface prior to sampling using a stainless steel shovel, spatula, knife, or spoon. The depth of soil removed depends on the technique used to expose the soil (excavation with a backhoe requires more removal than excavation with a shovel).

Homogenization - The process of mixing samples in order to minimize any bias of sample representativeness introduced by the natural stratification of constituents within the sample. To homogenize a sample, place each grab or core material into a single mixing bowl (made of suitable material i.e. stainless steel), remove any large objects such as sticks, leaves or stones, etc. and stir thoroughly with a spoon to homogenize. Scrape the soil from the sides, corners, and bottom of the pan; roll to the middle of the pan; and complete a final mixing in the center of the pan. Flatten the sample, if needed, quarter the sample, and move to the four corners of the pan. Each quarter of the sample should be mixed individually, and then rolled back to the center of the container and the entire sample mixed again. Aliquot as appropriate.

Sample Compositing - Discrete samples are taken and then proportionally combined into one sample container. Composite samples are generally used to estimate the average concentration of the individual samples that make up the composite.

5. PLANNING

The Project Manager provides any project specific information in written instructions for the sampling crew. Written instructions can be as simple as a sampling requirements report or as detailed as a Sampling and Analysis Plan (SAP). Items in written sampling instructions may include a sampling grid, depths to sample, analyses to sample for, etc.

Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific SAP and Quality Assurance Project Plan (QAPP). Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements typically suggest the collection of a sufficient quantity of quality control (QC) samples such as field duplicate, equipment and/or field blank, and matrix spike/matrix spike duplicate (MS/MSD) samples. These requirements should be outlined in the SAP/QAPP.

Information regarding the sample containers, preservation techniques and holding times for soil samples will be described in the project specific SAP/QAPP.

Soil sampling may involve chemical hazards associated with the materials being sampled. Adequate health and safety measures must be taken to protect project sampling personnel from potential chemical exposures or other hazards. These measures must be addressed in the project Health and Safety Plan (HASP). This plan must be approved by the project health and safety officer before work commences, must be distributed to all personnel performing sampling, and must be adhered to as field activities are performed.

6. EXCAVATION AND SAMPLE COLLECTION EQUIPMENT AND PROCEDURES

The tables in this section provide a description of equipment and sampling procedures for different soil sampling methods. The project work plan will specify the method and any site-specific requirements. Once the soil is sampled, the sample will be aliquoted in the following sequence or as stated in the project-specific planning documents: Volatile Organic Compounds (VOCs), Semi-volatile Organic Compounds (SVOCs), Pesticides, Herbicides, Polychlorinated Biphenyls (PCBs), and Metals.

Soil sampling equipment and supplies can include the following:

- Maps/plot plan
- Safety equipment, as specified in the site-specific HASP (PPE, etc.)
- Survey equipment or global positioning system (GPS) to locate sampling points
- Tape measure
- Survey stakes or flags
- Camera (extra batteries, extra memory chip)
- Stainless steel, plastic, or other appropriate homogenization bucket, bowl or pan - tools plated with chrome or other materials should not be used
- Sample containers
- Ziploc plastic bags
- Logbook
- Labels
- Chain-of-Custody records and custody seals
- Field data sheets and sample labels
- Cooler(s) with thermometer for checking temperature
- Ice
- Vermiculite
- Decontamination supplies/equipment
- Canvas or plastic sheet
- Spade or shovel
- Spatula
- Scoop
- Plastic or stainless steel spoons
- Trowel(s)
- Continuous flight (screw) auger
- Bucket auger
- Post hole auger

- Extension rods
- T-handle
- Portable electronic scale
- Sampling trier
- Thin wall tube sampler
- Split spoons
- Vehimeyer soil sampler outfit
- Tubes
- Points
- Drive head
- Drop hammer
- Puller jack and grip
- Backhoe

Soil VOC samples for site investigations conducted under the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) or the Resource Conservation and Recovery Act (RCRA), as well as some other state or federal regulatory programs, may require that collection and analysis in accordance with USEPA Method 5035. This method specifies the use of a special sampling device. USEPA instituted this type of sampling based upon studies that indicated this purge and trap method of sampling and analysis more accurately identified the types and concentrations of VOCs in soils than prior methods. USEPA and some states determined that prior methods introduced error based upon evaporative loss of VOCs that Method 5035 is believed to be eliminated.

When implementing Method 5035 sampling and analysis, an Encore™® or Terracore™® (or equivalent and EPA-approved) sampling device is required. The samplers are attached to a T-handle that serves to assist in pushing the sampler into the soil. Three (3) samplers are usually required to be used to complete a single sample; this provides the laboratory one sample for high contaminant analysis and two samples for low level analysis. The samples are either preserved by freezing upon receipt at the laboratory, or preserved within 48 hours with the appropriate preservative (methanol or sodium bisulfate). Please note that some regulatory programs do not accept freezing of samples as an adequate means of preservation. If freezing is not allowed, sample preparation in the laboratory must be performed within 48 hours of sample collection; therefore, advance coordination with the analytical laboratory regarding sample collection, delivery and preparation is critical to a successful sampling and analysis program.

The sampler must review the manufacturer's instructions that accompany the sampling device prior to use, and must be trained by KEMRON personnel experienced in use of the sampling device prior to conduct of sampling.

Additionally, hard clays and gravels may not be able to be sampled with Encore™® or Terracore™® samplers; the Project Manager should assess the site geology to determine feasibility of sampling protocol. The Project Manager also should be aware that EnCore samplers typically have a higher associated cost than Terracores.

Use of Terracore Samplers with Pre-prepared Vials:

An undisturbed soil sample is obtained by pushing the barrel of the coring tool into a freshly exposed surface and removing the corer once filed. An approximate five (5) gram weight sample is obtained for each Terracore™®, with sample weights determined in the field using a portable electronic scale. The sample is extruded into laboratory prepared, pre-tared and labeled 40 ml vials with a stir bar. The laboratory adds the necessary preservative upon log-in. A separate sample must be collected for soil moisture content measurement, using a wide-mouth glass jar; size of the sample is not critical.

Use of EnCore™® Samplers:

The EnCore™® sampler serves as the sample shipping container. An undisturbed sample is obtained by pushing the barrel of the coring tool into the freshly exposed surface and removing the corer once filled. The exterior of the barrel is quickly wiped with a clean disposable towel to ensure a tight seal. An airtight cap is then attached to create a complete sample package. The sample is labeled and inserted into a sealable (e.g., zip lock) pouch, immediately cooled to 4 ± 2°C, and prepared for shipment to the analytical laboratory.

TABLE 1: SOIL SAMPLING WITH SPOONS, SHOVELS, POST-HOLE DIGGERS, BOWLS

Item	Description
Soil Level Identification:	Surface Soils
Soil Depth:	Based on project specific requirements, typically in range of 0 to 12" bgs
Equipment:	Shovel, post-hole digger, stainless steel spoon, stainless steel bowl
Excavation Procedure:	Use the spoon, shovel or post-hole digger as intended.
Sampling Procedure:	***Preparation:*** 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required. 2. Obtain necessary sampling and monitoring equipment. 3. Decontaminate or pre-clean equipment, and ensure that it is in working order. 4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate. 5. Perform a general site survey prior to site entry in accordance with the site specific HASP. 6. Use stakes, flagging, or cones to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. Underground utility clearance should be performed in accordance with KEMRON's utility clearance SOP 12-UTL-00, and utility clearance should always be confirmed before beginning work. ***Sampling:*** 1. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade. 2. Using a pre-cleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade. 3. If VOC analysis is to be performed via Method 5035 (Encore or equivalent) sampling, the sample is to be collected directly from the soil. Otherwise, transfer the sample directly into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval or location into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly. 4. Place samples in cooler and cool to 4° Celsius (C) with wet ice. 5. Record sample observations in field log book. ***Post-Sampling Activities:*** Several activities need to be completed and documented once soil sampling has been completed. These activities include, but are not limited to: • Ensuring that all field equipment has been decontaminated and returned to

	proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible. • Processing all sample paperwork, including copies provided to laboratory. • Compiling all field data for site records. • Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

TABLE 2: HAND-AUGER OR THIN-WALL TUBE SAMPLER

Item	Description
Soil Level Identification:	• Surface Soils • Shallow Subsurface Soils
Soil Depth:	• Based on project specific requirements, typically in range of 0 to 12" bgs • 12 inches bgs to the depth that manual methods become impractical
Equipment:	Stainless steel hand auger, stainless steel spoon, stainless steel bowl
Excavation Procedure:	Typically, four ()-inch auger buckets with cutting heads are pushed and twisted into the ground and removed as the auger bucket is filled. The auger holes are advanced one bucket at a time. When the depth of a sampling interval has been reached, one auger bucket is used to advance the auger hole to the first desired sampling depth.
Sampling Procedure	***Preparation:*** 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required. 2. Obtain necessary sampling and monitoring equipment. 3. Decontaminate or pre-clean equipment, and ensure that it is in working order. 4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate. 5. Perform a general site survey prior to site entry in accordance with the site specific HASP. 6. Use stakes, flagging, or cones to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. Underground utility clearance should be performed in accordance with KEMRON's utility clearance SOP 12-UTL-00, and utility clearance should always be confirmed before beginning work. ***Sampling:*** This system consists of an auger, or a thin-wall tube sampler, a series of extensions, and a "T" handle (Figure 1, Appendix A). The auger is used to bore a hole to a desired sampling depth and is then withdrawn. The sample may be collected directly from the auger. If a core sample is to be collected, the auger tip is then replaced with a thin wall tube sampler. The system is then lowered down the borehole, and driven into the soil to the completion depth. The system is withdrawn and the core is collected from the thin wall tube sampler. Several types of augers are available; these include: bucket type, continuous flight (screw), and post-hole augers. Bucket type augers are better for direct sample recovery because they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the flights. The continuous flight augers are satisfactory when a composite of the complete soil column is desired. Post-hole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil and cannot be used below a depth of approximately three feet. The following procedure is used for collecting soil samples with the auger: 1. Attach the auger bit to a drill rod extension, and attach the "T" handle to the drill rod. 2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first three to six inches of surface soil for an

area approximately six inches in radius around the drilling location.

3. Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.
4. After reaching the desired depth, slowly and carefully remove the auger from the hole. When sampling directly from the auger, collect the sample after the auger is removed from the hole and proceed to Step 10.
5. Remove auger tip from the extension rods and replace with a pre-cleaned thin wall tube sampler. Install the proper cutting tip.
6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Do not scrape the borehole sides. Avoid hammering the rods as the vibrations may cause the boring walls to collapse.
7. Remove the tube sampler, and unscrew the drill rods.
8. Remove the cutting tip, and the core from the device.
9. Discard the top of the core (approximately 1 inch), as this possibly represents material collected before penetration of the layer of concern. Place the remaining core into the appropriate labeled sample container. Sample homogenization is not required.
10. If VOCs analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. If Method 5035 (Encore or equivalent) sampling is required, the sample is to be collected directly from the core. Otherwise, place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.
11. Place samples in cooler and cool to 4° Celsius (C) with wet ice.
12. If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.
13. Abandon the hole according to applicable state regulations. Generally, shallow holes can simply be backfilled with the removed soil material.
14. Record sample observations in field log book.

Post-Sampling Activities:

Several activities need to be completed and documented once soil sampling has been completed. These activities include, but are not limited to:

- Ensuring that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible.
- Processing all sample paperwork, including copies provided to laboratory.
- Compiling all field data for site records.

	• Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

TABLE 3: SHELBY OR "PUSH TUBE"

Item	Description
Soil Level Identification:	• Surface Soils • Shallow Subsurface Soils
Soil Depth:	• Based on project specific requirements, typically in range of 0 to 12" bgs • 12 inches bgs to depth that manual methods become impractical
Excavation Procedure:	Drive the tube into the soil with a drill rig, if necessary.
Sampling Procedure:	***Preparation:*** 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required. 2. Obtain necessary sampling and monitoring equipment. 3. Decontaminate or pre-clean equipment, and ensure that it is in working order. 4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate. 5. Perform a general site survey prior to site entry in accordance with the site specific HASP. 6. Use stakes, flagging, or cones to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. Underground utility clearance should be performed in accordance with KEMRON's utility clearance SOP 12-UTL-00, and utility clearance should always be confirmed before beginning work. ***Sampling:*** 1. Drill to first sampling depth, as described in the project planning documents. 2. Place decontaminated Shelby tube sampler on center rods. 3. Drive Shelby tube sampler with the push head of the drill rig, as described in ASTM Method D1587. Retrieve the sampling tube and remove the disturbed material from the top of the tube. In addition, remove 1 inch of soil from the base of the tube. Place an impervious disk at both ends of the tube seal with a wax plug prior to shipment to the laboratory. 4. If Shelby tubes are to be extruded in the field for composite sampling, the driller will use a hydraulic extruder to obtain the sample. Samples will then be composited, as necessary, for analysis. 5. Screen sampler with PID (if required). 6. Label and manage sample containers in accordance with the site-specific SAP section for shipping and handling of samples. The sample tube should be packed in Styrofoam™ plugs or other cushioning material to prevent disturbance of the sample while en route to the geotechnical laboratory for analyses. 7. Record sample observations in field log book. ***Post-Sampling Activities:*** Several activities need to be completed and documented once soil sampling has been completed. These activities include, but are not limited to: • Ensuring that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of

	individual responsible. • Processing all sample paperwork, including copies provided to laboratory. • Compiling all field data for site records. • Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

TABLE 4: BACKHOE TRENCH OR BUCKET SAMPLING

Item	Description
Soil Level Identification:	• Surface Soils • Shallow Subsurface Soils
Soil Depth:	• Based on project specific requirements, typically in range of 0 to 12" bgs • 12 inches bgs – depth manual methods become impractical or the depth that a backhoe can reach
Excavation Procedure:	Backhoe operator is instructed what area to excavate.
Sampling Procedure:	***Preparation:*** 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required. 2. Obtain necessary sampling and monitoring equipment. 3. Decontaminate or pre-clean equipment, and ensure that it is in working order. 4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate. 5. Perform a general site survey prior to site entry in accordance with the site specific HASP. 6. Use stakes, flagging, or cones to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. Underground utility clearance should be performed in accordance with KEMRON's utility clearance SOP 12-UTL-00, and utility clearance should always be confirmed before beginning work. ***Sampling:*** A backhoe can be used to remove sections of soil when detailed examination of soil characteristics is required. This is probably the most expensive sampling method because of the relatively high cost of backhoe operation. The following procedures are used for collecting soil samples from test pits or trenches: 1. Prior to any excavation with a backhoe, it is important to ensure that all sampling locations are clear of overhead and buried utilities. 2. Review the site specific HASP and ensure that all safety precautions including appropriate monitoring equipment are installed as required. 3. Using the backhoe, excavate a trench approximately three feet wide and approximately one foot deep below the cleared sampling location. Place excavated soils on plastic sheets. Trenches greater than four feet deep must be sloped or protected by a shoring system, as required by OSHA regulations. 4. A shovel is used to remove a one to two inch layer of soil from the vertical face of the pit where sampling is to be done. 5. Samples are taken using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling to remove any soil that may have fallen from above, and to expose fresh soil for sampling. In many instances, samples can be collected directly from the backhoe bucket. KEMRON employees are not to enter trenches for the purposes of sample collection unless done so under specific health & safety requirements, work plans or procedures.

	6. If VOC analyses are required, the Encore or equivalent sampling device is to be used to collect the sample. Otherwise, transfer the sample into an appropriate, labeled sample container with a stainless steel spoon or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly. 7. Place samples in cooler and cool to 4° Celsius (C) with wet ice. 8. Abandon the pit or excavation according to applicable state regulations. Generally, shallow excavations can simply be backfilled with the removed soil material. 9. Record sample observations in field log book. ***Post-Sampling Activities:*** Several activities need to be completed and documented once soil sampling has been completed. These activities include, but are not limited to: • Ensuring that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible. • Processing all sample paperwork, including copies provided to laboratory. • Compiling all field data for site records. • Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

TABLE 5: DRILL RIGS WITH SPLIT SPOON (BARREL) SAMPLER

Item	Description
Soil Level Identification:	Deep Subsurface Soils
Soil Depth:	> 20 feet
Excavation Procedure:	***Split-Spoon – Hammer driven:*** • Drive the split-spoon sampler either inside a hollow-stem auger or inside an open borehole after rotary drilling equipment has been temporarily removed. • Record the number of blows required to drive the spoon for each six-inch interval. • Remove the spoon. ***Continuous split-spoon:*** While drilling, advance the hollow-stem auger containing the split-spoon.
Sampling Procedure:	***Preparation:*** 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required. 2. Obtain necessary sampling and monitoring equipment. 3. Decontaminate or pre-clean equipment, and ensure that it is in working order. 4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate. 5. Perform a general site survey prior to site entry in accordance with the site specific HASP. 6. Use stakes, flagging, or cones to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. Underground utility clearance should be performed in accordance with KEMRON's utility clearance SOP 12-UTL-00, and utility clearance should always be confirmed before beginning work. ***Sampling:*** Split spoon sampling is generally used to collect undisturbed soil cores of 18 or 24inches in length. A series of consecutive cores may be extracted with a split spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augered hole and the core extracted. When split spoon sampling is performed to gain geologic information, all work should be performed in accordance with ASTM D1586-98, "Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils". The following procedures are used for collecting soil samples with a split spoon: 1. Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the head piece on top. 2. Place the sampler in a perpendicular position on the sample material. 3. Using a well ring, drive the tube. Do not drive past the bottom of the head piece or compression of the sample will result. 4. Record in the site log book or on field data sheets the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth. 5. Withdraw the sampler, and open by unscrewing the bit and head and splitting

the barrel. The amount of recovery and soil type should be recorded on the boring log. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is typically available in 2 and 3 ½ inch diameters. A larger barrel may be necessary to obtain the required sample volume.

6. Without disturbing the core, use the Encore or equivalent to collect the soil sample for VOC analysis. Otherwise, transfer portion for VOC analyses to appropriate labeled sample container(s) and seal tightly.
7. Place the remaining soil into a stainless steel bowl, homogenize, and transfer portions for remaining analyses to appropriate sample container(s) and seal tightly.
8. Place in cooler and cool to 4° Celsius (C) with wet ice.
9. If another sample is to be collected in the same hole, but at a greater depth, reattach a (decontaminated / clean) split spoon sampler to the drill rod, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.
10. Abandon the hole according to applicable state regulations or install monitoring well if applicable in accordance with the site work plan and KEMRON SOP 12-MWC-00.

Post-Sampling Activities:

Several activities need to be completed and documented once soil sampling has been completed. These activities include, but are not limited to:

- Ensuring that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible.
- Processing all sample paperwork, including copies provided to laboratory.
- Compiling all field data for site records.
- Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

7. FIELD RECORDS

7.1 Field Logbook

See the Field Documentation SOP, 07-DOC-00, for generic documentation items. Examples of specific soil sampling documentation include the following:

a. Site Name;
b. Date and time of arrival;
c. Weather conditions, including ambient air temperature and/or miscellaneous observations;
d. Location;
e. Date and time sampling;
f. Sample location and boring ID;
g. Sample depth;
h. Sample sequence number;
i. Sample Equipment ID;
j. Method of sampling;
k. Field parameters (pH, conductivity, and temperature);
l. Fractions sampled and preservation method(s);
m. Signature of Sampler and Date;
n. Physical characteristics, to include the general soil type (sand, silt, clay, and organic/other matter such as wood chips, as determined using the Unified Soil Classification System (USCS)), approximate grain size (fine, medium, coarse), presence of observable biota, odor, color, layers, depth of layer changes;
o. Pertinent field observations and comments;
p. Site drawing showing sample locations, dimensions, and prominent features.

7.2 Chain-of-Custody

Completed chain-of-custody (COC) forms must be maintained for all samples. Sample labels should be checked against the completed COC for accuracy. An example COC form is included as Attachment A. The original form will be sent to the laboratory with the samples and signed by the lab upon receipt. Original completed COC forms will be returned to KEMRON with the analytical report. A copy of the COC will be retained by the field crew (sampler) and kept with field records.

8. DECONTAMINATION

All reusable equipment taken to the field will be clean. The following procedures, or that specified in the site-specific documents, are completed during a sampling event for multiple uses of equipment, at the conclusion of equipment use at the site and if visual evidence indicates a need at the onset of equipment use.

1. Hand wash, or flush/pump through automatic samplers/equipment, with a non-phosphate laboratory detergent dissolved in tap water;

2. Rinse with tap water;
3. Rinse thoroughly with deionized water;
4. Allow to air dry;
5. Wrap in aluminum foil and a plastic bag, if needed, and store in a clean, dry area.

Decontamination solvent such as isopropanol, acetone or hexane may be used for equipment exposed to high levels of organics (e.g., NAPL). Use of specific decontamination solvents should be addressed in project-specific planning documents and the waste solvents managed and disposed in accordance with applicable regulations.

9. SAMPLE SHIPMENT

Equipment decontamination chemicals may need to be shipped to the site and samples may need to be shipped to the laboratory. All shipping is completed in accordance with the current federal and international regulations (DOT, EPA, and IATA). Shipping stipulations are to be included in the project documents, but should you have any questions regarding shipping of any materials to and from the site, contact the authorized shipping representative in your home office.

10. ASSOCIATED SOPs

Field Log Book 07-DOC-00

Well Installation 12-MWC-00

Utility Clearance 12-UTL-00

11. REFERENCES

ASTM D 1586-98, “Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils”

USEPA Environmental Response Team, Feb 2000. Standard Operating Procedure (SOP) #2012, Soil Sampling.

USEPA, July 2002. Draft Revision 1, Method 5035A, Appendix A, The Collection and Preservation of Aqueous and Solid Samples for Volatile Organic Compound (VOC) Analysis.

ATTACHMENT A –EXAMPLE CHAIN OF CUSTODY FORM

KEMRON
ENVIRONMENTAL SERVICES

COC No. A 39472

109 Starlite Park
Marietta, OH 45750

Phone: 740-373-4071
Fax: 740-373-4835

CHAIN-OF-CUSTODY RECORD

Company Name:
Project Contact:
Contact Phone #:
Turn Around Requirements:
Location:
Project #:
Project Name:
Sampler (print):
Signature:

Program: ☐ NPDES ☐ AFCEE ☐ RCRA ☐ USACE ☐ Other

Sample I.D. No.	Comp.*	Grab	Date	Time	Protocol: CWA	Protocol: SW846	NUMBER OF CONTAINERS: Hcl	ADDITIONAL REQUIREMENTS

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Relinquished by: (Signature)	Date	Time	Received for Laboratory by: (Signature)	Date	Time	Cooler Temp in °C	Remarks:	Received by: (Signature)

*Homogenize all composite samples prior to analysis

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STANDARD OPERATING PROCEDURE FOR GROUNDWATER MONITORING WELL INSTALLATION AND DEVELOPMENT SITE SPECIFIC TO AK STEEL MIDDLETOWN WORKS

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Revision 00

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Approved by:

Lou Ehrhard, Corporate Quality Assurance Manager — 04/10/2013 Date

John Dwyer, Executive Vice President — 04/10/2013 Date

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A T T A C H M E N T S

1 **Technical Guidance for Hydrogeological Investigations and Groundwater Monitoring.**
Chapter 7 – Monitoring Well Design and Installation

2 **Well Development Log**

P R O C E D U R E S

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the methods to be followed to install a groundwater monitoring well.

1.2 The purpose of this procedure is to establish a uniform method to install groundwater monitoring wells, to facilitate quality control in field operations, and to facilitate uniformity among different field teams.

2.0 SUMMARY OF METHOD

2.1 A borehole is advanced to the desired depth interval using various drilling techniques. *NOTE: No monitoring well will be installed using mud rotary techniques.*

2.2 The well screen and casing materials are lowered to the desired depth interval within the borehole and a filter pack is installed in the annular space surrounding the well screen. Sealing materials, such as bentonite chips, pellets, or grout, are installed in the annular space above the top of the filter pack.

2.3 An expandable locking cap and protective cover are installed over the well casing to ensure security.

2.4 The well is developed to remove drilling fluids, drilling-induced formation smear from the borehole walls, and sediment that may have accumulated during well installation; to consolidate the filter pack around the well screen; and to enhance the hydraulic connection between the formation target zone and the well.

2.5 The horizontal coordinates, ground surface elevation, and top-of-well casing are measured by a state-licensed land surveyor.

3.0 DEFINITIONS

3.1 DMP – Data Management Plan

3.2 GIS – Geographic Information System

3.3 HASP – Health and Safety Plan

3.4 HAZWOPER – Hazardous Waste Operations and Emergency Response

3.5 NTU – nephelometric turbidity unit

3.6 OSHA – Occupational Health and Safety Association

3.7 PPE – personal protective equipment

3.8 PVC – polyvinyl chloride

3.9 QAPP – Quality Assurance Project Plan

3.10 SAP – Sampling and Analysis Plan

3.11 SOP – standard operating procedure

4.0 HEALTH AND SAFETY

4.1 Review the site-specific HASP prior to conducting any procedures described in this SOP.

4.2 As dictated by the site-specific HASP, wear appropriate PPE when working with potentially hazardous materials and/or potentially hazardous environments.

5.0 PERSONNEL REQUIREMENTS

5.1 All involved personnel must be 40-hour OSHA HAZWOPER certified (29 CFR 1910.120).

5.2 All involved personnel must be in compliance with the Health and Safety Plan.

5.3 All involved personnel must be in compliance with any site-specific requirements.

6.0 POTENTIAL EQUIPMENT AND SUPPLIES

- Well casing: 2-inch or 4-inch diameter, flush-threaded, Schedule 40 PVC casing
- Well screen: 2-inch or 4-inch diameter, flush-threaded, Schedule 40 PVC screen with factory-milled 0.010-inch slots
- Flush-threaded bottom cap
- Surface completion cap
- Well-graded, clean sand filter material
- Bentonite
- Tremie-pipe
- Low-flow pump or new disposable Teflon® or PVC bailer
- Electronic water level meter
- Surge block
- Locks
- Steel well cover
- Wireline
- Turbidity meter
- Water quality instruments: temperature, conductivity, and pH
- Weighted tape measure

- Waterproof pens/markers
- Well Development Log
- Field notebook
- Alconox® (or similar) detergent
- Distilled water
- Laboratory grade, analyte-free water
- Appropriate PPE

7.0 PRE-WELL INSTALLATION METHODS

NOTE: Figure 7-1 of Attachment 1 presents a schematic of the construction of a typical monitoring well.

7.1 If required, acquire groundwater observation/monitoring well permits from the appropriate agency(s) before drilling is initiated.

7.2 Conduct an underground utility check before drilling begins.

7.3 Advance the borehole the total depth specified in the SAP or work plan.

7.4 Evaluate the stratigraphic log to verify that the boring depth is adequate for the anticipated design of the monitoring well. A schematic of a typical monitoring well

7.5 Verify the total depth of the borehole using a weighted tape measure.

7.6 Determine screen slot size and filter pack size based on state guidance (see Attachment 1).

8.0 WELL INSTALLATION METHODS

NOTE: Monitoring wells shall be constructed using new, factory packaged, two-inch diameter Schedule 40 well screen and well casing. All well materials that are not contained within factory packaging shall be steam cleaned prior to installation. All workers handling the well screen shall wear a new pair of cotton or synthetic gloves during each well installation. Each well will be constructed such that a slotted casing fitted with a threaded end cap extends from the bottom of the boring to approximately five feet above the static water level observed during drilling of the boring, or to base of the overlying clay or silt confining unit, whichever is deeper. No PVC cement or other solvents shall be used to fasten the well casing joints, well screen joints, or end caps. All leftover or unused drilling fluids, seal compounds, or neat cement shall be properly disposed. If SAP or work plan-specific well design requirements vary from this SOP, the SAP or work plan requirements shall prevail.

8.1 Measure well casing materials to the nearest 0.1 foot before being lowered into the borehole.

8.2 Lower the appropriate lengths of slotted casing and blank casing into the borehole. If necessary, a wireline may be used to hang the well casing in the borehole while completing the well installation.

8.3 Once the well has been situated at the desired depth, secure well surface cap onto top of casing to prevent entry of sand or grout. Then, place an artificial filter pack consisting of clean, chemically inert, appropriately-sized silica sand to fill the annular space surrounding the well screen to approximately 2 to 3 feet above the top of the well screen. The anticipated volume of filter pack material required to complete the installation shall be calculated prior to installation. Install the filter pack using gravity emplacement for shallow monitoring wells or a tremie pipe for deeper monitoring wells. *NOTE: No well filter pack shall extend into any water bearing zone other than the zone to be monitored. All attempts shall be made to allow for a sufficient filter pack without the threat of interconnecting zones.*

8.4 The installation method will be chosen by the field engineer. If using hollow-stem augers, install the filter pack material as the augers are pulled back in one to two foot increments to reduce caving effects and to ensure proper placement. At regular intervals, measure the depth to the top of the filter pack using a tremie pipe or a weighted tape. At the discretion of the field engineer, a surge block may be repeatedly raised and lowered through the water column to promote settlement of the filter pack firmly around the screen interval of the well. Additional filter pack material will be installed as necessary such that the top of the filter pack is approximately 2 to 3 feet above the top of the well screen. In the field notebook, explain any discrepancy between the actual and calculated volumes of filter pack material used.

8.5 If conditions allow, place a bentonite seal consisting of bentonite chips, medium grade bentonite pellets, or bentonite slurry to fill the annular space to a level of about 2 to 3 feet above the top of the filter pack. Place this material in approximately 6-inch lifts by slowly hand pouring the pellets or chips into the annular space at the top of the well. Measure the depth to the top of the bentonite seal regularly using a tremie pipe or a weighted tape measure. These devises will also be used to tamp the sealing materials into place and to prevent bridging. If the bentonite seal is being constructed above the water level, hydrate the bentonite pellets/chips using approximately 1 gallon of tap water per lift.

8.6 If the depth of the water column above the filter pack prevents the installation of a bentonite seal, place a two-foot thick transition zone consisting of fine-grained sand on top of the filter pack prior to installing a thick bentonite slurry using a grout pump and tremie pipe.

8.7 Verify the depth to the top of the bentonite seal using the tremie pipe or a weighted tape measure.

8.8 Install a neat cement/bentonite grout or bentonite grout from the top of the bentonite seal to the ground surface using a tremie pipe. Grout/additive/water mixtures will be determined on a site-specific basis. The typical neat cement/grout mixture consists of a mixture of one sack (94 pounds) of Portland Type I/II cement, approximately 2 to 5 percent by weight (of cement) powdered bentonite, and approximately 6 to 8 gallons of water. Only potable water will be used to prepare the grout. Augers or surface casings (if used), will be slowly removed as the slurry is introduced into the borehole. The well shall not be disturbed for a minimum of 48 hours to permit the grout to gain sufficient strength. *NOTE: Periodically, check the annular space to ensure that no settling of the grout has occurred. Add additional grout as necessary prior to well completion.*

8.9 Fit well with a stick-up or traffic-rated flush mount well cover, depending on site activities occurring around the well location. Construct a concrete pad around the well monument sloping slightly away from the monument to promote proper drainage. Three protective "bumper" posts may be installed around stick-up covers at the discretion of the field engineer.

9.0 WELL DEVELOPMENT METHODS

NOTE: All information relating to well development will be recorded in a Well Development Log (Attachment 2). See Section 12 for disposal of well development fluids.

9.1 Develop each well as soon as practical, but no sooner than 24 hours after placing the annular seal.

9.2 Prior to commencing development, measure the depth to water and total well depth using an electronic water level meter.

9.3 Calculate the amount of water in one well volume by multiplying the water column length in feet by 0.163.

9.4 Remove at least three well volumes of water during development.

9.5 Use a bailer or submersible pump to remove sediment and turbid water from the bottom of the well. *NOTE: High capacity submersible pumps are preferred. Bailers should be used only as a last resort or when yields are low.*

9.6 Use a surge block within the entire screened interval to flush the filter pack of fine sediment and formation smear from the borehole walls. Conduct surging slowly to reduce disruption to the filter pack and screen.

9.7 Following surging, use a bailer or submersible pump to remove sediment and turbid water from the well.

9.8 Repeat steps 9.5 through 9.7 until relatively sediment-free water is obtained from the well.

9.9 During development, monitor and record groundwater parameters including temperature, pH, conductivity, and turbidity, as well as drawdown in the wells. In addition, periodically record development method, flow rate, water clarity, odor, water levels, recovery rates, and quantity of water evacuated. Each well will be developed until the following criteria are met within three consecutive well volumes: pH ± 0.1 pH units, specific conductance ± 3%; temperature ± 3°C; turbidity ± 10% if a turbidity is greater than 10 NTU; oxidation reduction potential ± 10 millivolts; and dissolved oxygen ± 0.3 milligrams per liter. If the screened interval is too long to be developed adequately in one stage, multiple stages will be employed, in which the end of the pump intake will be raised or lowered to various depths, as required. Final groundwater parameters will be recorded in the field notebook. *NOTE: The pH is checked primarily to ensure there is no grout contamination. The majority of the groundwater falls in a pH range of between 7 and 9. If the pH of the purged water at the end of development is above 9.5, the condition of the well should be evaluated for grout invasion. This is especially apparent if the pH is higher at the start of development and slowly declines as more water is removed from the well, but rises to higher levels upon ceasing the purging process. Further development may need to be performed until a lower pH is obtained. If additional development measures do not lower the pH, an investigation will take place to determine subsequent actions.*

9.10 The well can be further developed using air-lift or pumping methods. Use a bailer for low-yield wells. If possible, the well will be developed at a higher pumping rate than the anticipated rate of future purging. Development will proceed until the well meets development criteria specified in Section 9.9.

9.11 Following development, collect one round of groundwater samples from each of the new monitoring wells. *NOTE: The initial round of groundwater sampling should not begin until the static water level has fully recovered in the well, and a minimum of 3 days has passed. If this cannot be achieved, then justification should be provided.*

10.0 SURVEYING METHODS

NOTE: Traditionally, the North American Datum 1983, State Plane Coordinate System shall be utilized by surveyors and within GIS. Refer to the site-specific DMP, SAP and/or work plan to determine if an alternate coordinate system is to be used.

10.1 Following development, the wells will be surveyed by a state-licensed land surveyor.

10.2 The surveyor will measure and record horizontal coordinates of each well, as well as vertical elevations for well casings, well boxes, and ground surface. Survey coordinates will be made in reference to a nearby benchmark of known mean sea level elevation.

11.0 DECONTAMINATION METHODS

11.1 Follow decontamination methods described in the site-specific QAPP.

12.0 WASTE MANAGEMENT PROCEDURES

12.1 Follow waste management procedures, as described in the SAP or work plan, for all well installation- and development-derived fluids collected during this SOP that are remaining following the completion of well installation and development activities.

12.2 Note the disposal of any fluids within the field notebook.

13.0 DOCUMENTATION AND RECORD MANAGEMENT

13.1 A well construction diagram for each well will be completed in the field on the Well Development Log by the field geologist/engineer and submitted to the reviewing geologist or engineer upon completion of each well.

13.2 Well installation and construction data will be summarized in the field notebook.

13.3 Well development notes and field measurements of water quality parameters will be summarized in the field notebook and in the Well Development Log.

13.4 Following review by the project manager, the original records will be kept in the project file.

14.0 QUALITY ASSURANCE/QUALITY CONTROL

14.1 Review all field data records for accuracy and completeness.

14.2 In order to ensure that decontamination procedures are effective, equipment rinseate blanks may be collected, as dictated by the SAP or site-specific QAPP.

14.3 In order to ensure that sampling methods are effective and representative, field duplicates may be collected, as dictated by the SAP or site-specific QAPP.

15.0 REFERENCES

15.1 Ohio Environmental Protection Agency, *Technical Manual for Hydrogeologic Investigations and Ground Water Monitoring*, 1995.

TECHNICAL GUIDANCE MANUAL FOR HYDROGEOLOGIC INVESTIGATIONS AND GROUND WATER MONITORING

CHAPTER 7
MONITORING WELL DESIGN AND INSTALLATION

February 1995

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CHAPTER 7

MONITORING WELL DESIGN AND INSTALLATION

In order to collect representative ground water samples, it is necessary to construct monitoring wells to gain access to the subsurface. This chapter covers installation and construction of single-riser/limited interval wells, which are designed such that only one discrete zone is monitored in a given borehole. It is important that efforts focus on intervals less than 10 feet thick and be specific to a single saturated zone.

All monitoring wells should be designed and installed in conformance with site hydrogeology, geochemistry, and contaminant(s). While it is not possible to provide specifications for every situation, it is possible to identify certain design components. Figure 7.1 is a schematic drawing of a single-riser/limited interval well. The ***casing*** provides access to the subsurface. The ***intake*** consists of a filter pack and screen. The ***screen*** allows water to enter the well and, at the same time, minimizes the entrance of filter pack materials. The ***filter pack*** is an envelope of uniform, clean, well-rounded sand or gravel that is placed between the formation and the screen. It helps to prevent sediment from entering the well. Installation of a filter pack and screen may not be necessary for wells completed in competent bedrock. The ***annular seal*** is emplaced between the borehole wall and the casing and is necessary to prevent vertical movement of ground water and infiltration of surface water and contaminants. ***Surface protection***, which includes a surface seal and protective casing, provides an additional safeguard against surface water infiltration and protects the well casing from physical damage.

DESIGN OF MULTIPLE-INTERVAL SYSTEMS

It is often necessary to sample from multiple discrete intervals at a given location if more than one potential pathway exists or a saturated zone is greater than 10 feet thick. Chapter 5 discusses the concepts involved in selecting zones to monitor. Multiple-interval monitoring can be accomplished by installing single-riser/limited interval wells in side-by-side boreholes (well clusters) or using systems that allow sampling of more than one interval from the same borehole (multi-level wells, well nests, or single-riser/flow-through wells).

WELL CLUSTERS

When monitoring multiple intervals at one location, single-riser/limited interval wells should be installed in adjacent, separate boreholes. These well clusters can be used to determine vertical gradients when distinct differences in head exist. They may be used to monitor discrete zones or evaluate chemical stratification within a thick zone. If flow direction has been determined prior to installation, the shallow well should be placed hydraulically upgradient of the deeper well to avoid the potential influence on its samples caused by the presence of grout in the annular space of the deeper well.

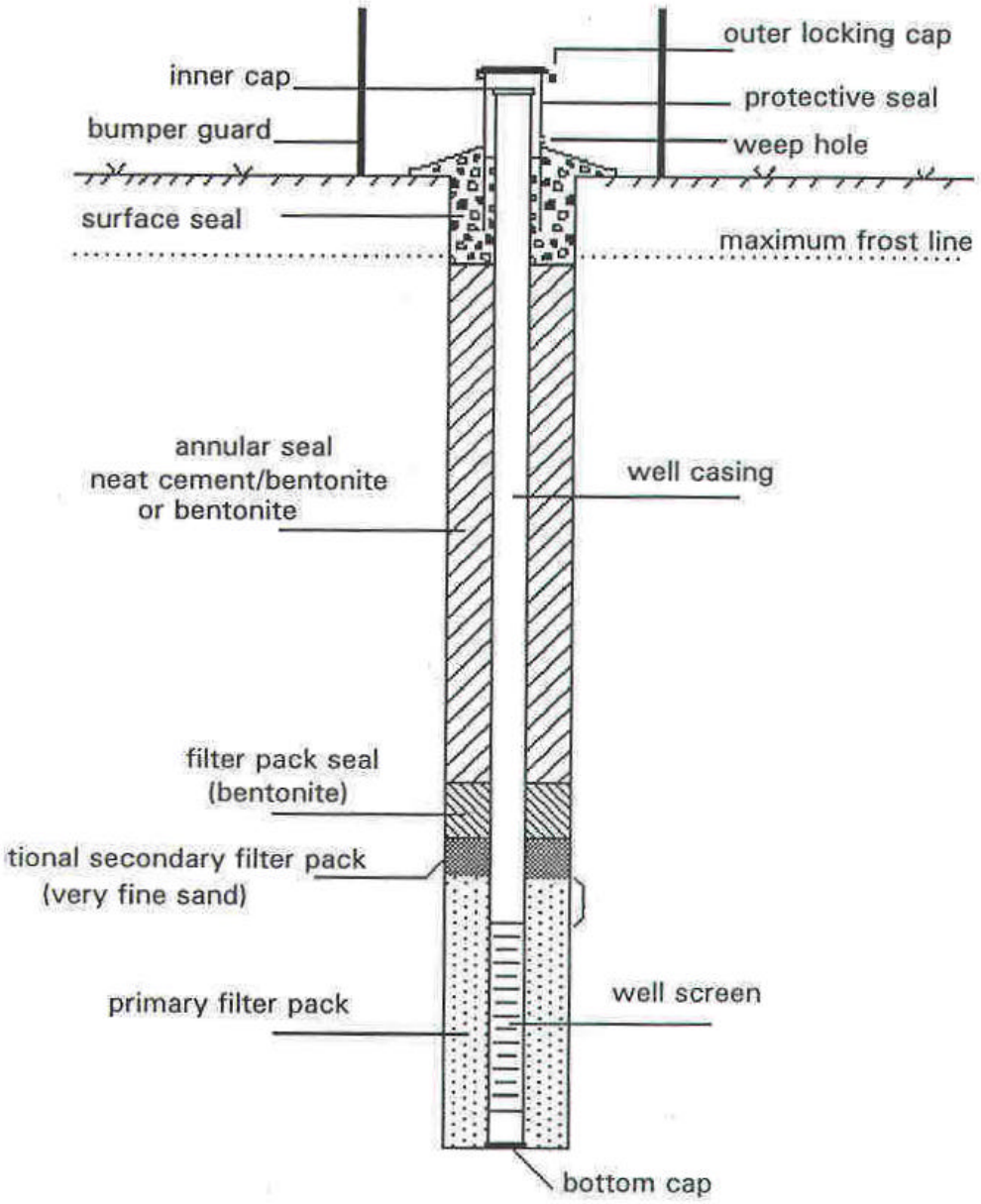

Figure 7.1 Cross-section of a typical single-riser/limited interval monitoring well.

MULTI-LEVEL WELLS

Multi-level wells allow sampling of more than one interval in a single borehole. Individual tubes run from sampling levels to the surface. These levels are isolated within the well either by packers or grout. Probes, lowered into the casing, can locate, isolate and open a valve into a port coupling to measure the fluid pressure outside the coupling or obtain a sample.

The use of multiple-level monitoring wells in Ohio has been limited due to: 1) cost of installation, 2) difficulty in repairing clogs, and 3) difficulty in preventing and/or evaluating sealant and packer leakage. Detailed workplans (including construction and installation, methods to measure water levels and obtain samples, references to situations where these types of wells have been used successfully, and advantages and disadvantages) should be submitted prior to installing multi-level systems. Until more site-specific data is available concerning their performance, multi-level wells should only be considered when a single zone having no to little vertical flow is being monitored at different depths.

NESTED WELLS

Nested wells involve the completion of a series of single-riser wells in a borehole. Each well is screened to monitor a specific zone, with filter packs and seals employed to isolate the zones. Nested wells are not recommended because they are difficult to install in a manner that ensures that all screens, filter packs, and seals are properly placed and functioning. It is more efficient to install single-riser wells for each interval to ensure that representative samples can be collected. Aller et al. (1991) indicated that individual completions generally are more economical at depths less than 80 feet. According to Dalton et al. (1991), the cost of installing well clusters is usually only marginally higher than the cost for nested wells. Well clusters can enable savings on sampling and future legal costs that may be necessary to prove the accuracy of nested wells.

SINGLE RISER/FLOW-THROUGH WELLS

Single riser/flow-through wells are monitoring wells that, in general, are screened across the entire thickness of a water-bearing zone. These wells are typically small in diameter and provide a "transparent" cross-section of the flow field (Aller et al., 1991). If purging is performed immediately before sampling, only composite water samples are yielded, which are not adequate for most monitoring studies. If natural, flow-through conditions can be maintained, and if a sampling device can be lowered with minimal disturbance of the water column, vertical water quality profiles potentially can be identified. To achieve and document the collection of such samples is very difficult, however, and the resulting data may be questioned. Furthermore, these wells are conducive to allowing cross-contamination between different zones and, therefore, should not be used in contaminated areas. Flow-through wells are not recommended.

CASING

The purpose of casing is to provide access to the subsurface for sampling of ground water and measurement of water levels. A variety of casing has been developed. Items that must be considered during well design include casing type, coupling mechanism, diameter, and installation.

CASING TYPES

There are three categories of casing commonly used for ground water monitoring, including fluoropolymers, metallics, and thermoplastics (Aller et al., 1991). All have distinctive characteristics that determine their appropriateness.

Fluoropolymers

Fluoropolymers are synthetic "plastics" composed of organic material. They are resistant to chemical and biological attack, oxidation, weathering, and ultraviolet (UV) radiation. They have a broad useful temperature range, a high dielectric constant, a low coefficient of friction, display anti-stick properties, and have a greater coefficient of thermal expansion than most other plastics and materials (Aller et al., 1991). A variety of fluoropolymers are marketed under various trademarks. Some manufacturers use one trade name to refer to several of their own materials, which may not always be interchangeable in service or performance (U.S. EPA, 1992). Standard properties of the various materials have been provided by Nielsen and Schalla (1991) and Aller et al. (1991).

The most common fluoropolymer used for monitoring wells is polytetrafluoroethylene (PTFE). It can withstand strong acids and organic solvents and, therefore, it is useful for environments characterized by the presence of these chemicals. It maintains a low tensile strength, which theoretically limits installation of Schedule 40 PTFE to an approximate depth of 250[1]. It is also very flexible, which makes it difficult to install with the retention of straightness that is needed to ensure successful insertion of sampling or measurement devices. Dablow et al. (1988) found that the ductile nature of PTFE can result in the partial closing of screen slots due to the compressive forces of the casing weight. This makes slot size selection very difficult. The inert nature of PTFE often prevents the annular seal from bonding with the casing completely, which can allow infiltration of surface water. PTFE is costly, generally ten times more expensive than thermoplastics.

Metallics

Metallic materials include low carbon, carbon, galvanized, and stainless steel. Metallics are very strong and rigid and can be used to virtually unlimited depths. Corrosion problems are the major disadvantage for low carbon, carbon, and galvanized casings. Electrochemical and chemical attack alters water sample quality. U.S.EPA (1992) has listed the following as indicators of corrosive conditions (modified from Driscoll, 1986):

- Low pH (< 7.0).
- Dissolved oxygen exceeds 2 ppm.
- Hydrogen sulfide in quantities as low as 1 ppm.
- Total dissolved solids (TDS) greater than 1000 ppm.
- Carbon dioxide exceeds 50 ppm.
- Chloride (Cl^-), bromide (Br^-), and fluoride (F^-) content together exceeds 500 ppm.

According to Barcelona et al. (1983), flushing before sampling does not minimize the bias of low carbon steel due to the inability to predict the effects of disturbed surface coatings and corrosion products accumulated at the bottom of the well. Due to their high corrosion potential, all metallics except stainless steel are unacceptable for monitoring wells.

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[1] The maximum depth for PTFE casing is dependent on site hydrogeology. If the casing largely penetrates unsaturated soils, the depth may be limited to approximately 100 feet. However, if the casing is placed mostly in water-bearing zones, then depth may be as great as 375 feet.

Stainless steel is manufactured in two common types, 304 and 316. Type 304 is composed of iron with chromium and nickel. Type 316's composition is the same as Type 304's, but includes molybdenum, which provides further resistance to sulfuric acid solutions. Stainless steel is readily available in a wide variety of diameters.

Stainless steel can perform quite well in most corrosive environments. In fact, oxygen contact develops an external layer that enhances corrosion resistance (Driscoll, 1986). Yet, under very corrosive conditions, even stainless steel can corrode and release nickel and chromium into ground water samples (Barcelona et al., 1983). Combinations and/or extremes of the factors indicating corrosive conditions generally are an indication of highly corrosive environments. For example, Parker et al. (1990) found that both 304 and 316 showed rapid rusting (<24 hrs.) when exposed to water containing chloride above 1000 mg/l. Like PTFE, stainless steel is relatively expensive in comparison with thermoplastics Nielsen and Schalla (1991) and Aller et al. (1991) provided additional information on the properties of stainless steel.

Thermoplastics

Thermoplastics are composed of large, synthetic organic molecules. The most common type used for monitoring wells is polyvinyl chloride (PVC), while a material used less often is acrylonitrile butadiene styrene (ABS). These materials are weaker, less rigid, and more temperature-sensitive than metallics. Thermoplastics are very popular due to their light weight, high strength to weight ratio, low maintenance, ease of joining, and low cost.

Common, acceptable PVC types are Schedule 40 and Schedule 80. The greater wall thickness of Schedule 80 piping enhances durability and strength, provides greater resistance to heat attack from cement, and allows construction of deeper wells. Only rigid PVC should be used for monitoring wells. Flexible PVC is composed of a high percentage of plasticizers (30 - 50%), which tend to degrade and contaminate samples (Jones and Miller, 1988). All PVC casing should meet Standard 14 of NSF International. This standard sets control levels for the amount of chemical additives to minimize leaching of contaminants (NSF International, 1988). Additional specifications have been provided by Nielsen and Schalla (1991) and Aller et al. (1991).

Drawbacks of PVC include brittleness caused by ultraviolet (UV) radiation, low tensile strength, relative buoyancy in water, and susceptibility to chemical attack. It is immune to corrosion and is resistant to most acids, oxidizing agents, salts, alkalies, oils, and fuels (NWWA/PPI, 1981). Additionally, Schmidt (1987) showed that no degradation of PVC occurred after six months immersion in common gasolines. However, studies have shown that high concentrations (parts-per-thousand or percentage concentrations) of tetrahydrafuran, methyl ethyl ketone, methyl isobutyl ketone, and cyclohexane degrade PVC (Nielsen and Schalla, 1991). Barcelona et al. (1983) reported that low molecular weight ketones, aldehydes, amines, and chlorinated alkenes and alkanes may cause degradation. There is a lack of published information regarding the concentrations of these compounds at which deterioration is significant enough to affect either the structural integrity of casing or ground water sample quality.

TYPE SELECTION

Many regulated parties choose PVC casing because of its lower cost; however, well integrity and sample representativeness are more important criteria. The high cost of analysis and the extreme precision of laboratory instruments necessitate the installation of wells that produce representative samples. Above all, the burden of proof is on the regulated party to demonstrate that casing is appropriate. The proper selection can be made by considering casing characteristics in conjunction with site conditions.

Casing characteristics include strength, chemical resistance and chemical interference potential. The ***strength*** must withstand the extensive tensile, compressive, and collapsing forces involved in maintaining an open borehole. Since the forces exerted are, in large part, related to well depth, strength often is important when planned depth exceeds the maximum range of the weakest acceptable material (100 to 375 ft. - PTFE). In these instances, either stainless steel or PVC should be chosen. Strength can be the overriding factor because the concern for chemical resistance and interference become insignificant if an open borehole cannot be maintained. Nielsen and Schalla (1991) provided specific strength data for commonly used materials.

The casing also must withstand ***electrochemical corrosion and chemical attack*** from natural ground water and any contaminant(s). Chemical resistance is most important in highly corrosive environments, when contaminants are present at extremely high levels, and when wells are intended to be part of a long-term monitoring program. For extended monitoring in corrosive environments, PTFE and PVC are preferred over stainless steel because of the potential for the metallic material to degrade. If high concentration of organics (parts per thousand) are present, either PTFE or stainless steel should be selected. U.S. EPA (1992) recommended that PVC not be used if a PVC solvent/softening agent[2] is present or the aqueous concentration of a solvent/softening agent exceeds 0.25 times its solubility in water. It is suitable in most situations where low (parts per billion to low parts per million) levels of most organic constituents are present (Nielsen and Schalla, 1991).

The casing also should not interfere with sample quality by ***adding (leaching) or removing contaminants.*** In most cases, the magnitude of this interference is a function of the ground water's contact time with the casing. The longer the contact, the greater the potential for leaching and sorption. Various studies have been conducted [Barcelona and Helfrich (1988), Curran and Tomson (1983), Gillham and O'Hannesin (1989), Jones and Miller (1988), Miller (1982), Parker and Jenkins (1986), Parker et al. (1990), Reynolds and Gillham (1985), Schmidt (1987), Sykes et al. (1986), Tomson et al. (1979), Hewitt (1992, 1994), Parker and Ranney (1994)] to compare the sorbing and leaching characteristics of the three favored materials. No conclusive results have been obtained to indicate that any one is best. Most of these studies involved contact lasting days, weeks, and even months and, therefore, the results cannot be correlated to field conditions where contact is often minimal because sampling is generally conducted soon after purging.

[2]Known PVC solvent/softening agents include: tetrahydrofuran, cyclohexane, methyl ethylketone, methyl isobutyl ketone, methylene chloride, trichloromethane, 1-1-dichloroethane, trichloroethene, benzene, acetone, and tetrachloroethene.

In many cases, concern about sorption or leaching may be exaggerated. Barcelona et al. (1983) and Reynolds and Gillham (1985) both concluded that the potential sorption biases for casing may be discounted due to the short contact after purging. Also, Parker et al. (1990) indicated that sorption of various constituents never exceeded 10 percent in the first 8 hours of their tests. They concluded that, on the basis of overall sorption potential for organic and inorganic compounds, PVC is the best compromise.

In summary, the appropriate casing should be determined on a case-by-case basis. PVC is acceptable when free product is not present and the solubility limits of organic contaminants are not approached (e.g., levels that exceed 0.25 times the solubility). Ohio EPA recognizes the difficulty inherent in establishing a "cut-off" level for when aqueous concentrations of organics cause failure of PVC. To be certain that casing will retain integrity, particularly when monitoring is planned for long periods of time (e.g., 30 years), Ohio EPA may require a more resistant casing when aqueous concentrations are relatively high but still below the criteria mentioned above.

HYBRID WELLS

Casing not in contact with the saturated zone generally is not subject to attack. Therefore, it may be possible to install less chemically resistant material above the highest seasonal water level and more inert material where ground water continually contacts the casing. Such a "hybrid well" commonly is installed for cost reduction reasons only. For example, when monitoring a zone with high concentrations of organic compounds, stainless steel or PTFE could be installed opposite the saturated materials, while PVC could be used opposite the unsaturated materials. Thus, resistant, more expensive casing would be present where contact with highly contaminated ground water may occur, while less resistant, inexpensive casing would be present where contact does not occur.

Different varieties of steel never should be installed in the same well. Each type is characterized by its own electro-chemical properties. Installation of different types in contact can increase the potential for corrosion.

COUPLING MECHANISMS

Casing sections should be connected using threaded joints that provide for uniform inner and outer diameters along the entire length of the well. Such "flush" coupling is necessary to accommodate the insertion of tools and sampling devices without obstruction and to help prevent bridging during the installation of the filter pack and annular seal. It should be noted that thread types vary between manufacturers and matching can be difficult. A union among non-matching joints should never be forced, otherwise structural integrity of the joint and the entire well could be compromised. To alleviate these problems, the American Society of Testing and Materials has developed Standard F 480-90 (1992) to create a uniformly manufactured flush-threaded joint. Most manufacturers now produce the F 480 joint, which is available in both PVC and stainless steel.

It is recommended that either nitrile, ethylene propylene, or Viton O-rings be used between sections to prevent the seal and/or affected water from entering (Nielsen and Schalla, 1991). Nielsen and Schalla (1991) indicated that Teflon tape can be used in place of O-rings, although it does not ensure as good a seal. Solvent cements should never be used because they are known to leach organics. Although welding stainless steel can produce a flush joint that is of equal or greater

strength than the casing itself, this method is not recommended due to the extra assembly time, welding difficulty, corrosion enhancement, ignition danger, and the potential to lose materials into the well (Nielsen and Schalla, 1991).

Threaded steel casing provides inexpensive, convenient connections. It should be noted that threaded joints reduce the tensile strength of the casing; however, this does not cause a problem for most shallow wells. Also, threaded joints may limit or hinder the use of various sampling devices when thin-walled stainless steel (Schedules 5 and 10) is employed. Thin-walled casing is too thin for threads to be machined, so the factory welds a short, threaded section of Schedule 40 stainless steel pipe to the end of the thin-walled pipe. These joints are made to be flush on the outside, but not the inside.

If hybrid wells are installed, it is essential that the joint threads be matched properly. This can be accomplished by purchasing casing screen that is manufactured to ASTM F480-90 (1992) standard coupling.

DIAMETER

Choice of casing diameter is also site-specific. Small wells are considered to be from 2 to 4 inches in diameter. The minimum diameter for use in monitoring wells is 2 inches. Advantages of small diameter wells are as follows:

- Water levels require less time to recover after purging.
- They produce a smaller volume of purged water that must be disposed.
- Construction costs are lower.

Some disadvantages of small diameter wells include:

- Access may be limited for sampling devices.
- Filter packs and seals are more difficult to install.
- They offer a lower depth capability due to lesser wall thickness.
- Development can be more difficult.
- Less ground water is pumped during a hydraulic test or a remediation extraction.
- The amount of available water may be too small for chemical analyses.

Further discussion of well diameter can be found in articles by Schalla and Oberlander (1983), Schmidt (1982), and Rinaldo-Lee (1983).

INSTALLATION

Ceasing should be cleaned thoroughly before installation. Strong detergents and even steam cleaning may be necessary to remove oils, cleansing solvents, lubricants, waxes, and other substances. (Curran and Tomson, 1983; Barcelona et al., 1983). It is strongly recommended that only factory-cleaned materials be used for monitoring wells. Casing can be certified by the supplier and individually wrapped in sections to retain cleanliness. If it has not been factory-cleaned and sealed, it should be washed thoroughly with a non-phosphate, laboratory grade detergent (e.g., Liquinox) and rinsed with clean water or distilled/deionized water as suggested by Curran and

Tomson (1983) and Barcelona et al. (1983). The materials should be stored in a clean, protected place to prevent contamination by drilling and site activities.

When installing casing, it is important that it remain centered in the borehole to ensure proper placement and even distribution of the filter pack and annular seal. In addition, centering helps ensure straightness for sampling device access. If a hollow-stem auger is used, no additional measures are necessary because the auger acts as a centralizing device. If casing is installed in an open borehole, centralizers made of stainless steel or PVC can be used. They are adjustable and generally attached just above the screen and at 10 to 20 foot intervals along the riser. If centralizers are used, measures should be taken to prevent them from bridging the filter pack and seal material during their installation.

INTAKES

Although every well is unique, most have a screen and filter pack. Together, these comprise an "intake" . Monitoring wells in cohesive bedrock may incorporate open borehole intakes.

FILTER PACK

Wells monitoring unconsolidated and some poorly consolidated materials typically need to have a screen (discussed later) surrounded by more hydraulically conductive material (filter pack). In essence, the filter pack increases the effective well diameter and prevents fine-grained material from entering.

Types of Filter Packs

Filter packs can be classified by two major categories, natural and artificial. ***Natural packs*** are created by allowing the formation to collapse around the screen. In general, natural packs are recommended for formations that are coarse-grained, permeable, and uniform in grain size. According to Nielsen and Schalla (1991), they may be suitable when the effective grain size (sieve size that retains 90%, or passes 10%) is greater than 0.010 inch and the uniformity coefficient (the ratio of the sieve size that retains 40% and the size that retains 90%) is greater than 3. Ideally, all fine-grained particles are removed when the well is developed, leaving the natural pack as a filter to the surrounding formation.

Installation of ***artificial packs*** involves the direct placement of coarser-grained material around the screen. The presence of this filter allows the use of a larger slot size than if the screen were placed in direct contact with the formation. Artificial packs generally are necessary where: 1) the formation is poorly sorted; 2) the intake spans several formations and/or thin, highly stratified materials with diverse grain sizes; 3) the formation is a uniform fine sand, silt or clay; 4) the formation consists of thinly-bedded materials, poorly cemented sandstones, and highly weathered, fractured, and solution-channeled bedrock; 5) shales and coals that provide a constant source of turbidity are monitored; and 6) the borehole diameter is significantly greater than the diameter of the screen (Aller et al., 1991). Artificial packs generally are used opposite unconsolidated materials when the effective grain size is less than 0.010 inches and when the uniformity coefficient is less than 3.0 (Nielsen and Schalla, 1991).

An artificial pack may include two components. The ***primary pack*** extends from the bottom of the borehole to above the top of the screen. In some cases, it may be desirable to place a ***secondary pack*** directly on top of the primary pack. Its purpose is to prevent the infiltration of the annular seal into the primary pack, which can partially or totally seal the screen.

Nature of Artificial Filter Pack Material

The artificial pack material should be well-sorted, well-rounded, clean, chemically inert, of known origin, and free of all fine-grained clays, particles and organic material. Barcelona et al. (1983) recommended clean quartz sand or glass beads. Quartz is the best natural material due to its non-reactive properties and availability. Crushed limestone should never be used because of the irregular particle size and potential chemical effects. Materials should be washed, dried, and packaged at the factory, and typically are available in 100 lb. bags (approximately one cubic foot of material) (Nielsen and Schalla, 1991).

Selection of material should be based on the formation particle size. If chosen grains are too small, it is possible that loss of the pack to the formation can occur (Nielsen and Schalla, 1991), which could lead to the settling of the annular seal into the screened interval. On the other hand, if the grains are too large, the pack will not effectively filter fine-grained material, leading to excessively turbid samples.

The primary pack generally should range in grain size from a medium sand to a cobbled gravel. Most materials are available in ranges, such as 20- to 40-mesh (0.033 to 0.016 inches, Table 7.1). The grain size of the primary filter pack should be determined by multiplying the 70% retention size of the formation by a factor of 3 to 6 (U.S. EPA, 1975). A factor of 3 is used for fine, uniform formations; a factor of 6 is used for coarse, non-uniform formations (Figure 7.2). In situations where the material is less uniform and the uniformity coefficient ranges from 6 to 10, it may be necessary to use the 90% retention (10% passing) size multiplied by 6 (Nielsen and Schalla, 1991). This is to ensure that the bulk of the formation will be retained. The ratio of the particle size to the formation grain size should not exceed 6, otherwise, the pack will become clogged with fine-grained material from the formation (Lehr et al., 1988). If the ratio is less than 4, a smaller screen slot size will be necessary, full development of the well may not be possible, and well yield may be inhibited. When monitoring in very heterogeneous, layered stratigraphy, a type of pack should be chosen that suits the layer with the smallest grain size.

It is preferred that the filter pack be of uniform grain size. Ideally, the uniformity coefficient should be as close to 1.0 as possible and should not exceed 2.5 (Nielsen and Schalla, 1991, ASTM D5092-90, 1994). Uniform material is much easier to install. If non-uniform material is used, differing fall velocities cause the materials to grade from coarse to fine upwards along the screen. This can result in the loss of the upper fine-grained portion to the well during development.

The secondary filter pack material should consist of a 90% retention sieve size (10% passing) that is larger than the voids of the primary pack to prevent the secondary pack from entering the primary pack (Nielsen and Schalla, 1991). In general, the secondary 90% retention size should be one-third to one-fifth of the primary 90% retention size (Nielsen and Schalla, 1991).

Table 7.1 Common filter pack characteristics for typical screen slot sizes (From Nielsen and Schalla, 1991).

Size of Screen Opening [mm (in.)]	Slot No.	Sand Pack Mesh Size	1% Passing Size (D_1) (mm)	Effective Size (D_{10}) (mm)	30% Passing Size (D_{30}) (mm)	Range of Uniformity Coefficient	Roundness (Powers Scale)	Fall Velocities[a] (cm/s)
0.125(0.005)	5	40-140	0.09-0.12	0.14-0.17	0.17-0.21	1.3-2.0	2-5	6-3
0.25 (0/010)	10	20-40	0.25-0.35	0.4-0.5	0.5-0.6	1.1-1.6	3-5	6-6
0.50 (0.020)	20	10-20	0.7-0.9	1.0-1.2	1.2-1.5	1.1-1.6	3-6	14-9
0.75 (0.030)	30	10-20	0.7-0.9	1.0-1.2	1.2-1.5	1.1-1.6	3-6	14-9
1.0 (0.040)	40	8-12	1.2-1.4	1.6-1.8	1.7-2.0	1.1-1.6	4-6	16-13
1.5 (0.060)	60	6-9	1.5-1.8	2.3-2.8	2.5-3.0	1.1-1.7	4-6	18-15
2.0 (0.080)	80	4-8	2.0-2.4	2.4-3.0	2.6-3.1	1.1-1.7	4-6	22-16

[a] Fall velocities in centimeters per second are approximate for the range of sand pack mesh sizes named in this table . If water in the annular space is very turbid, fall velocities may be less than half the values shown here. If a viscous drilling mud remains in the annulus, fine particles may require hours to settle.

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Figure 7.2 Artificial filter pack design criteria (Source: Design and Installation of Ground Water Monitoring Wells by D.M. Nielsen and R. Schalla, *Practical Handbook of Ground Water Monitoring*, edited by David M. Nielsen, Copyright © 1991 by Lewis Publishers, an imprint of CRC Press, Boca Raton, Florida. With permission.)

Dimension of Artificial Filter Pack

The distance between the casing and the borehole wall should be at least 2 to 4 inches to allow for proper placement of the filter pack and annular seal. Therefore, the filter pack thickness should be 2 to 4 inches. It is important that the thickness not be excessive, otherwise the potential for effective development is reduced.

The primary pack should extend from the bottom of the screen to at least 2 feet above its top. In deeper wells (i.e., >200 feet), the pack may not compress initially. Compression may occur after installation of the annular seal, which may allow the seal to be in close contact with the screen. Therefore, additional pack material may be needed to account for settling and, at the same time, provide adequate separation of the seal and the screen. However, extension of the pack should not be excessive because it enlarges the zone that contributes ground water to the well, which may cause excess dilution. The length of the secondary pack should be one-foot or less.

Artificial Filter Pack Installation

Methods that have been used for artificial pack installation include tremie pipe, gravity emplacement, reverse circulation, and backwashing (Nielsen and Schalla, 1991). The material must be placed in a manner that prevents bridging and particle segregation. Bridging can cause the presence of large voids and may prevent material from reaching the intended depth. Segregation can cause a well to produce turbid samples. During installation, regular measurements with a weighted tape should be conducted to determine when the desired height has been reached, and also act as a tamping device to reduce bridging. The anticipated volume of filter pack should be calculated.[3] Any discrepancy between the actual and calculated volumes should be explained.

The preferred method for artificial pack installation is to use a ***tremie pipe*** to emplace material directly around the screen. The pipe is raised periodically to help minimize the risk of bridging. The pipe generally should be at least 1.5 inches ID, but larger diameters may be necessary where coarser-grained packs are being installed. When driven casing or hollow-stem augering is used to penetrate non-cohesive formations, the material should be tremied as the casing and auger is pulled back in one to two foot increments to reduce caving effects and ensure proper placement (Nielsen and Schalla, 1991). When installing wells through cohesive formations, the tremie pipe can be used after removal of the drilling device.

Gravity emplacement is accomplished by allowing material to free-fall to the desired position around the screen. Placement by gravity should be restricted to shallow wells with an annular space greater than 2 inches, where the potential for bridging or segregation is minimized (Nielsen and Schalla, 1991). For low-yielding formations, it may be possible to bail the borehole dry to facilitate placement; however, segregation is generally only a problem for deep wells with shallow water levels. Also, segregation is generally not a problem if the pack has a uniformity coefficient of 2.5 or less. Gravity placement also can cause grading if the material is not uniform. In addition, formation materials are often incorporated during placement, which can contaminate the pack and reduce its effectiveness. For most cases, gravity placement is not recommended.

Reverse circulation involves the insertion of a sand and water mixture through the annulus. Sand is deposited around the screen as the water returns to the surface through the casing. Due to the potential water quality alteration, this method generally is not recommended.

Backwashing is accomplished by allowing material to free-fall through the annulus while clean water is pumped down the casing. The water returns up the annulus carrying fine-grained material with it. This creates a more uniform pack; however, the method is not commonly used for monitoring well installation and generally is not recommended due to the potential for alteration of ground water quality. Nonetheless, it is sometimes used for placing packs opposite non-cohesive heaving sands and silts.

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[3] Anticipated filter pack volume can be calculated by determining the difference in volume between the borehole and casing (using outside diameter of the well) from the bottom of the borehole to the appropriate height above the well screen.

SCREEN

The screen is the final link to retaining the borehole and keeping unwanted formation particles out of ground water samples.

Screen Types

Recommended screen compositions are stainless steel, PTFE, and PVC. The same discussion and concerns for casing material apply to screens. Only manufactured screens should be used, since these are available with slots sized precisely for specific grain sizes. Field-cut or punctured screen should never be used, due to the inability to produce the necessary slot size and the potential for the fresh surface to leach or sorb contaminants. A bottom cap or plug should be placed at the base of the screen to prevent sediments from entering and to ensure that all water enters the well through the screen openings.

Slotted and continuous slot, wire-wound screen are the common types used for monitoring wells. In deep wells, slotted screen generally retains structural integrity better than wire-wound; however, continuous slot, wire-wound screens provide almost twice the open area of slotted casing. More open area per unit length enhances well recovery and development. A slot type should be chosen that provides the maximum amount of open area in relation to the effective porosity of the formation. Driscoll (1986) recommended that the percentage of open area should be at least equal to the effective porosity of the formation and filter pack. In common situations with 10 to 30 percent effective porosities, continuous slot screens are preferred, although not required (Nielsen and Schalla, 1991).

Slot Size

When selecting a screen slot size for an artificially filter-packed well, a sieve analysis should be conducted on the pack material. The selected size should retain at least 90% of the pack. In many situations it is preferable to retain 99% (Nielsen and Schalla, 1991 and ASTM D 5092-90, 1994) (Figure 7.3). See Table 7.1 for a guide to the selection of slot sizes for various packs.

For naturally-packed wells, the screen should retain from 30 to 60% (Aller et al., 1991). As a rule of thumb, a 50% retention may be adequate (based on Wisconsin Administrative Code, 1990). With small diameter (4-inch or less), low yield wells, development may not be effective to remove a sufficient amount of fines and a 60 to 70% retention size may be more desirable. For additional information on pack and screen selection, see Aller et al. (1991), Nielsen and Schalla, (1991), and ASTM D 5092-90 (1994).

It should be noted that if a PTFE screen is used in a deep well, a slightly larger slot size than predicted should be selected due to the material's lower compressive strength, which allows the openings to compress (Dablow et al., 1988).

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Figure 7.3 Selection of screen slot size based on the filter pack grain size. (Source: Design and Installation of Ground Water Monitoring Wells by D.M. Nielsen and R. Schalla, *Practical Handbook of Ground Water Monitoring*, edited by David M. Nielsen, Copyright © 1991 by Lewis Publishers, an imprint of CRC Press, Boca Raton, Florida. With permission.)

Length

Screen length should be tailored to the desired zone and generally should not exceed 10 ft. A 2 to 5 ft. screen is desirable for more accurate sampling and discrete head measurements. Longer screens produce composite samples that may be diluted by uncontaminated water. As a result, concentrations of contaminants may be underestimated. Furthermore, the screen should not extend through more than one water-bearing zone to avoid cross-contamination. When a thick formation must be monitored, a cluster of individual, closely spaced wells, screened at various depths, can be installed to monitor the entire formation thickness. The length of screens that monitor the water table surface should account for seasonal fluctuation of the water table. For related information on screen length, refer to Chapter 5.

OPEN BOREHOLE INTAKES

When constructing monitoring wells in competent bedrock, an artificial intake is often unnecessary because an open hole can be maintained and sediment movement is limited. Installing a filter pack in these situations may be difficult due to loss of material into the surrounding formation. In some cases, however, intakes are a necessary component of bedrock wells. A screen and filter pack should be installed in highly weathered, poorly cemented, and fractured bedrock (Nielsen and Schalla, 1991). They are usually necessary when monitoring the unconsolidated/consolidated interface in Ohio.

Open hole wells often are completed by casing and grouting the annulus prior to drilling into the monitoring zone. In cases where the zone has been drilled prior to sealing the annulus, a bridge (cement basket or formation packer shoe) must be set in the hole to retain the grout/slurry to the desired depth (Driscoll, 1986).

If an open hole well is installed, the length of open hole generally should not exceed 10 feet to prevent sample dilution. To maintain a discrete monitoring zone in consolidated formations, the casing should be extended and grouted to the appropriate depth to maintain the 10 foot limit. Driven casing may be necessary to avoid loss of the annular seal into the surrounding formation.

ANNULAR SEALS

The open, annular space between the borehole wall and the casing must be sealed properly to: 1) isolate a discrete zone, 2) prevent migration of surface water, 3) prevent vertical migration of ground water between strata, and 4) preserve confining conditions by preventing the upward migration of water along the casing. An effective seal requires that the annulus be filled completely with sealant and the physical integrity of the seal be maintained throughout the lifetime of the well (Aller et al., 1991).

MATERIALS

The sealant must be of very low permeability (generally 10^{-7} to 10^{-9} cm/sec), capable of bonding with casing, and chemically inert with the highest anticipated concentration of chemicals expected. Cuttings from the existing borehole, no matter what the type of materials, should never be used. They generally exhibit higher permeability and cannot form an adequate seal.
The most common materials used are bentonite and neat cement grout. Each has specific, unique, and desirable properties. These materials are discussed briefly here. Additional information can be found in Gaber and Fisher (1988), ASTM Method C-150 (1992), and Nielsen and Schalla (1991).

Neat Cement Grout

Neat cement grout is comprised of portland cement and water, with no aggregates added. It is a hydraulic cement produced by pulverizing cement clinker consisting essentially of hydrated calcium silicates, and usually containing one or more forms of calcium sulfate as an interground addition. Several types of portland cements are manufactured to accommodate various conditions that may be encountered. Table 7.2 lists the types as classified by ASTM C150-92 (1992). Type I is most commonly used for monitoring wells.

Air-entraining portland cements have been specially processed to form minute air bubbles within the hardened structure. The air-entraining materials are added during the grinding of the clinker. The finished product is more resistant to freeze-thaw action. Air-entraining cements are designated with an "A" after the ASTM cement type. They have been used to construct water supply wells; however, they are less desirable than standard cements because of their greater permeability. Therefore, air-entraining varieties are not recommended for subsurface sealing of monitoring wells.

Water added to the neat cement should be potable and contain less than 500 ppm total dissolved solids (Gaber and Fisher, 1988). Low chloride and sulfate concentrations also are desirable (Campbell and Lehr, 1973). As the water to cement ratio increases, the compressive strength of the cement decreases and shrinkage increases. The American Petroleum Institute recommends a ratio of 5.2 gallons of water per 94 pound sack of cement. Additional water makes it easier to pump, but adversely affects the grout's sealing properties. Excess water can cause shrinkage and separation of the cement particles, which compromises seal integrity (Nielsen and Schalla, 1991).

Table 7.2 ASTM cement designation (modified from Gaber and Fisher 1988).

CEMENT TYPE	DESCRIPTION
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 to 1500 ppm.
Type III	High early strength. Ground to finer particle size, which increases surface area and reduces curing time period before drilling may resume from 48 hours to 12 hours. 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 designated for applications where the rate and amount of heat generated by the cement must be kept to a minimum. Develops strength at a lower rate than Type I.
Type V	Sulfate-resistant cement for use where ground water has a high sulfate content. Recommended for use when levels in ground water exceed 1500 ppm.
Type IA, IIA, and IIIA	Air entraining cements for the same use as Types I, II, and III. Not recommended for monitoring well construction.

The major disadvantages of neat cement are its heat of hydration, shrinkage upon curing, and its effect on water quality. During curing, heat is released, which is generally of little concern; however, generally if large volumes of cement are used or the heat is not rapidly dissipated, the resulting high temperatures can compromise the integrity of PVC casing. However, the borehole for most monitoring wells is small, and heat significant enough to cause damage generally is not created.

Shrinkage is undesirable because it causes cracks and voids. To reduce shrinkage, up to 5 percent bentonite by weight can be added (Gaber and Fisher, 1988), with an additional 1.3 gallons of water for each 2 percent of bentonite per 94 pound sack of cement. Such bentonite/neat cement grout mixtures are highly recommended. Bentonite should not be pre-mixed with water, but should be added dry to the cement/water slurry (ASTM 5092-90, 1994). The addition of bentonite also retards settling time and reduces peak temperatures. Other additives, such as accelerators (e.g., calcium chloride) and retarders, are commercially available but are not recommended due to their potential to leach (ASTM Method 5092-90, 1994).

Upon settling, neat cement grouts often lose water into the formation and affect water quality. Neat cement typically ranges in pH from 10 to 12; therefore, it is important to isolate the annular seal from the screen and filter pack.

Bentonite

Bentonite is composed of clay particles that expand many times their original volume when hydrated. The most acceptable form is a sodium (Na) rich montmorillonite clay that exhibits a 10 to 12-fold expansion when hydrated. Other types, such as calcium (Ca) bentonite, are less desirable because they offer lower swelling ability and surface area to mass ratios. However, other types should be considered if Na bentonite is incompatible with the formation or analyses of concern. For example, the capability of bentonite may be adversely affected by chloride salts, acids, alcohols, ketones, and other polar compounds. Ca bentonite may be more appropriate for calcareous sediments.

Bentonite is available in a variety of forms, including pelletized, coarse grade, granular and powder. ***Pellets*** are uniform in size and consist of compressed, powdered Na montmorillonite. They typically range from 1/4 to 1/2 inch in size. Pellets expand at a relatively slower rate when compared to other forms. ***Coarse grade***, also referred to as crushed or chipped, consists of irregularly shaped, angular particles of montmorillonite that range from 1/4 to 3/4 inches in size. ***Granular*** particles range from 0.025 to 0.10 inches in size. ***Powdered*** bentonite is pulverized montmorillonite, factory-processed after mining. Powered and granular forms are generally mixed with water to form a slurry. Risk of losing a slurry to the underlying filter pack and surrounding formation should be considered. High-solids, bentonite (>30% clay solids) has been developed specifically for monitoring well construction and provides an effective seal.

SEAL DESIGN

It is important that the design of annular seals incorporate measures to prevent infiltration into the filter pack. Contact with the seal can cause sampled ground water to be artificially high in pH. Additionally, bentonite has a high cation exchange capacity, which may affect the chemistry of samples (Aller et al., 1991). In the saturated zone, a 2-foot pure bentonite seal can minimize the threat of infiltration. Above the bentonite seal, neat cement, bentonite, or neat cement/bentonite grouts should be placed in the remainder of the annulus to within a few feet of the surface. Because bentonite requires a sufficient quantity and quality of water in order to achieve and retain hydration, bentonite generally, should only be used in the saturated zone. Where saturated conditions do not exist, neat cement-bentonite should be used.

SEAL INSTALLATION

It is important that annular seals are installed using techniques that prevent bridging, which may cause gaps, cracking or shrinking. Surface water and/or contaminants potentially can migrate through any voids created. The 2 foot bentonite seal above the filter pack is commonly installed by placing granular bentonite, bentonite pellets, or bentonite chips around the casing by dropping them directly down the annulus. If feasible, this practice is acceptable for wells less than 30 feet deep if a tamping device is used. However, for wells deeper than 30 feet, coarse-grained bentonite should be placed by means of a tremie pipe.

The bentonite should be allowed to hydrate or cure prior to sealing the remainder of the annular space. This will help prevent invasion of grout into the screened interval. If a two foot bentonite seal is desired in the unsaturated zone, granular material should be used. It should be added and hydrated in stages using water that is potable and free of analytes of concern.

For the remainder of the annulus, sealants should be in slurry form (e.g., cement grout, bentonite slurry) and should be placed with a tremie pipe (Figure 7.4). The bottom of the pipe should be equipped with a side discharge deflector to prevent the slurry from jetting a hole through the filter pack.[4] The seal should be allowed to completely hydrate, set, or cure in conformance with the manufacturer's specifications prior to completing the surface seal and developing the well.

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Figure 7.4 Tremie pipe emplacement of annular seal material (Source: Design and Installation of Ground Water Monitoring Wells by D.M. Nielsen and R. Schalla, *Practical Handbook of Ground Water Monitoring*, edited by David M. Nielsen, Copyright © 1991 by Lewis Publishers Division, an imprint of CRC Press, Boca Raton, Florida. With permission.)

[4]Side discharge deflectors may not be necessary when a bentonite seal has been placed properly.

SURFACE SEAL/PROTECTIVE CASING COMPLETIONS

SURFACE SEAL

A neat cement or concrete surface seal should be placed around a protective casing to a depth just below the frost line (3-5 ft.). If the same material was used in the annular seal, the surface seal can be a continuation; otherwise, the surface seal is installed directly over the annular seal after settling and curing. The surface seal should slope away from the well and extend beyond the edge of the borehole to divert surface water. Air-entraining cements may be desirable in cold climates to alleviate cracking caused by freezing and thawing.

ABOVE-GROUND COMPLETIONS

Whenever possible, monitoring wells should extend above the ground surface to prevent surface water from entering and to enhance visibility. From the frost line upward, a steel protective casing should encompass the well. The protective casing should be at least two inches larger in diameter than the inner casing, extend above it, and have a locking cap. The lock should be protected by plastic or rubber covers so the use of lubricants to free and maintain locking mechanisms can be avoided. A small drain or "weep hole" should be located just above the surface seal to prevent the accumulation of water between the casings (See Figure 7.1). This is especially useful in cold climates, where the freezing of trapped water can damage the inner casing. A permanent reference point on the well inner casing must be surveyed to the nearest 0.01 ft. This permanent marker should be used for all water level measurements. Additionally, the well identification number or code should be marked permanently and clearly.

Bumper or barrier guards should be placed beyond the edge of the surface seal or within 3 to 4 feet of the well (See Figure 7.1). These guards are necessary to reduce and prevent accidental damage from vehicles. Painting the guard posts yellow or orange and installing reflectors can increase visibility and help prevent mishaps.

FLUSH-TO-GROUND COMPLETIONS

Flush-to-ground completions are discouraged because the design increases the potential for surface water infiltration; however, they are occasionally unavoidable. This type of completion should be used only when the location of a well would disrupt traffic areas such as streets, parking lots, and gas stations, or where easements require them (Nielsen and Schalla, 1991).

If flush-to-ground completion is installed, very careful procedures must be followed. A highly secure subsurface vault generally is completed in the surface seal, allowing the well casing to be cut below grade (Figure 7.5). An expandable locking cap on the casing and a water-proof gasket should be installed around the vault lid to prevent surface water infiltration. The completion should be raised slightly above grade and sloped away to help divert surface water. It should be marked clearly and locked to restrict access. This is especially important at gas stations to prevent the misidentification of wells as underground tank filling points.

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Figure 7.5 Typical flush-to-ground monitoring well completion (Source: Design and Installation of Ground Water Monitoring Wells by D.M. Nielsen and R. Schalla, *Practical Handbook of Ground Water Monitoring*, edited by David M. Nielsen, Copyright © 1991 by Lewis Publishers Division, an imprint of CRC Press, Boca Raton, Florida. With permission.)

DOCUMENTATION

During monitoring well installation, pertinent information should be documented, including design and construction, the drilling procedure, and the materials encountered (see Chapter 3 for a listing of the particular geologic information needs). Accurate "as-built" diagrams should be prepared that, in general, include the following:

- Date/time of start and completion of construction.
- Boring/well number.
- Drilling method and drilling fluid used.
- Borehole diameter and well casing diameter.
- Latitude and longitude.
- Well location (± 0.5 ft.) with sketch of location.
- Borehole depth (± 0.1 ft.).
- Well depth (± 0.1 ft.).
- Casing length and materials.
- Screened interval(s).
- Screen materials, length, design, and slot size.
- Casing and screen joint type.

- Depth/elevation of top and bottom of screen.
- Filter pack material/size, volume calculations, and placement method.
- Depth/elevation to top and bottom of filter pack.
- Annular seal composition, volume (calculated and actual), and placement method.
- Surface seal composition, placement method, and volume (calculated and actual).
- Surface seal and well apron design/construction.
- Depth/elevation of water.
- Well development procedure and ground water turbidity.
- Type/design of protective casing.
- Well cap and lock.
- Ground surface elevation ($\pm$ 0.01 ft.).
- Surveyed reference point ($\pm$ 0.01 ft.) on well casing.
- Detailed drawing of well (include dimensions).
- Point where water encountered.
- Water level after completion of well development.

In addition, the following should be documented in work plans (when appropriate) and reports:

- Selection and rationale materials for selection of casing and screen.
- Selection and rationale for well diameter, screen length, and screen slot size.
- Filter pack selection and emplacement.
- Annular sealant selection and emplacement.
- Security measures.
- Locations and elevations of wells.
- Well development.

A complete, ongoing history of each well should be maintained. This can include sample collection dates, dates and procedures for development, water level elevation data, problems, repairs, personnel, and methods of decommissioning. This information should be kept as a permanent on-site file, available for agency review upon request.

On July 18, 1990, Ohio House Bill 476 went into effect. This bill requires that all logs for monitoring wells drilled in Ohio be submitted to the Ohio Department of Natural Resources, Division of Water (ODNR). The ODNR can be contacted for further information.

MAINTENANCE

The condition of wells must be maintained to keep them operational and insure that representative samples can be obtained. Maintenance consists of conducting inspections and periodic checks on performance. Proper documentation (see previous section) is needed to serve as a benchmark for evaluation. Maintenance includes, but is not limited to, the following:

- Ensuring visibility and accessibility
- Inspecting locks for rusting
- Inspecting surface seals for cracking.
- Checking survey marks to insure visibility.

- Determining depth (see Chapter 10 for recommended procedures).
- Removing sediments (if needed).
- Evaluating performance by doing hydraulic conductivity tests.
- Evaluating turbidity and re-developing or replacing well if turbidity increases.

Routine inspections generally can be conducted during sampling. Additional evaluation can be conducted by comparing new ground water quality data with previous data. If the maintenance check indicates a problem, rehabilitation should be conducted.

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KEMRON
ENVIRONMENTAL SERVICES

WELL DEVELOPMENT LOG

PROJECT NAME:	WELL NUMBER:
PROJECT NUMBER:	DATE:
PROJECT LOCATION:	SAMPLING PERSONNEL:
	WEATHER CONDITIONS:
DEVELOPMENT METHOD:	CASING RADIUS: (in.)
MONITORING METHOD:	DEPTH TO WATER (DTW): (ft.)
GALLONS PURGED:	DEPTH TO BOTTOM OF WELL: (ft.)
	WATER COLUMN HEIGHT: (ft.)
	1 WELL VOLUME (CH)(CR^2)(0.163)= (gal.)
PURGE START TIME:	PURGE RATE (GPM):
PURGE STOP TIME:	WELL VOLUMES PURGED:

TIME / GALLONS PURGED	TEMPERATURE C°	pH	CONDUCTIVITY (mnos/cm)	TURBIDITY (NTU)	OTHER

SAMPLE ID:	--	SAMPLE APPEARANCE:	--
SAMPLE COLLECTION TIME:	--	LABORATORY NAME:	--

OBSERVATIONS / COMMENTS:

Document Control #: 12-PID-AKMW

STANDARD OPERATING PROCEDURE FOR FIELD SCREEN OF SAMPLES USING PHOTO-IONIZATION DETECTOR SITE SPECIFIC TO AK STEEL MIDDLETOWN WORKS

Issue Date: April 10, 2013
Revision 00

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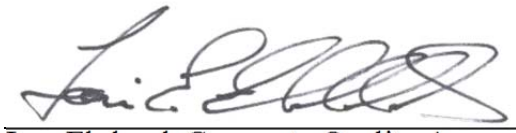
Approved by:



Lou Ehrhard, Corporate Quality Assurance Manager — 04/10/2013 Date

John Dwyer, Executive Vice President — 04/10/2013 Date

C O N T E N T S

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P R O C E D U R E S

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure describes the methods to be followed to assess soil or fluids for volatile hydrocarbon contamination from the field using a PID.

1.2 The purpose of this procedure is to ensure a uniform method of screening soils or fluids in the field for VOCs. Screening may be used to select samples for submittal for laboratory analysis.

2.0 SUMMARY OF METHOD

2.1 Collected soil or fluids are sealed within a sealable bag (Ziploc® bag or similar) container and volatiles are allowed to equilibrate within the headspace of the bag or container. A PID is inserted into the bag or container to acquire a reading.

3.0 DEFINITIONS

3.1 HASP – Health and Safety Plan

3.2 HAZWOPER – Hazardous Waste Operations and Emergency Response

3.3 OSHA – Occupational Health and Safety Association

3.4 PID – photo-ionization detector

3.5 PPE – personal protective equipment

3.6 SAP – Sampling and Analysis Plan

3.7 SOP – standard operating procedure

3.8 VOC – volatile organic compound

4.0 HEALTH AND SAFETY

4.1 Review the site-specific HASP prior to conducting any procedures described in this SOP.

4.2 As dictated by the site-specific HASP, wear appropriate PPE when working with potentially hazardous materials and/or potentially hazardous environments.

5.0 PERSONNEL REQUIREMENTS

5.1 All involved personnel must be 40-hour OSHA HAZWOPER certified (29 CFR 1910.120).

5.2 All involved personnel must be in compliance with the Health and Safety Plan.

5.3 All involved personnel must be in compliance with any site-specific requirements.

6.0 POTENTIAL EQUIPMENT AND SUPPLIES

- PID
- Sealable bags (Ziploc® or similar)
- Wide mouth glass containers with lids (8 or 16 oz.)
- Thermometer or temperature strip
- Aluminum foil
- Power drill with ¼" drill bit, or similar
- Field notebook
- Waterproof pens/markers
- Appropriate PPE

7.0 BAG COLLECTION METHODS

NOTE: Headspace screening methods must be conducted while sample is at an ambient temperature (>10°C). A replicate soil sample should be collected as described below. Include a thermometer or temperature strip within the sealed bag of the replicate sample. Once sealed, the sample and replicate sample may be moved to a warmed vehicle (or similar) to achieve ambient temperature. After the replicate sample reaches ambient temperature, PID reading can be taken for all samples.

7.1 Collect soil samples according to the site-specific SAP or work plan.

7.2 Transfer a sufficient amount of the sample to fill approximately one-half the volume of the sealable bag (Ziploc® or similar) and seal the bag.

7.3 Knead the contents of the bag until the consistency of the sample is uniform.

7.4 Allow at least 15 minutes, but not more than 2 hours, for the VOCs to develop headspace equilibrium.

7.5 Warm up and calibrate the PID instrument to be used according to the manufacturer's manual or SOP.

7.6 Knead contents of bag for another 30 seconds.

7.7 Quickly open the seal from the corner of the bag just enough to insert the PID probe and allow the probe to measure the VOC concentration for approximately 15 seconds. Do not insert the probe tip into the sample as the probe may become obstructed.

7.8 During those 15 seconds, record the maximum reading response on the probe in a field notebook.

8.0 CONTAINER COLLECTION METHODS

NOTE: Headspace screening methods must be conducted while sample is at an ambient temperature (>10°C). A replicate soil sample should be collected as described below. Include a thermometer or temperature strip within the sealed container of the replicate sample. Once sealed, the sample and replicate sample may be moved to a warmed vehicle (or similar) to achieve ambient temperature. After the replicate sample reaches ambient temperature, PID reading can be taken for all samples.

8.1 Collect soil or fluid samples according to the site-specific SAP or work plan.

8.2 Transfer a sufficient amount of the sample to fill approximately one-half the volume of the container.

8.3 Cover the mouth of the container with aluminum foil and seal the container over the foil with the lid. *NOTE: To allow PID probe penetration, use the threaded piece of a two-piece type lid or create a ¼" hole (or similar) through the top of a one-piece lid prior to sample collection.*

8.4 Shake the contents of the container for 15-30 seconds.

8.5 Allow at least 15 minutes, but not more than 2 hours, for the VOCs to develop headspace equilibrium.

8.6 Warm up and calibrate the PID instrument to be used according to the manufacturer's manual or SOP.

8.7 Again, shake the contents of the container for 15-30 seconds

8.8 Quickly insert the PID probe through the aluminum foil seal and allow the probe to measure the VOC concentration for approximately 15 seconds. Do not insert the probe tip into the soil or fluid sample as the probe may become obstructed.

8.9 During those 15 seconds, record the maximum reading response on the probe in a field notebook.

9.0 DECONTAMINATION METHODS

9.1 Follow specific methods described in PID operation manual for decontamination procedures relating to the PID lamp and PID probe.

9.2 Containers or bag are intended for one time use. Do not reuse.

10.0 DOCUMENTATION AND RECORD MANAGEMENT

10.1 Field notes should include the probe model, specific lamp energy setting and calibration standards.

11.0 QUALITY ASSURANCE/ QUALITY CONTROL

11.1 Review all field data records for accuracy and completeness.

12.0 REFERENCES

12.1 "*DNAPL Site Characterization*" EPA/540/F-94/049, U.S. EPA September 1994.

12.2 "*Site Characterization Technologies for DNAPL Investigations*." U.S. EPA, EPA/542-R-04-017, September 2004.

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STANDARD OPERATING PROCEDURE
FOR SOIL BORING COLLECTION
SITE SPECIFIC TO AK STEEL MIDDLETOWN WORKS

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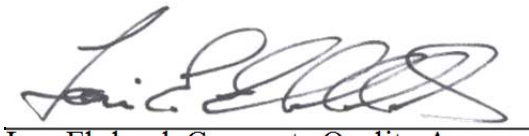

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P R O C E D U R E S

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure describes the methods to be followed during drilling of soil borings. Direct-push methods will be considered for use initially. If the direct-push sampler cannot penetrate or are not expected to penetrate to a sufficient depth as specified in the site-specific SAP or work plan, drilling methods (e.g., HSA, rotosonic) will be used.

1.2 The purpose of this procedure is to establish a uniform method for drilling soil borings to assure quality control in field operations and uniformity among different field teams.

2.0 SUMMARY OF METHOD

2.1 Advance the direct push sampler (e.g., Geoprobe®) or other drilling method (e.g., HSA or rotosonic) to the desired sampling depth.

2.2 Once the boring has been advanced, use a subsurface soil sampler to collect the sample.

2.3 Record blow counts, recovery, and sampling depth in Boring Log and field notebook.

3.0 DEFINITIONS

3.1 HASP – Health and Safety Plan

3.2 HAZWOPER – Hazardous Waste Operations and Emergency Response

3.3 HSA – hollow stem auger

3.4 OSHA – Occupational Health and Safety Association

3.5 PPE – personal protective equipment

3.6 PVC – polyvinyl chloride

3.7 QAPP – Quality Assurance Project Plan

3.8 SAP – Sampling and Analysis Plan

3.9 SOP – standard operating procedure

4.0 HEALTH AND SAFETY

4.1 Review the site-specific HASP prior to conducting any procedures described in this SOP.

4.2 As dictated by the site-specific HASP, wear appropriate PPE when working with potentially hazardous materials and/or potentially hazardous environments.

5.0 PERSONNEL REQUIREMENTS

5.1 All involved personnel must be 40-hour OSHA HAZWOPER certified (29 CFR 1910.120).

5.2 All involved personnel must be in compliance with the Health and Safety Plan.

5.3 All involved personnel must be in compliance with any site-specific requirements.

6.0 POTENTIAL EQUIPMENT AND SUPPLIES

- Direct-push sampler (Geoprobe® or equivalent) or drill rig (HSA or similar)
- Sampling tube (e.g., Shelby Tube, core liner) with end caps
- 4-inch threaded Schedule 40 PVC
- Disposable aluminum pans or stainless steel pans
- Boring logs
- Soil/sediment sampling logs
- Stainless steel trowels/spoons
- Waterproof pens/markers
- Tape measure
- Field notebook
- Alconox® (or similar) solution
- Distilled water
- Laboratory grade, analyte-free water
- Appropriate PPE

7.0 PRE-DRILLING/COLLECTION METHODS

7.1 If required, obtain soil boring permits from the appropriate agency or agencies before drilling is initiated.

7.2 Conduct an underground utility check before drilling begins.

8.0 DIRECT-PUSH COLLECTION METHODS (TWO TUBE)

NOTE: Methods may vary by direct-push rig. Always consult the respective operating manuals and SOPs.

8.1 Thread the cutting shoe onto the bottom the outer casing. Place the internal liner drive head onto a decontaminated sampling tube and insert this configuration inside the outer casing. Place drive bumper on top of internal liner drive head and

thread the outer drive cap onto the outer casing. *NOTE: Alternatively, a solid drive tip with internal rods and internal drive cap may be used in place of sampling tube, internal liner drive head, and drive bumper if certain depth intervals are to be bypassed.*

8.2 Drive direct-push sampler (Geoprobe® or equivalent) into the subsurface.

8.3 Remove outer drive cap and drive bumper. Thread internal rod to internal drive cap.

8.4 Rotate sampling tube within borehole at least 2 rotations to shear the bottom of the sample core. Retrieve the sampling tube and cap for processing.

8.5 Place the internal liner drive head onto a decontaminated sampling tube and insert this configuration inside the outer casing. Thread internal rod to internal liner drive head. Attach internal drive cap. *NOTE: At the subsequent depths, use internal rod clamp to hold internal rod as the sampling tube is lowered and additional internal rods are threaded on until sampling tube reaches the base of the outer casing.*

8.6 Lower additional outer casing over internal rod and thread to submerged outer casing. Place internal drive head on top of the internal rod and thread outer drive cap to outer casing. Repeat steps 8.2 through 8.5 to continue the collection of soil samples until desired sampling depth is reached.

9.0 HSA DRILLING METHODS

NOTE: Methods may vary slightly based on site conditions. If soils are saturated and/or may be contaminated, then a PVC casing and annulus seal must be used to prevent water/contaminant migration into the borehole. If soils are neither saturated nor contaminated, then a smaller auger (4½-inch) can be used and the borehole does not need to be cased with PVC.

9.1 Obtain surface soil sample, if required by site-specific SAP or work plan.

9.2 Advance 6¼-inch HSA (or 4½-inch HSA, as appropriate) flights with auger head to desired sampling depth. Cuttings are rotated to the surface as the borehole is advanced. *NOTE: A pilot or center bit can be held at the base of the first flight with drill rods to prevent cuttings from entering if soil collection is not necessary.*

9.3 Remove the bit and attach a decontaminated sampling split spoon or thin-wall sampler onto the drive rod and lower the sampler to the base of the borehole.

9.4 Once the sampler is at the desired depth, collect the sample by driving it into the subsurface with a 140 pound weight (i.e., “hammer”). Drive the sampler about 6 inches less than its length to avoid sample compression. Record tin the Boring Log and the field notebook the number of blows required to achieve the desired sample depth. *NOTE: Sample refusal occurs when blows exceed 50 with little or no downward progress.*

9.5 Retrieve the sampling tube and note sample recovery in the Boring Log and field notebook.

9.6 Remove the disturbed soil from the top of the tube and one inch of soil from the bottom of the tube. The remainder of the sample may be used for chemical analyses and physical characterization.

9.7 Any cuttings should be disposed as specified in the site-specific SAP or work plan.

9.8 Repeat steps 9.2 through 9.7 to continue the advancement of the HSA flights and collection of soil samples until desired sampling depth is reached.

10.0 ROTOSONIC DRILLING METHODS

NOTE: Methods may vary slightly based on site conditions. If soils are saturated and/or may be contaminated, then a PVC casing and annulus seal must be used to prevent water/contaminant migration into the borehole. If soils are neither saturated nor contaminated, then a smaller auger (4½-inch) can be used and the borehole does not need to be cased with PVC.

10.1 Obtain surface soil sample, if required by site-specific SAP or work plan.

10.2 Advance the 6-inch or 8-inch coring barrel to desired sampling depth by using a combination of rotation and high frequency vibration. Cuttings are rotated to the surface as the borehole is advanced. A pilot or center bit can be held at the base of the first flight with drill rods to prevent cuttings from entering.

10.3 Once the desired depth is reached, stop the vibration.

10.4 Retrieve the core barrel by vibrating or hydraulically extracting into a plastic sleeve or sampling tray. Remove the disturbed soil from the top of the tube and one inch of soil from the bottom of the tube. *NOTE: Alternatively, a split spoon sampler may be lowered within the casing and driven into the subsurface with a 140 pound weight (i.e., "hammer") for sample collection.*

10.5 Any cuttings should be disposed as specified in the SAP or work plan.

11.0 DECONTAMINATION METHODS

11.1 Follow decontamination methods described in the QAPP.

12.0 QUALITY ASSURANCE/QUALITY CONTROL

12.1 Review all field data records for accuracy and completeness.

12.2 In order to ensure that decontamination procedures are effective, equipment rinseate blanks may be collected, as dictated by the SAP or QAPP.

12.3 In order to ensure that sampling methods are effective and representative, field duplicates may be collected, as dictated by the SAP or QAPP.

13.0 REFERENCES

13.1 OEPA. 2005. *Technical Guidance for Ground Water Investigations: Sealing Abandoned Monitoring Wells and Boreholes.* Ohio Environmental Protection Agency, Division of Drinking and Ground Waters.

TERRACON LAB SOPS

Construction Material Testing Laboratory **SOP Number: SOP-GT-002**
Terracon **Effective: June 1999**
Revision: April 2006

STANDARD OPERATING PROCEDURE FOR PROCESSING BULK SAMPLES

STEP 1

Set up Soil Laboratory Worksheet. See SOP-GT-001, Step 4 for reference. Weigh entire content of the bulk sample and record in Remarks Section on worksheet.

STEP 2

Lay out entire sample on a flat pan in proctor area.

STEP 3

Completely fill out two sample tags and place one with sample and one on a bucket for use later. Include project name and number, sample number or I.D. Collect approximately 1500 grams for sieve analysis, place into bucket or flat pan and set aside.

STEP 4

Collect a representative 300-500 gram sample for Natural Moisture Content (NMC) prior to any drying of the bulk sample. Place into a sealed plastic bag and leave with bulk sample. (The NMC sample may be used for P-200, See SOP-GT-07.4).
NOTE: Should the bulk sample be in excess of 10% greater than the estimated Optimum Moisture Content (OMC), allow the entire bulk sample to air dry until the moisture content has been reduced to about 6% greater than the estimated OMC. Then proceed with STEP 5.

STEP 5

Dice and chop bulk sample in the flat pan and remove, weigh and record on worksheet any large stones; place bulk samples into the Rapid Soil Processor. Make the necessary hook-ups on the Soil Processor. Place the larger black tub under, and the smaller black tub towards the back of the Soil Processor. Turn on the Soil Processor and allow it to shave the entire bulk sample until all the soil has been “Processed”. USE

CAUTION!

STEP 6

From the Processed Bulk Sample, collect about 200 grams of moist soil for Atterberg and place into a marked container. Record container's ID on worksheet in Atterberg Limits Section. Place distilled water in container covering soil and place in classification testing area of the laboratory.

STEP 6.1

From the Processed Bulk Sample, collect approximately 200 grams of moist soil for the hydrometer analysis (Wet Method) and place into a marked container. Record container ID on worksheet in Hydrometer Section. Place distilled water in container covering soil and place in classification testing area.

STEP 7

Check worksheet for completeness. Leave worksheet with bulk samples.

Construction Material Testing Laboratory **SOP Number: SOP-GT-005**
Terracon **Effective: June 1999**
Revision: August 2011

STANDARD OPERATING PROCEDURE
FOR
ASTM D4318 – Liquid Limit, plastic limit, and Plasticity Index of Soils

STEP 1
Complete all project information on the data sheet.

STEP 2
Collect 200 gms from the processed bulk sample - see SOP-GT-002, Step 5. Pulverize the sample in a mortar with a rubber covered pestle. Sieve the pulverized sample over a #40 sieve. Collect the minus #40 soil in a container. Place material retained on the #40 sieve in a dish and soak in a small amount of water. Wash the soaked material over a #40 sieve. Add the material passing #40 from wash to the minus #40 material from dry sieve. Mix the material with distilled water to about plastic limit moisture. Soak the material in the covered container at least 16 hours before test.

STEP 3 (Method- A)
Calibrate drop of liquid limit cup using the grooving tool handle or gauge block. Place approximately 150 gms of the soil into a mixing dish and thoroughly mix. Spread soil into the liquid limit cup approximately 10 mm thick. Cover the soil box with a wet towel. Cut a grove through the center of the filled cup from highest point of the cup to the lowest using the grooving tool. Turn the handle on the limit cup at a rate of about two blows (drops) per second and count the number of blows (drops) of the limit cup required to close the groove between the two halves approximately ½ inch. Point one should require 25 to 35 blows to close the groove. Point two should require 20 to 30 blows to close the groove. Point three should require 15 to 25 blows to close the groove. Collect a sample of this material for each point perpendicular to and across the closed groove and place in a tare and weigh and record the weight of each sample plus tare on the data sheet and place them in the drying oven.

STEP 4
Take the four 1.5 to 2.0 gms per each of the two test plastic limit samples and roll them out on a glass plate, one sample at a time. Continue rolling and kneading until the soil

sample can no longer be rolled out to an 1/8" thread. Collect 1 sample of this rolled out material and place in a tare (minimum 6 gms each). Weigh and record the weight of each sample and tare and place them in the drying oven in accordance with Test Method D2216.

STEP 5

Check the data sheet for completeness.

Method - B

The one-point method should not be used, unless the client specifies. If the one-point method is used, run the test twice with the same moisture content. The blows should be between 20 and 30 and the difference in blows of the two tests should be no more than two blow difference. Take another moisture content specimen. If the difference is greater than two blows, remix the entire specimen and repeat the procedure until achieved. When two successful specimens have been taken, multiply the one content by a correction factor to determine the liquid limit.

Designation: D4318 – 10

Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils[1]

This standard is issued under the fixed designation D4318; 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 liquid limit, plastic limit, and the plasticity index of soils as defined in Section 3 on Terminology.

1.2 Two methods for preparing test specimens are provided as follows: *Wet preparation method*, as described in 10.1. *Dry preparation method*, as described in 10.2. The method to be used shall be specified by the requesting authority. If no method is specified, use the wet preparation method.

1.2.1 The liquid and plastic limits of many soils that have been allowed to dry before testing may be considerably different from values obtained on non-dried samples. If the liquid and plastic limits of soils are used to correlate or estimate the engineering behavior of soils in their natural moist state, samples should not be permitted to dry before testing unless data on dried samples are specifically desired.

1.3 Two methods for determining the liquid limit are provided as follows: *Method A*, Multipoint test as described in Sections 11 and 12. *Method B*, One-point test as described in Sections 13 and 14. The method to be used shall be specified by the requesting authority. If no method is specified, use Method A.

1.3.1 The multipoint liquid limit method is generally more precise than the one-point method. It is recommended that the multipoint method be used in cases where test results may be subject to dispute, or where greater precision is required.

1.3.2 Because the one-point method requires the operator to judge when the test specimen is approximately at its liquid limit, it is particularly not recommended for use by inexperienced operators.

1.3.3 The correlation on which the calculations of the one-point method are based may not be valid for certain soils, such as organic soils or soils from a marine environment. It is strongly recommended that the liquid limit of these soils be determined by the multipoint method.

1.4 The plastic limit test is performed on material prepared for the liquid limit test.

1.5 The liquid limit and plastic limit of soils (along with the shrinkage limit) are often collectively referred to as the Atterberg limits. These limits distinguished the boundaries of the several consistency states of plastic soils.

1.6 The composition and concentration of soluble salts in a soil affect the values of the liquid and plastic limits as well as the water content values of soils (see Method D4542). Special consideration should therefore be given to soils from a marine environment or other sources where high soluble salt concentrations may be present. The degree to which the salts present in these soils are diluted or concentrated must be given careful consideration.

1.7 The methods described herein are performed only on that portion of a soil that passes the 425-μm (No. 40) sieve. Therefore, the relative contribution of this portion of the soil to the properties of the sample as a whole must be considered when using these tests to evaluate properties of a soil.

1.8 The values stated in SI units are to be regarded as the standard, except as noted below. The values given in parentheses are for information only.

1.8.1 The standard units for the resilience tester covered in Annex A1 are inch-pound, not SI. The SI values given are for information only.

1.9 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.9.1 For purposes of comparing a measured or calculated value(s) with specified limits, the measured or calculated value(s) shall be rounded to the nearest decimal or significant digits in the specified limits

1.9.2 The procedures used to specify how data are collected/recorded or 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 do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the

[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.

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

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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 this standard to consider significant digits used in analysis methods for engineering design.

1.10 *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]

C702 Practice for Reducing Samples of Aggregate to Testing Size
D75 Practice for Sampling Aggregates
D420 Guide to Site Characterization for Engineering Design and Construction Purposes
D653 Terminology Relating to Soil, Rock, and Contained Fluids
D1241 Specification for Materials for Soil-Aggregate Subbase, Base, and Surface Courses
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)
D3282 Practice for Classification of Soils and Soil-Aggregate Mixtures for Highway Construction Purposes
D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction
D4542 Test Method for Pore Water Extraction and Determination of the Soluble Salt Content of Soils by Refractometer
D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing
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 Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 For common definitions of terms in this standard, refer to Terminology D653.

3.1.2 *Atterberg Limits*—Originally, six "limits of consistency" of fine-grained soils were defined by Albert Atterberg: the upper limit of viscous flow, the liquid limit, the sticky limit, the cohesion limit, the plastic limit, and the shrinkage limit. In current engineering usage, the term usually refers only to the liquid limit, plastic limit, and in some references, the shrinkage limit.

3.1.3 *consistency*—the relative ease with which a soil can be deformed.

3.1.4 *liquid limit (LL, w_L)*—the water content, in percent, of a soil at the arbitrarily defined boundary between the semi-liquid and plastic states.

3.1.4.1 *Discussion*—The undrained shear strength of soil at the liquid limit is considered to be approximately 2 kPa (0.28 psi).

3.1.5 *plastic limit (PL, w_p)*—the water content, in percent, of a soil at the boundary between the plastic and semi-solid states.

3.1.6 *plastic soil*—a soil which has a range of water content over which it exhibits plasticity and which will retain its shape on drying.

3.1.7 *plasticity index (PI)*—the range of water content over which a soil behaves plastically. Numerically, it is the difference between the liquid limit and the plastic limit.

3.1.8 *liquidity index*—the ratio, expressed as a percentage of (1) the water content of a soil minus its plastic limit, to (2) its plasticity index.

3.1.9 *activity number (A)*—the ratio of (1) the plasticity index of a soil to (2) the percent by mass of particles having an equivalent diameter smaller than 2 µm.

4. Summary of Test Method

4.1 The specimen is processed to remove any material retained on a 425-µm (No. 40) sieve. The liquid limit is determined by performing trials in which a portion of the specimen is spread in a brass cup, divided in two by a grooving tool, and then allowed to flow together from the shocks caused by repeatedly dropping the cup in a standard mechanical device. The multipoint liquid limit, Method A, requires three or more trials over a range of water contents to be performed and the data from the trials plotted or calculated to make a relationship from which the liquid limit is determined. The one-point liquid limit, Method B, uses the data from two trials at one water content multiplied by a correction factor to determine the liquid limit.

4.2 The plastic limit is determined by alternately pressing together and rolling into a 3.2-mm (⅛-in.) diameter thread a small portion of plastic soil until its water content is reduced to a point at which the thread crumbles and can no longer be pressed together and re-rolled. The water content of the soil at this point is reported as the plastic limit.

4.3 The plasticity index is calculated as the difference between the liquid limit and the plastic limit.

5. Significance and Use

5.1 These test methods are used as an integral part of several engineering classification systems to characterize the fine-grained fractions of soils (see Practices D2487 and D3282) and to specify the fine-grained fraction of construction materials (see Specification D1241). The liquid limit, plastic limit, and plasticity index of soils are also used extensively, either individually or together, with other soil properties to correlate

[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.

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with engineering behavior such as compressibility, hydraulic conductivity (permeability), compactibility, shrink-swell, and shear strength.

5.2 The liquid and plastic limits of a soil and its water content can be used to express its relative consistency or liquidity index. In addition, the plasticity index and the percentage finer than 2-μm particle size can be used to determine its activity number.

5.3 These methods are sometimes used to evaluate the weathering characteristics of clay-shale materials. When subjected to repeated wetting and drying cycles, the liquid limits of these materials tend to increase. The amount of increase is considered to be a measure of a shale's susceptibility to weathering.

5.4 The liquid limit of a soil containing substantial amounts of organic matter decreases dramatically when the soil is oven-dried before testing. Comparison of the liquid limit of a sample before and after oven-drying can therefore be used as a qualitative measure of organic matter content of a soil (see Practice D2487.

NOTE 1—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 D3740, generally, are considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard 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.

6. Apparatus

6.1 *Liquid Limit Device*—A mechanical device consisting of a brass cup suspended from a carriage designed to control its drop onto the surface of a block of resilient material that serves as the base of the device. Fig. 1 shows the essential features and critical dimensions of the device. The device may be operated by either a hand crank or electric motor.

6.1.1 *Base*—A block of material having a resilience rebound of at least 77 % but no more than 90 %. Conduct resilience tests on the finished base with the feet attached. Details for measuring the resilience of the base are given in Annex A1.

6.1.2 *Rubber Feet*, supporting the base, designed to provide dynamic isolation of the base from the work surface.

6.1.3 *Cup*, brass, with a mass, including cup hanger, of 185 to 215 g.

6.1.4 *Cam*—Designed to raise the cup smoothly and continuously to its maximum height, over a distance of at least 180° of cam rotation, without developing an upward or downward velocity of the cup when the cam follower leaves the cam. (The preferred cam motion is a uniformly accelerated lift curve.)

NOTE 2—The cam and follower design in Fig. 1 is for uniformly accelerated (parabolic) motion after contact and assures that the cup has no velocity at drop off. Other cam designs also provide this feature and may be used. However, if the cam-follower lift pattern is not known, zero velocity at drop off can be assured by carefully filing or machining the cam and follower so that the cup height remains constant over the last 20 to 45° of cam rotation.

6.1.5 *Carriage*, constructed in a way that allows convenient but secure adjustment of the height-of-drop of the cup to 10 mm (0.394 in.), and designed such that the cup and cup hanger assembly is only attached to the carriage by means of a

DIMENSIONS

LETTER	A △	B △	C △	E △	F	G	H	J △	K △	L △	M △
MM	54 ± 0.5	2 ± 0.1	27 ± 0.5	56 ± 2.0	32	10	16	60 ± 1.0	50 ± 2.0	150 ± 2.0	125 ± 2.0

LETTER	N	P	R	T	U △	V	W	Z
MM	24	28	24	45	47 ± 1.0	3.8	13	6.5

△ ESSENTIAL DIMENSIONS

CAM ANGLE DEGREES	CAM RADIUS
0	0.742 R
30	0.753 R
60	0.764 R
90	0.773 R
120	0.784 R
150	0.796 R
180	0.818 R
210	0.854 R
240	0.901 R
270	0.945R
300	0.974R
330	0.995R
360	1.000 R

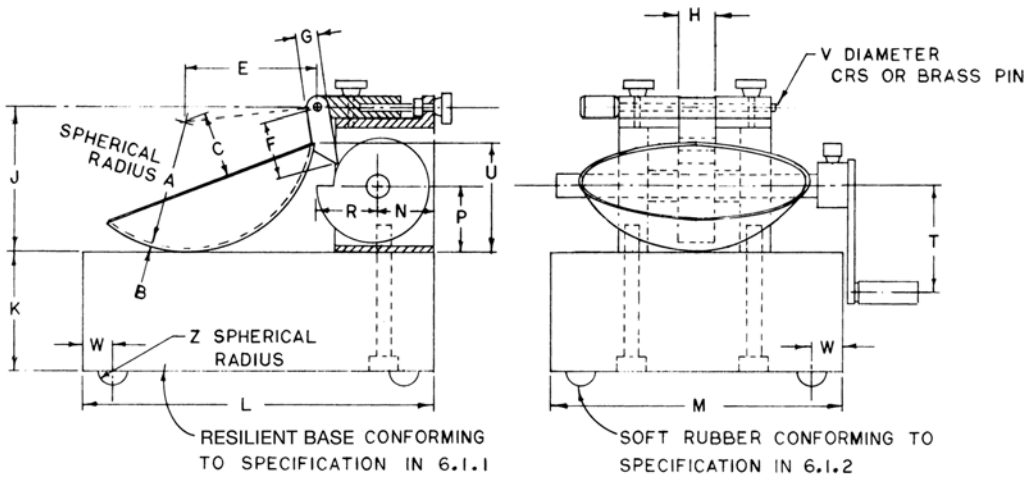

FIG. 1 Hand-Operated Liquid Limit Device

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removable pin. See Fig. 2 for definition and determination of the height-of-drop of the cup.

6.1.6 *Motor Drive (Optional)*—As an alternative to the hand crank shown in Fig. 1, the device may be equipped with a motor to turn the cam. Such a motor must turn the cam at 2 ± 0.1 revolutions per second and must be isolated from the rest of the device by rubber mounts or in some other way that prevents vibration from the motor being transmitted to the rest of the apparatus. It must be equipped with an ON-OFF switch and a means of conveniently positioning the cam for height-of-drop adjustments. The results obtained using a motor-driven device must not differ from those obtained using a manually operated device.

6.2 *Flat Grooving Tool*—A tool made of plastic or noncorroding-metal having the dimensions shown in Fig. 3. The design of the tool may vary as long as the essential dimensions are maintained. The tool may, but need not, incorporate the gauge for adjusting the height-of-drop of the liquid limit device.

NOTE 3—Prior to the adoption of this test method, a curved grooving tool was specified as part of the apparatus for performing the liquid limit test. The curved tool is not considered to be as accurate as the flat tool described in 6.2 since it does not control the depth of the soil in the liquid limit cup. However, there are some data which indicate that typically the liquid limit is slightly increased when the flat tool is used instead of the curved tool.

6.3 *Gauge*—A metal gauge block for adjusting the height-of-drop of the cup, having the dimensions shown in Fig. 4. The design of the tool may vary provided the gauge will rest securely on the base without being susceptible to rocking, and the edge which contacts the cup during adjustment is straight, at least 10 mm (⅜ in.) wide, and without bevel or radius.

6.4 *Water Content Containers*—Small corrosion-resistant containers with snug-fitting lids for water content specimens. Aluminum or stainless steel cans 2.5 cm (1 in.) high by 5 cm (2 in.) in diameter are appropriate.

6.5 *Balance*, conforming to Specification D4753, Class GP1 (readability of 0.01 g).

6.6 *Mixing and Storage Container*—A container to mix the soil specimen (material) and store the prepared material. During mixing and storage, the container shall not contaminate the material in any way, and prevent moisture loss during storage. A porcelain, glass, or plastic dish about 11.4 cm (4½ in.) in diameter and a plastic bag large enough to enclose the dish and be folded over is adequate.

6.7 *Plastic Limit:*

6.7.1 *Ground Glass Plate*—A ground glass plate of sufficient size for rolling plastic limit threads.

6.7.2 *Plastic Limit-Rolling Device (optional)*—A device made of acrylic conforming to the dimensions shown in Fig. 5.[3,4] The type of unglazed paper attached to the top and bottom plate (see 16.2.2) shall be such that it does not add foreign matter (fibers, paper fragments, etc.) to the soil during the rolling process.

6.8 *Spatula*—A spatula or pill knife having a blade about 2 cm (¾ in.) wide, and about 10 to 13 cm (3 to 4 in.) long.

6.9 *Sieve(s)*—A 200-mm (8-in.) diameter, 425-µm (No. 40) sieve conforming to the requirements of Specification E11 and having a rim at least 5 cm (2 in.) above the mesh. A 2.00-mm (No. 10) sieve meeting the same requirements may also be needed.

6.10 *Wash Bottle*, or similar container for adding controlled amounts of water to soil and washing fines from coarse particles.

6.11 *Drying Oven*, thermostatically controlled, preferably of the forced-draft type, capable of continuously maintaining a temperature of 110 ± 5°C (230 ± 9°F) throughout the drying chamber.

6.12 *Washing Pan*, round, flat-bottomed, at least 7.6 cm (3 in.) deep, and slightly larger at the bottom than a 20.3-cm (8-in.) diameter sieve.

7. Reagents and Materials

7.1 *Purity of Water*—Where distilled water is referred to in this test method, either distilled or demineralized water may be used. See Note 7 covering the use of tap water.

[3] The plastic limit-rolling device is covered by a patent (U.S. Patent No. 5,027,660).[7] Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible subcommittee, which you may attend.

[4] Bobrowski, L. J., Jr. and Griekspoor, D. M., "Determination of the Plastic Limit of a Soil by Means of a Rolling Device," *Geotechnical Testing Journal*, GTJODJ, Vol 15, No. 3, September 1992, pp. 284–287.

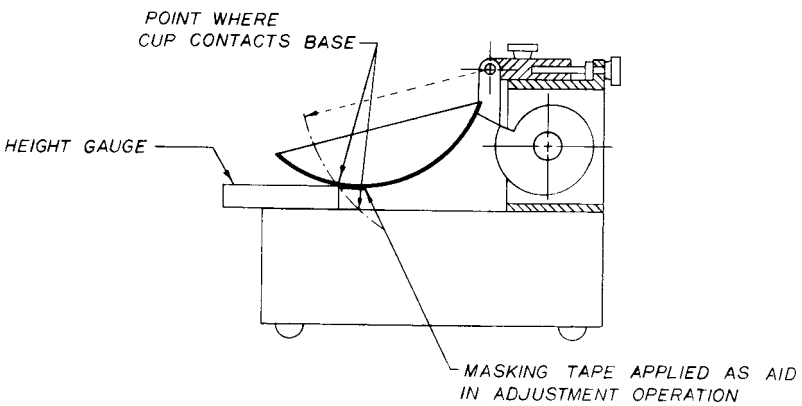

FIG. 2 Calibration for Height-of-Drop

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DIMENSIONS

LETTER	A △	B △	C △	D △	E △	F △
MM	2 ± 0.1	11 ± 0.2	40 ± 0.5	8 ± 0.1	50 ± 0.5	2 ± 0.1
LETTER	G	H	J	K △	L △	N
MM	10 MINIMUM	13	60	10 ±0.05	60 DEG ± 1 DEG	20

△ ESSENTIAL DIMENSIONS

□ BACK AT LEAST 15 MM FROM TIP

NOTE: DIMENSION A SHOULD BE 1.9–2.0 AND DIMENSION D SHOULD BE 8.0–8.1 WHEN NEW TO ALLOW FOR ADEQUATE SERVICE LIFE

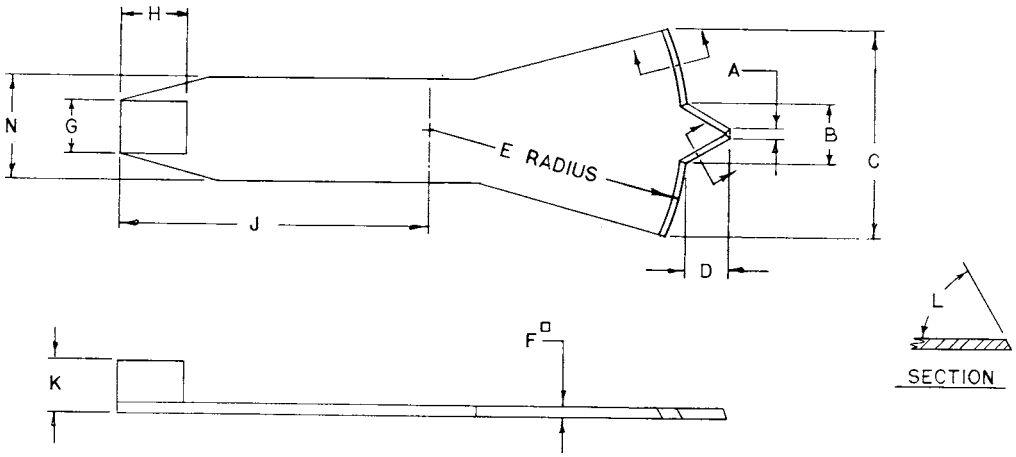
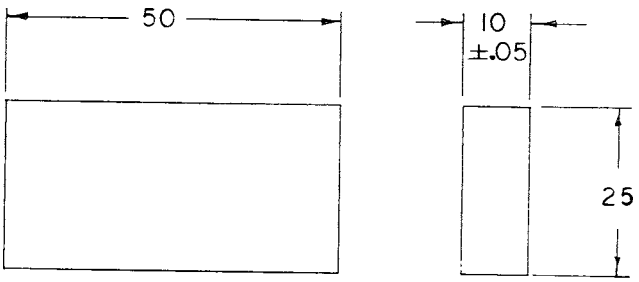


FIG. 3 Grooving Tool (Optional Height-of-Drop Gauge Attached)



DIMENSIONS IN MILLIMETRES

FIG. 4 Height-of-Drop Gauge

8. Sampling and Specimen

8.1 Samples may be taken from any location that satisfies testing needs. However, Practices C702, D75, and D420 should be used as guides for selecting and preserving samples from various types of sampling operations. Samples in which specimens will be prepared using the wet-preparation method (10.1) must be kept at their as–sampled water content prior to preparation.

8.1.1 Where sampling operations have preserved the natural stratification of a sample, the various strata must be kept separated and tests performed on the particular stratum of interest with as little contamination as possible from other strata. Where a mixture of materials will be used in construction, combine the various components in such proportions that the resultant sample represents the actual construction case.

8.1.2 Where data from these test methods are to be used for correlation with other laboratory or field test data, use the same material as used for those tests where possible.

8.2 *Specimen*—Obtain a representative portion from the total sample sufficient to provide 150 to 200 g of material passing the 425-µm (No. 40) sieve. Free flowing samples (materials) may be reduced by the methods of quartering or splitting. Non-free flowing or cohesive materials shall be mixed thoroughly in a pan with a spatula or scoop and a representative portion scooped from the total mass by making one or more sweeps with a scoop through the mixed mass.

9. Calibration of Apparatus

9.1 *Inspection of Wear:*

9.1.1 *Liquid Limit Device*—Determine that the liquid limit device is clean and in good working order. Check the following specific points.

9.1.1.1 *Wear of Base*—The spot on the base where the cup makes contact should be worn no greater than 10 mm (⅜ in.) in diameter. If the wear spot is greater than this, the base can be machined to remove the worn spot provided the resurfacing does not make the base thinner than specified in 6.1 and the other dimensional relationships are maintained.

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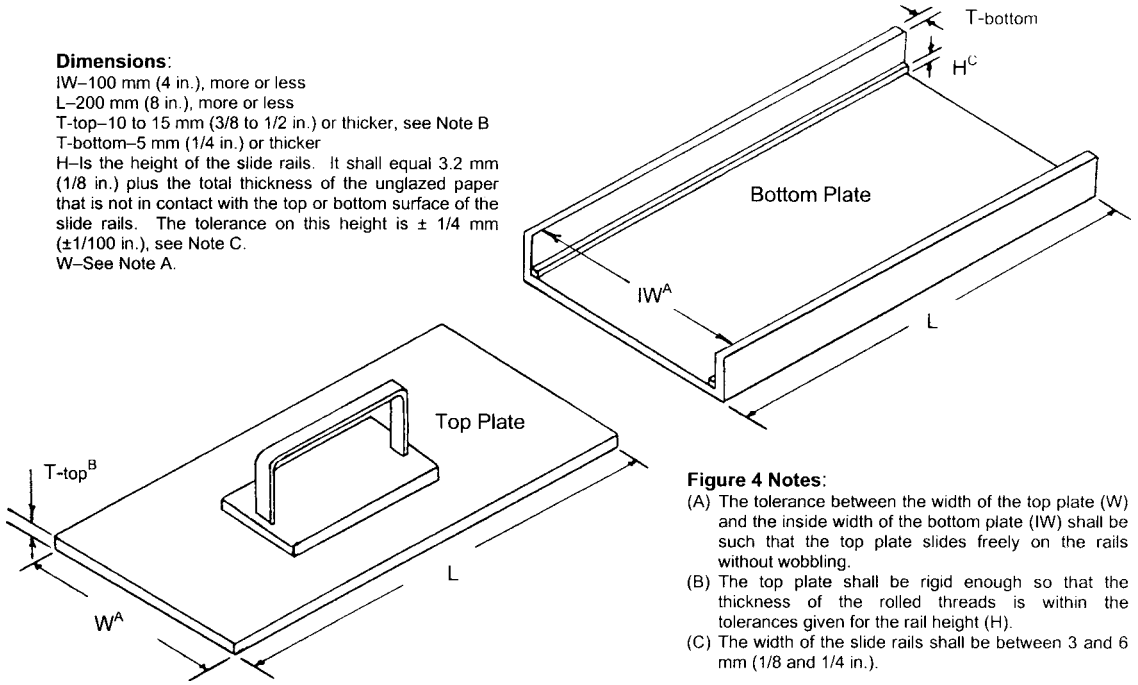

FIG. 5 Plastic Limit-Rolling Device

9.1.1.2 *Wear of Cup*—Replace the cup when the grooving tool has worn a depression in the cup 0.1 mm (0.004 in.) deep or when the rim of the cup has been reduced to half its original thickness. Verify that the cup is firmly attached to the cup hanger.

9.1.1.3 *Wear of Cup Hanger*—Verify that the cup hanger pivot does not bind and is not worn to an extent that allows more than 3 mm (⅛ in.) side-to-side movement of the lowest point on the rim.

9.1.1.4 *Wear of Cam*—The cam shall not be worn to an extent that the cup drops before the cup hanger (cam follower) loses contact with the cam.

9.1.1.5 *Rubber Feet*—The feet should prevent the base from bouncing or sliding on the work surface. Replace rubber feet that become hard, cracked, or brittle from age.

9.1.2 *Grooving Tools*—Inspect grooving tools for wear on a frequent and regular basis. The rapidity of wear depends on the material from which the tool is made, and the types of soils being tested. Soils containing a large proportion of fine sand particles may cause rapid wear of grooving tools; therefore, when testing these materials, tools should be inspected more frequently than for other soils.

NOTE 4—The width of the tip of grooving tools is conveniently checked using a pocket-sized measuring magnifier equipped with a millimeter scale. Magnifiers of this type are available from most laboratory supply companies. The depth of the tip of grooving tools can be checked using the depth-measuring feature of vernier calipers.

9.2 *Adjustment of Height-of-Drop*—Adjust the height-of-drop of the cup so that the point on the cup that comes in contact with the base rises to a height of 10 ± 0.2 mm. See Fig. 2 for proper location of the gauge relative to the cup during adjustment.

NOTE 5—A convenient procedure for adjusting the height-of-drop is as follows: place a piece of masking tape across the outside bottom of the cup parallel with the axis of the cup hanger pivot. The edge of the tape away from the cup hanger should bisect the spot on the cup that contacts the base. For new cups, placing a piece of carbon paper on the base and allowing the cup to drop several times will mark the contact spot. Attach the cup to the device and turn the crank until the cup is raised to its maximum height. Slide the height gauge under the cup from the front, and observe whether the gauge contacts the cup or the tape. (See Fig. 2.) If the tape and cup are both simultaneously contacted, the height-of-drop is ready to be checked. If not, adjust the cup until simultaneous contact is made. Check adjustment by turning the crank at 2 revolutions per second while holding the gauge in position against the tape and cup. If a faint ringing or clicking sound is heard without the cup rising from the gauge, the adjustment is correct. If no ringing is heard or if the cup rises from the gauge, readjust the height-of-drop. If the cup rocks on the gauge during this checking operation, the cam follower pivot is excessively worn and the worn parts should be replaced. Always remove tape after completion of adjustment operation.

10. Preparation of Test Specimen

10.1 *Wet Preparation Method*—Except where the dry method of specimen preparation is specified (10.2), prepare the specimen for testing as described in the following sections.

10.1.1 *Material Passes the 425-µm (No. 40) Sieve:*

10.1.1.1 Determine by visual and manual methods that the specimen from 8.2 has little or no material retained on a 425-µm (No. 40) sieve. If this is the case, prepare 150 to 200 g of material by mixing thoroughly with distilled or demineralized water on the glass plate or mixing dish using the spatula. If desired, soak the material in a mixing/storage dish with a small amount of water to soften the material before the start of mixing. If using Method A, adjust the water content of the material to bring it to a consistency that would require about 25 to 35 blows of the liquid limit device to close the groove (Note 6). For Method B, the number of blows should be between about 20 and 30 blows.

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10.1.1.2 If, during mixing, a small percentage of material is encountered that would be retained on a 425-µm (No. 40) sieve, remove these particles by hand (if possible). If it is impractical to remove the coarser material by hand, remove small percentages (less than about 15 %) of coarser material by working the material (having the above consistency) through a 425-µm sieve. During this procedure, use a piece of rubber sheeting, rubber stopper, or other convenient device provided the procedure does not distort the sieve or degrade material that would be retained if the washing method described in 10.1.2 were used. If larger percentages of coarse material are encountered during mixing, or it is considered impractical to remove the coarser material by the procedures just described, wash the sample as described in 10.1.2. When the coarse particles found during mixing are concretions, shells, or other fragile particles, do not crush these particles to make them pass a 425-µm sieve, but remove by hand or by washing.

10.1.1.3 Place the prepared material in the mixing/storage dish, check its consistency (adjust if required), cover to prevent loss of moisture, and allow to stand (cure) for at least 16 h (overnight). After the standing period and immediately before starting the test, thoroughly remix the soil.

NOTE 6—The time taken to adequately mix a soil will vary greatly, depending on the plasticity and initial water content. Initial mixing times of more than 30 min may be needed for stiff, fat clays.

10.1.2 *Material Containing Particles Retained on a 425-µm (No. 40) Sieve:*

10.1.2.1 Place the specimen (see 8.2) in a pan or dish and add sufficient water to cover the material. Allow the material to soak until all lumps have softened and the fines no longer adhere to the surfaces of the coarse particles (Note 7).

NOTE 7—In some cases, the cations of salts present in tap water will exchange with the natural cations in the soil and significantly alter the test results if tap water is used in the soaking and washing operations. Unless it is known that such cations are not present in the tap water, distilled or demineralized water should be used. As a general rule, water containing more than 100 mg/L of dissolved solids should not be used for either the soaking or washing operations.

10.1.2.2 When the material contains a large percentage of particles retained on the 425-µm (No. 40) sieve, perform the following washing operation in increments, washing no more than 0.5 kg (1 lb) of material at one time. Place the 425-µm sieve in the bottom of the clean pan. Transfer, without any loss of material, the soil-water mixture onto the sieve. If gravel or coarse sand particles are present, rinse as many of these as possible with small quantities of water from a wash bottle, and discard. Alternatively, transfer the soil-water mixture over a 2.00-mm (No. 10) sieve nested atop the 425-µm sieve, rinse the fine material through and remove the 2.00-mm sieve. After washing and removing as much of the coarser material as possible, add sufficient water to the pan to bring the level to about 13 mm (½ in.) above the surface of the 425-µm sieve. Agitate the slurry by stirring with the fingers while raising and lowering the sieve in the pan and swirling the suspension so that fine material is washed from the coarser particles. Disaggregate fine soil lumps that have not slaked by gently rubbing them over the sieve with the fingertips. Complete the washing operation by raising the sieve above the water surface and rinsing the material retained with a small amount of clean water. Discard material retained on the 425-µm sieve.

10.1.2.3 Reduce the water content of the material passing the 425–µm (No. 40) sieve until it approaches the liquid limit. Reduction of water content may be accomplished by one or a combination of the following methods: (*a*) exposing to air currents at room temperature, (*b*) exposing to warm air currents from a source such as an electric hair dryer, (*c*) decanting clear water from surface of the suspension, (*d*) filtering in a Büchner funnel or using filter candles, or (*e*) draining in a colander or plaster of Paris dish lined with high retentivity,[5] high wet-strength filter paper. If a plaster of Paris dish is used, take care that the dish never becomes sufficiently saturated that it fails to absorb water into its surface. Thoroughly dry dish between uses. During evaporation and cooling, stir the material often enough to prevent over-drying of the fringes and soil pinnacles on the surface of the mixture. For materials containing soluble salts, use a method of water reduction (*a* or *b*) that will not eliminate the soluble salts from the test specimen.

10.1.2.4 If applicable, remove the material retained on the filter paper. Thoroughly mix this material or the above material on the glass plate or in the mixing dish using the spatula. Adjust the water content of the mixture, if necessary, by adding small increments of distilled or demineralized water or by allowing the mixture to dry at room temperature while mixing on the glass plate. If using Method A, the material should be at a water content that would require about 25 to 35 blows of the liquid limit device to close the groove. For Method B, the number of blows should be between about 20 and 30. Put, if necessary, the mixed material in the storage dish, cover to prevent loss of moisture, and allow to stand (cure) for at least 16 h. After the standing period and immediately before starting the test, thoroughly remix the specimen.

10.2 *Dry Preparation Method:*

10.2.1 Dry the specimen from 8.2 at room temperature or in an oven at a temperature not exceeding 60°C until the soil clods will pulverize readily. Disaggregation is expedited if the material is not allowed to completely dry. However, the material should have a dry appearance when pulverized.

10.2.2 Pulverize the material in a mortar with a rubber-tipped pestle or in some other way that does not cause breakdown of individual particles. When the coarse particles found during pulverization are concretions, shells, or other fragile particles, do not crush these particles to make them pass a 425-µm (No. 40) sieve, but remove by hand or other suitable means, such as washing. If a washing procedure is used, follow 10.1.2.1-10.1.2.4.

10.2.3 Separate the material on a 425-µm (No. 40) sieve, shaking the sieve by hand to assure thorough separation of the finer fraction. Return the material retained on the 425-µm sieve to the pulverizing apparatus and repeat the pulverizing and sieving operations. Stop this procedure when most of the fine material has been disaggregated and material retained on the 425-µm sieve consists of individual particles.

[5] S and S 595 filter paper available in 320-mm circles has proven 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.

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10.2.4 Place material retained on the 425-µm (No. 40) sieve after the final pulverizing operations in a dish and soak in a small amount of water. Stir this mixture and transfer it to a 425-µm sieve, catching the water and any suspended fines in the washing pan. Pour this suspension into a dish containing the dry soil previously sieved through the 425-µm sieve. Discard material retained on the 425-µm sieve.

10.2.5 Proceed as described in 10.1.2.3 and 10.1.2.4.

MULTIPOINT LIQUID LIMIT—METHOD A

11. Procedure

11.1 Thoroughly remix the specimen (soil) in its mixing dish, and, if necessary, adjust its water content until the consistency requires about 25 to 35 blows of the liquid limit device to close the groove. Using a spatula, place a portion(s) of the prepared soil in the cup of the liquid limit device at the point where the cup rests on the base, squeeze it down, and spread it into the cup to a depth of about 10 mm at its deepest point, tapering to form an approximately horizontal surface. Take care to eliminate air bubbles from the soil pat, but form the pat with as few strokes as possible. Keep the unused soil in the mixing/storage dish. Cover the dish with a wet towel (or use other means) to retain the moisture in the soil.

11.2 Form a groove in the soil pat by drawing the tool, beveled edge forward, through the soil on a line joining the highest point to the lowest point on the rim of the cup. When cutting the groove, hold the grooving tool against the surface of the cup and draw in an arc, maintaining the tool perpendicular to the surface of the cup throughout its movement. See Fig. 6. In soils where a groove cannot be made in one stroke without tearing the soil, cut the groove with several strokes of the grooving tool. Alternatively, cut the groove to slightly less than required dimensions with a spatula and use the grooving tool to bring the groove to final dimensions. Exercise extreme care to prevent sliding the soil pat relative to the surface of the cup.

11.3 Verify that no crumbs of soil are present on the base or the underside of the cup. Lift and drop the cup by turning the crank at a rate of 1.9 to 2.1 drops per second until the two halves of the soil pat come in contact at the bottom of the groove along a distance of 13 mm (½ in.). See Fig. 7 and Fig. 8. The base of the machine shall not be held with the hand, or hands, while the crank is turned.

NOTE 8—Use of a scale is recommended to verify that the groove has closed 13 mm (½ in.).

11.4 Verify that an air bubble has not caused premature closing of the groove by observing that both sides of the groove have flowed together with approximately the same shape. If a bubble has caused premature closing of the groove, reform the soil in the cup, adding a small amount of soil to make up for that lost in the grooving operation and repeat 11.1-11.3. If the soil slides on the surface of the cup, repeat 11.1-11.3 at a higher water content. If, after several trials at successively higher water contents, the soil pat continues to slide in the cup or if the number of blows required to close the groove is always less than 25, record that the liquid limit could not be determined, and report the soil as nonplastic without performing the plastic limit test.

11.5 Record the number of drops, *N*, required to close the groove. Remove a slice of soil approximately the width of the spatula, extending from edge to edge of the soil cake at right

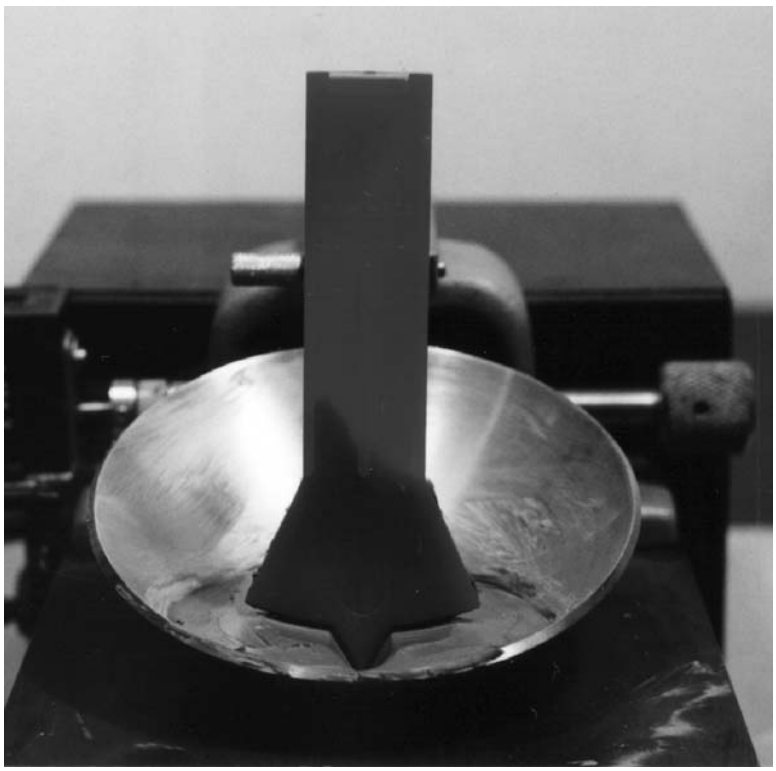

FIG. 6 Example of Grooving Tool Placed in a Properly Grooved Soil Pat

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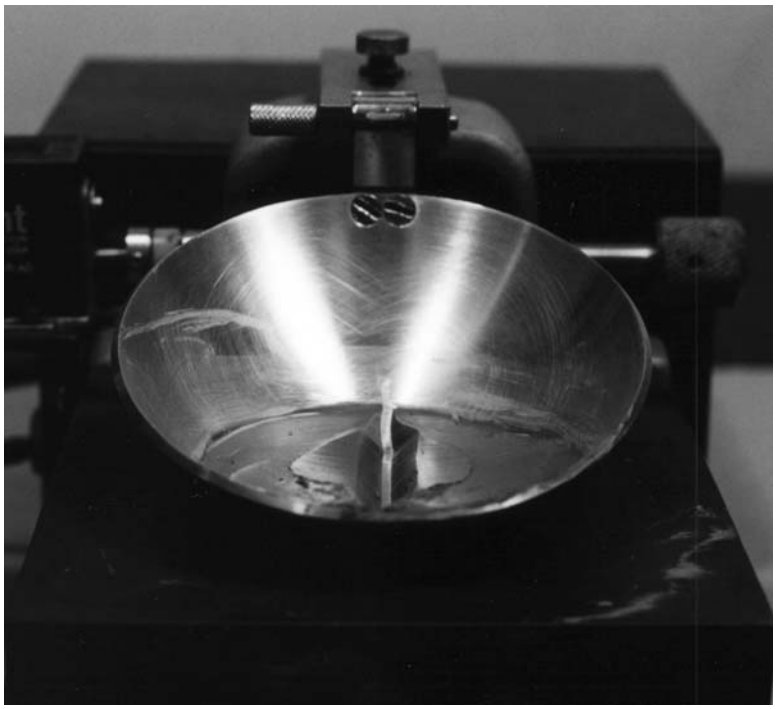

FIG. 7 Grooved Soil Pat in Liquid Limit Device

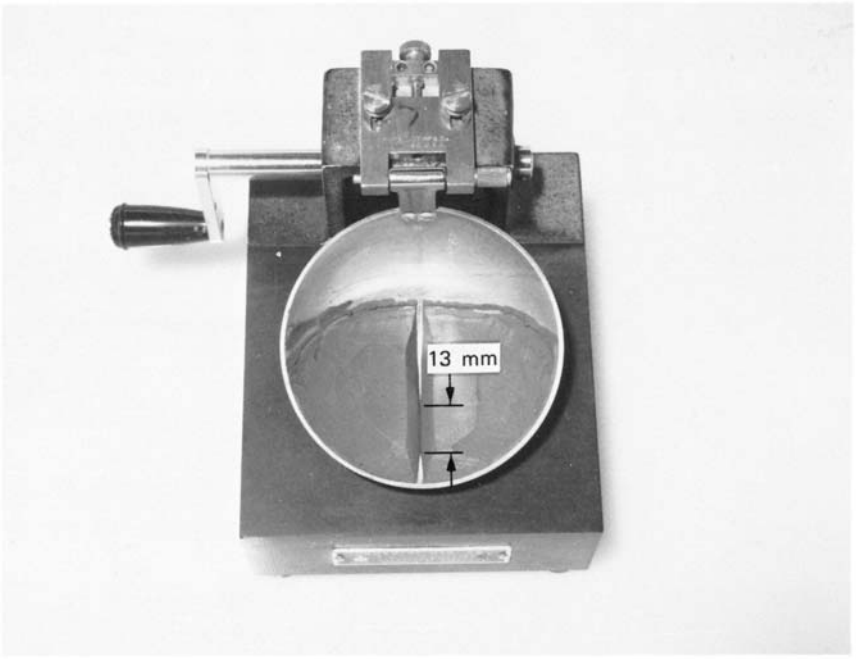

FIG. 8 Soil Pat After Groove Has Closed

angles to the groove and including that portion of the groove in which the soil flowed together, place in a container of known mass, and cover.

11.6 Return the soil remaining in the cup to the dish. Wash and dry the cup and grooving tool and reattach the cup to the carriage in preparation for the next trial.

11.7 Remix the entire soil specimen in the dish adding distilled water to increase the water content of the soil and decrease the number of blows required to close the groove. Repeat 11.1-11.6 for at least two additional trials producing successively lower numbers of blows to close the groove. One of the trials shall be for a closure requiring 25 to 35 blows, one for closure between 20 and 30 blows, and one trial for a closure requiring 15 to 25 blows.

11.8 Determine the water content, W^n, of the soil specimen from each trial in accordance with Test Method D2216.

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11.8.1 Determination of initial masses (container plus moist soil) should be performed immediately after completion of the test. If the test is to be interrupted for more than about 15 minutes, determine the mass of the water content specimens already obtained at the time of the interruption.

12. Calculation

12.1 Plot the relationship between the water content, W^n, and the corresponding number of drops, N, of the cup on a semilogarithmic graph with the water content as ordinates on the arithmetical scale, and the number of drops as abscissas on a logarithmic scale. Draw the best straight line through the three or more plotted points.

12.2 Take the water content corresponding to the intersection of the line with the 25-drop abscissa as the liquid limit of the soil and round to the nearest whole number. Computational methods may be substituted for the graphical method for fitting a straight line to the data and determining the liquid limit.

ONE-POINT LIQUID LIMIT—METHOD B

13. Procedure

13.1 Proceed as described in 11.1-11.5 except that the number of blows required to close the groove shall be 20 to 30. If less than 20 or more than 30 blows are required, adjust the water content of the soil and repeat the procedure.

13.2 Immediately after removing a water content specimen as described in 11.5, reform the soil in the cup, adding a small amount of soil to make up for that lost in the grooving and water content sampling processes.

13.2.1 As an alternative to reforming the soil in the brass cup after removing the water content specimen, the soil remaining in the cup can be removed from the cup, remixed with the soil in the mixing container and a new specimen placed in the cup as described in 11.1.

13.3 Repeat 11.2-11.5

13.4 If the second closing of the groove requires the same number of drops or no more than two drops difference, secure another water content specimen. If the difference of the number of drops between the first and second closings of the groove is greater than two, remix the entire specimen and repeat the procedure, beginning at 13.1, until two successive closures having the same number of drops or no more than two drops difference are obtained.

NOTE 9—Excessive drying or inadequate mixing will cause the number of blows to vary.

13.5 Determine water contents of the two specimens in accordance with 11.8.

14. Calculation

14.1 Determine the liquid limit for each water content specimen using one of the following equations:

$$LL^n = W^n \cdot \left(\frac{N}{25}\right)^{0.121}$$

or

$$LL^n = k \cdot W^n$$

where:

LL^n = one point liquid limit for given trial, %,
N = number of blows causing closure of the groove for given trial,
W^n = water content for given trial, %, and
k = factor given in Table 1.

14.1.1 The liquid limit, LL, is the average of the two trial liquid-limit values, to the nearest whole number (without the percent designation).

14.2 If the difference between the two trial liquid-limit values is greater than one percentage point, repeat the test as described in 13.1 through 14.1.1.

PLASTIC LIMIT

15. Preparation of Test Specimen

15.1 Select a 20-g or more portion of soil from the material prepared for the liquid limit test; either, after the second mixing before the test, or from the soil remaining after completion of the liquid limit test. Reduce the water content of the soil to a consistency at which it can be rolled without sticking to the hands by spreading or mixing continuously on the glass plate or in the mixing/storage dish. The drying process may be accelerated by exposing the soil to the air current from an electric fan, or by blotting with paper, that does not add any fiber to the soil. Paper such as hard surface paper toweling or high wet-strength filter paper is adequate.

16. Procedure

16.1 From this plastic-limit specimen, select a 1.5 to 2.0 g portion. Form the selected portion into an ellipsoidal mass.

16.2 Roll the soil mass by one of the following methods (hand or rolling device):

16.2.1 *Hand Method*—Roll the mass between the palm or fingers and the ground-glass plate with just sufficient pressure to roll the mass into a thread of uniform diameter throughout its length (see Note 10). The thread shall be further deformed on each stroke so that its diameter reaches 3.2 mm (⅛ in.), taking no more than 2 min (see Note 11). The amount of hand or finger pressure required will vary greatly according to the soil being tested, that is, the required pressure typically increases with increasing plasticity. Fragile soils of low plasticity are best rolled under the outer edge of the palm or at the base of the thumb.

TABLE 1 Factors for Obtaining Liquid Limit from Water Content and Number of Drops Causing Closure of Groove

N (Number of Drops)	k (Factor for Liquid Limit)
20	0.973
21	0.979
22	0.985
23	0.990
24	0.995
25	1.000
26	1.005
27	1.009
28	1.014
29	1.018
30	1.022

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NOTE 10—A normal rate of rolling for most soils should be 80 to 90 strokes per minute, counting a stroke as one complete motion of the hand forward and back to the starting position. This rate of rolling may have to be decreased for very fragile soils.

NOTE 11—A 3.2-mm (⅛-in.) diameter rod or tube is useful for frequent comparison with the soil thread to ascertain when the thread has reached the proper diameter.

16.2.2 *Rolling Device Method*—Attach smooth unglazed paper to both the top and bottom plates of the plastic limit-rolling device. Place the soil mass on the bottom plate at the midpoint between the slide rails. Place the top plate in contact with the soil mass(es). Simultaneously apply a slight downward force and back and forth motion to the top plate so that the top plate comes into contact with the side rails within 2 min (see Notes 10 and 12). During this rolling process, the end(s) the soil thread(s) shall not contact the side rail(s). If this occurs, roll a smaller mass of soil (even if it is less than that mentioned in Section 16.1).

NOTE 12—In most cases, two soil masses (threads) can be rolled simultaneously in the plastic limit-rolling device.

16.3 When the diameter of the thread becomes 3.2 mm, break the thread into several pieces. Squeeze the pieces together, knead between the thumb and first finger of each hand, reform into an ellipsoidal mass, and re-roll. Continue this alternate rolling to a thread 3.2 mm in diameter, gathering together, kneading and re-rolling, until the thread crumbles under the pressure required for rolling and the soil can no longer be rolled into a 3.2-mm diameter thread (see Fig. 9). It has no significance if the thread breaks into threads of shorter length. Roll each of these shorter threads to 3.2 mm in diameter. The only requirement for continuing the test is that these threads can be reformed into an ellipsoidal mass and rolled out again. The operator shall at no time attempt to produce failure at exactly 3.2-mm diameter by allowing the thread to reach 3.2 mm, then reducing the rate of rolling or the hand pressure, or both, while continuing the rolling without further deformation until the thread falls apart. It is permissible, however, to reduce the total amount of deformation for feebly plastic soils by making the initial diameter of the ellipsoidal mass nearer to the required 3.2-mm final diameter. If crumbling occurs when the thread has a diameter greater than 3.2 mm, this shall be considered a satisfactory end point, provided the soil has been previously rolled into a thread 3.2 mm in diameter. Crumbling of the thread will manifest itself differently with the various types of soil. Some soils fall apart in numerous small aggregations of particles, others may form an outside tubular layer that starts splitting at both ends. The splitting progresses toward the middle, and finally, the thread falls apart in many small platy particles. Fat clay soils require much pressure to deform the thread, particularly as they approach the plastic limit. With these soils, the thread breaks into a series of barrel-shaped segments about 3.2 to 9.5 mm (⅛ to ⅜ in.) in length.

16.4 Gather the portions of the crumbled thread together and place in a container of known mass. Immediately cover the container.

16.5 Select another 1.5 to 2.0-g portion of soil from the plastic–limit specimen and repeat the operations described in 16.1 and 16.2 until the container has at least 6 g of soil.

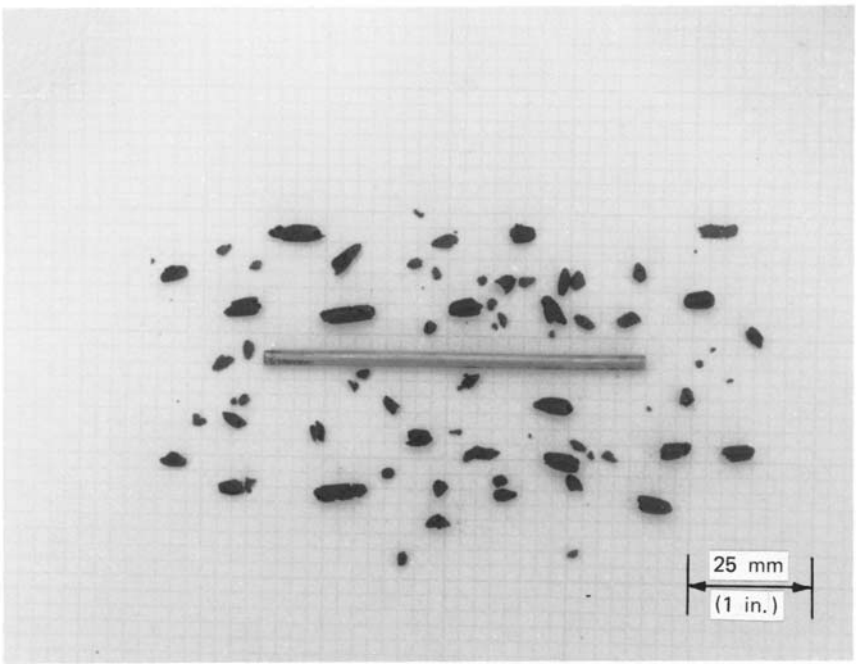

FIG. 9 Lean Clay Soil at the Plastic Limit

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16.6 Repeat 16.1-16.5 to make another container holding at least 6 g of soil. Determine the water content of the soil contained in the containers in accordance with Test Method D2216. See 11.8.1.

17. Calculation

17.1 Compute the average of the two water contents (trial plastic limits) and round to the nearest whole number. This value is the plastic limit, *PL*. Repeat the test if the difference between the two trial plastic limits is greater than the acceptable range for two results listed in Table 2 for single-operator precision, that is, 1.4 percentage points; i.e., (2.8 × 0.5).

PLASTICITY INDEX

18. Calculation

18.1 Calculate the plasticity index as follows:

$$PI = LL - PL$$

where:
LL = liquid limit (whole number), and
PL = plastic limit (whole number).

18.1.1 Both LL and PL are whole numbers. If either the liquid limit or plastic limit could not be determined, or if the plastic limit is equal to or greater than the liquid limit, report the soil as nonplastic, NP.

19. Report: Test Data Sheet(s)/Form(s)

19.1 The terminology used to specify how data are recorded on the test data sheet(s)/form(s), as given below, is covered in 1.9.

19.2 Record as a minimum the following information:

19.2.1 Sample/specimen identifying information, such as project name , project number, boring number, depth (m or ft).

19.2.2 Description of sample, such as approximate maximum grain size, estimate of the percentage of sample retained on the 425-µm (No. 40) sieve, as-received water content.

19.2.3 Details of specimen preparation, such as wet or dry (air-dried or oven-dried), method of removing particles larger than the 425-µm (No. 40) sieve.

19.2.4 Any special specimen selection process used, such as removal of sand lenses from an intact (undisturbed) sample.

19.2.5 Equipment used, such as hand rolled or mechanical rolling device for plastic limit, manual or mechanical liquid limit device, metal or plastic grooving tool.

19.2.6 Liquid limit, plastic limit, and plasticity index to the nearest whole number, omitting the percent designation. If the liquid limit or plastic limit tests could not be performed, or if the plastic limit is equal to or greater than the liquid limit, report the soil as nonplastic, NP.

19.2.7 Procedure by which liquid limit was performed, if it differs from the multipoint method.

20. Precision and Bias

20.1 *Precision*—Criteria for judging the acceptability of test results obtained by these test methods on a range of soil types are given in Tables 2 and 3. In performing these test methods, Method A and the Wet Preparation Method (except soil was air-dried) were used.

20.1.1 These estimates of precision are based on the results of the interlaboratory program conducted by the ASTM Reference Soils and Testing Program.[6] 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 20.1.5. The precision estimates vary with soil type and method(s) used. Judgment is required when applying these estimates to another soil and method used (Method A or B, or Wet or Dry Preparation Method).

20.1.2 The data in Table 2 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 2, Column 4, were obtained in accordance with Practice E691, which recommends each testing laboratory perform a minimum of three replicate tests. Results of two properly conducted tests performed by the same operator on

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D18-1013.

TABLE 2 Summary of Test Results from Triplicate Test Laboratories (Atterberg Limits)

(1) Soil Type	(2) Number of Triplicate Test Laboratories			(3) Average Value[A] (Percentage Points)			(4) Standard Deviation[B] (Percentage Points)			(5) Acceptable Range of Two Results[C] (Percentage Points)		
	Type Test											
	LL	PL	PI	LL	PL	PI	LL	PL	PI	LL	PL	PI
	Single-Operator Results (Within-Laboratory Repeatability)											
CH	13	13	13	59.8	20.6	39.2	0.7	0.5	0.8	2	1	2
CL	14	13	13	33.4	19.9	13.6	0.3	0.4	0.5	1	1	1
ML	12	11	11	27.4	23.4[D]	4.1[D]	0.5	0.3	0.6	2	1	2
	Multilaboratory Results (Between-Laboratory Reproducibility)											
CH	13	13	13	59.8	20.6	39.2	1.3	2.0	2.5	4	6	7
CL	14	13	13	33.4	19.9	13.6	1.0	1.2	1.7	3	3	5
ML	12	11	11	27.4	23.4[D]	4.1[D]	1.3	0.9	1.9	4	3	5

[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 can not 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 $1.960 \cdot \sqrt{2} \cdot 1s$, 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 this test method 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.
[D] For the ML soil, 2 out of 14 triplicate test laboratories reported the soil as nonplastic.

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TABLE 3 Summary of Single-Test Result from Each Laboratory (Atterberg Limits)[A]

(1)	(2)	(3)			(4)			(5)		
Soil Type	Number of Test Laboratories	Average Value (Percentage Points)			Standard Deviation (Percentage Points)			Acceptable Range of Two Results (Percentage Points)		
		Type Test								
		LL	PL	PI	LL	PL	PI	LL	PL	PI
CH	24	59.9	20.4	39.5	2.1	2.7	3.1	6	7	9
CL	24	33.3	19.9	13.4	0.8	1.3	1.6	2	4	4
ML	18	27.1	23.2[B]	3.9[B]	1.3	1.2	1.8	4	3	5

[A] For column footnotes, see Table 3.
[B] For the ML soil, 6 out of 24 laboratories reported the soil as nonplastic.

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 2, Column 5. For definition of d2*s* see Footnote C in Table 2. 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 2, Column 5.

20.1.3 In the ASTM Reference Soils and Testing Program, many of the laboratories performed only a single test on each soil type. This is common practice in the design and construction industry. The data for each soil type in Table 3 are based upon the first test results 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 3, Column 5. The results in Table 2 and Table 3 are dissimilar because the data sets are different.

20.1.4 Table 2 presents a rigorous interpretation of triplicate test data in accordance with Practice E691 from pre-qualified laboratories. Table 3 is derived from test data that represents common practice.

20.1.5 *Soil Types*—Based on the multilaboratory test results, the soils used in the program are described below in accordance with Practice D2487. In addition, the local names of the soils are 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

20.2 *Bias*—There is no acceptable reference value for these test methods; therefore, bias cannot be determined.

21. Keywords

21.1 activity; Atterberg limits; liquid limit; plasticity index; plastic limit

ANNEX

(Mandatory Information)

A1. Resilience Tester

A1.1 A device for measuring the resilience of liquid limit device bases is shown in Fig. A1.1. The device consists of a clear acrylic plastic tube and cap, a 5⁄16-in. diameter steel ball, and a small bar magnet. The cylinder may be cemented to the cap or threaded as shown. The small bar magnet is held in the recess of the cap and the steel ball is fixed into the recess in the underside of the cap with the bar magnet. The cylinder is then turned upright and placed on the top surface of the base to be tested. Holding the tube lightly against the liquid limit device base with one hand, release the ball by pulling the magnet out of the cap. Use the scale markings on the outside of the cylinder to determine the highest point reached by the bottom of the ball. Repeat the drop at least three times, placing the tester in a different location for each drop. Tests should be conducted at room temperature.

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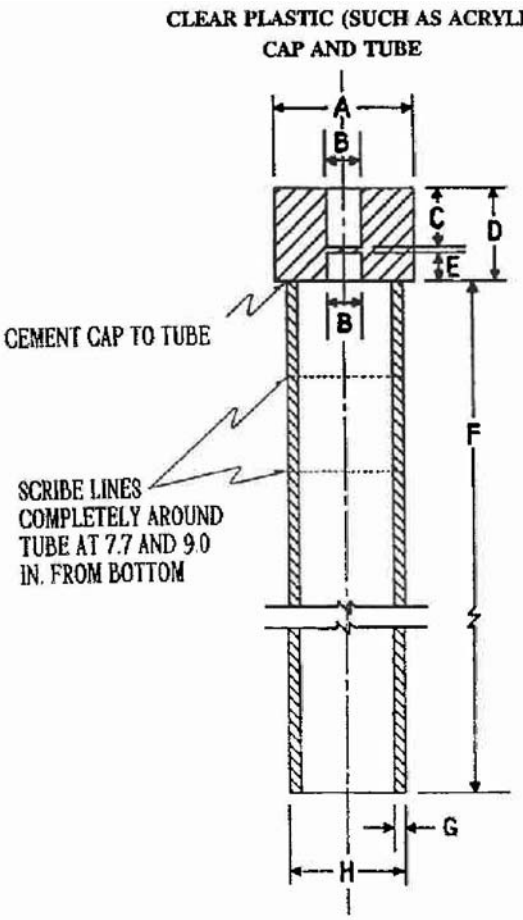

TABLE OF MEASUREMENTS

DIMENSION	DESCRIPTION	ENGLISH, in.	SI, mm
A	DIAM. OF CAP	1 1/2	38.10
B	DIAM. OF HOLE	3/8	9.52
C	DEPTH OF HOLE	10/16	15.88
D	HEIGHT OF CAP	1	25.40
E	DEPTH OF HOLE	5/16	7.94
F	LENGTH OF TUBE	10	254.00
G	WALL THICKNESS	1/8	3.18
H	O.D. OF TUBE	1 1/4	31.75

FIG. A1.1 Resilience Tester

APPENDIX

X1. Sample Data Sheet

X1.1 See Fig. X1.1.

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Laboratory Name: Example | Project Name: Example | Project Number: Example
Soil Type: CH-1 | Boring Number: N/A | Depth (m or ft): N/A
Initial Visual Description: CH, brown, highly plastic clay
Sample Number: N/A

INITIAL VISUAL USCS GROUP SYMBOL:	CH

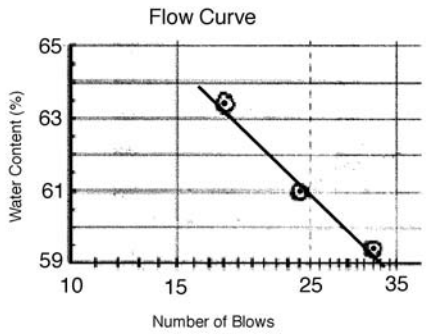
SPECIMEN PREPARATION			
Wet:	X	Washed on #40 Sieve	
Dry (Air):		Dry Sieved on #40 Sieve	
Dry (Oven):		Mechanically Pushed Through #40 Sieve	
Mixed on Glass Plate and Removed Medium Plus Sand Particles			X
Mixing Water: Distilled	X	Demineralized:	Other:

TESTING EQUIPMENT USED		
Plastic Limit:	Hand Rolled	X
	Mechanical Rolling Device	
Liquid Limit:	Manual	X
Apparatus No. ( )	Mechanical	
Casagrande/ASTM	Metal	
Grooving Tool:	Plastic	X

AS-RECEIVED WATER CONTENT (OVEN DRIED)

Container No.	example	example	
Mass Moist Soil + Container, M1 (g)	n/a	n/a	
Mass Dry Soil + Container, M2 (g)			
Mass Container, M3 (g)			Average
WATER CONTENT, w, (%)			

Circle Approximate Max. Grain Size in Sample 3" 1-1/2" 3/4" 3/8" #4 #10 <#10



PLASTIC LIMIT

Container No.	151	s-110	
Mass Moist Soil + Container, M1 (g)	24.44	23.75	
Mass Dry Soil + Container, M2 (g)	22.96	22.13	
Mass Container, M3 (g)	16.76	15.32	Average
WATER CONTENT, w, (%)	23.9	23.8	23.9

LIQUID LIMIT

Container No.	147	232	152	
Mass Moist Soil + Container, M1 (g)	35.24	37.79	35.88	
Mass Dry Soil + Container, M2 (g)	28.52	30.50	28.46	
Mass Container, M3 (g)	17.19	18.55	16.75	
WATER CONTENT, w, (%)	59.3	61.0	63.4	
NUMBER OF BLOWS, N	32	24	18	Average
LIQUID LIMIT, ASTM SINGLE POINT	n/a	n/a	n/a	n/a

LINEAR REGRESSION ANALYSIS	
w at N = 25 Blows	
Coef. Of Determination, r^2	

Recommended range of Blow Count for Multiple Point Method A: 15 to 25, 25 to 30, and 25 to 35
Recommended range of Blow Count for Single Point Method B: 20 to 30
wo or w = ((M1-M2)/(M2-M3))x100
LL = Water Content at N= 25 blows, from Flow Curve
LL by Single Point = w x (N/25)0.121
PI = LL - PL
LI = (wo-PL) / (LL - PL)

SUMMARY

TEST METHOD	A	X	B		Wet	X	Dry	
AS-RECEIVED WATER CONTENT, w, (%)								n/a
LIQUID LIMIT, LL								61
PLASTIC LIMIT, PL								24
PLASTICITY INDEX, PI								37
LIQUIDITY INDEX, LI								n/a
PERCENTAGE POINTS ABOVE/BELOW A-LINE								n/a
PLASTICITY CHART CLASSIFICATIOIN								CH

PREPARED BY: _____ DATE: _____ DRY MASS BY: _____ CHECKED BY: _____
TESTED BY: _____ DATE: _____ CALCULATED BY: _____ SPOT CHECKED BY: _____
REVIEWED BY: _____

FIG. X1.1 Sample Data Sheet

SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (D4318 – 05) that may impact the use of this standard. (Approved January 15, 2010.)

(*1*) Corrected 1.6 to reference D4542 and added D4542 to Referenced Documents in Section 2.
(*2*) In 1.8 and 1.8.1, clarified use of SI units.
(*3*) Added 1.9 referencing D6026 and the use of significant digits and renumbered 1.9 as 1.10.
(*4*) In 6.1 and 6.1.1 reworded the requirements for the composition of the base and removed the word “rubber.” “Rubber” was also removed from the label in Fig. 1.
(*5*) In 6.1.2 removed the Durometer hardness requirement for the rubber feet.
(*6*) In 6.7.1 removed the dimensional requirements for the Ground Glass Plate.
(*7*) In 9.1.1.5 added guidance for replacement of rubber feet.
(*8*) In 11.1 changed “cup” to “dish” for consistency.
(*9*) In 11.3 added instruction that the base shall not be held during testing.
(*10*) In 13.2 to 13.5 clarified the instructions to allow two alternative test procedures.
(*11*) Section 19 was updated to comply with the D18.91 Special Memorandum on Report Section.

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Construction Material Testing Laboratory **SOP Number: SOP GT-007**
Terracon **Effective: June 1999**
Revision: August 2011

STANDARD OPERATING PROCEDURE
FOR
SOIL CLASSIFICATION - GRAIN SIZE ANALYSIS OF SOIL ASTM D422

STEP 1
Complete all project information on the soil classification data sheet.

STEP 2
Oven dry at least 1,500 grams or the appropriate weight depending on maximum particle size, obtained from the bulk or tube sample. Place sample into container for drying. Record the container's I.D. number on the soil classification data sheet, grain size section.

NOTE: All Hydrometers Samples Shall be Tested using the Wet Method, SOP-GT-07.2 , unless instructed otherwise, in which case, use SOP-GT-07.1.

STEP 3
After drying, record dry weight of sample minus any tare weight on worksheet. Place sample into Beaker or Plastic Bucket. Record container's Id on worksheet and cover sample with tap water in container. Allow sample to soak.

STEP 4
After soaking, agitate sample in container using a wire wisp. Wash thoroughly over a # 10 sieve or # 200 sieve depending on type of test.

NOTE: If Hydrometer is in progress, wash over # 10 sieve. If a grain-size with # 200 wash has been requested, wash sample over a # 200 sieve.

STEP 5
After washing, return sample to tare and place into over for drying. (110 C or 230 F). Record tare Id on worksheet.

STEP 6

After drying, seive the sample over a nest of sieves as: $1^{1}/_{2}$, 1, $^{3}/_{4}$,$^{1}/_{2}$, $^{3}/_{8}$, #4,#8, #10, #16, #30, #50, #100, #200 if a grain-size with wash # 200 has been requested).

Record the weights of the material retained on each sieve accumulatively. Once the total weight retained on the #10 or # 200, has been determined and recorded, place sieved sample into a labeled plastic bag. **DO NOT DISCARD SAMPLE**. Check the data sheet for completeness. Turn paperwork to the Data Reduction Basket.

Designation: D422 – 63 (Reapproved 2007)

Standard Test Method for Particle-Size Analysis of Soils[1]

This standard is issued under the fixed designation D422; 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 quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 µm (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 µm is determined by a sedimentation process, using a hydrometer to secure the necessary data (Note 1 and Note 2).

NOTE 1—Separation may be made on the No. 4 (4.75-mm), No. 40 (425-µm), or No. 200 (75-µm) sieve instead of the No. 10. For whatever sieve used, the size shall be indicated in the report.

NOTE 2—Two types of dispersion devices are provided: (*1*) a high-speed mechanical stirrer, and (*2*) air dispersion. Extensive investigations indicate that air-dispersion devices produce a more positive dispersion of plastic soils below the 20-µm size and appreciably less degradation on all sizes when used with sandy soils. Because of the definite advantages favoring air dispersion, its use is recommended. The results from the two types of devices differ in magnitude, depending upon soil type, leading to marked differences in particle size distribution, especially for sizes finer than 20 µm.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D421 Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E100 Specification for ASTM Hydrometers

2.2 *ASTM Adjuncts:*

Air-Jet Dispersion Cup for Grain-Size Analysis of Soil[3]

3. Apparatus

3.1 *Balances*—A balance sensitive to 0.01 g for weighing the material passing a No. 10 (2.00-mm) sieve, and a balance sensitive to 0.1 % of the mass of the sample to be weighed for weighing the material retained on a No. 10 sieve.

3.2 *Stirring Apparatus*—Either apparatus A or B may be used.

3.2.1 Apparatus A shall consist of a mechanically operated stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10 000 rpm without load. The shaft shall be equipped with a replaceable stirring paddle made of metal, plastic, or hard rubber, as shown in Fig. 1. The shaft shall be of such length that the stirring paddle will operate not less than ¾ in. (19.0 mm) nor more than 1½ in. (38.1 mm) above the bottom of the dispersion cup. A special dispersion cup conforming to either of the designs shown in Fig. 2 shall be provided to hold the sample while it is being dispersed.

3.2.2 Apparatus B shall consist of an air-jet dispersion cup (See drawing[3]) (Note 3) conforming to the general details shown in Fig. 3 (Note 4 and Note 5).

NOTE 3—The amount of air required by an air-jet dispersion cup is of the order of 2 ft^3/min; some small air compressors are not capable of supplying sufficient air to operate a cup.

NOTE 4—Another air-type dispersion device, known as a dispersion tube, developed by Chu and Davidson at Iowa State College, has been shown to give results equivalent to those secured by the air-jet dispersion cups. When it is used, soaking of the sample can be done in the sedimentation cylinder, thus eliminating the need for transferring the slurry. When the air-dispersion tube is used, it shall be so indicated in the report.

NOTE 5—Water may condense in air lines when not in use. This water must be removed, either by using a water trap on the air line, or by blowing the water out of the line before using any of the air for dispersion purposes.

3.3 *Hydrometer*—An ASTM hydrometer, graduated to read in either specific gravity of the suspension or grams per litre of suspension, and conforming to the requirements for hydrometers 151H or 152H in Specifications E100. Dimensions of both hydrometers are the same, the scale being the only item of difference.

3.4 *Sedimentation Cylinder*—A glass cylinder essentially 18 in. (457 mm) in height and 2½ in. (63.5 mm) in diameter, and marked for a volume of 1000 mL. The inside diameter shall be such that the 1000-mL mark is 36 ± 2 cm from the bottom on the inside.

3.5 *Thermometer*—A thermometer accurate to 1°F (0.5°C).

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[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 Oct. 15, 2007. Published October 2007. Originally approved in 1935. Last previous edition approved in 2002 as D422 – 63 (2002)$^{\varepsilon 1}$. DOI: 10.1520/D0422-63R07.

[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 ASTM International Headquarters. Order Adjunct No. ADJD0422.

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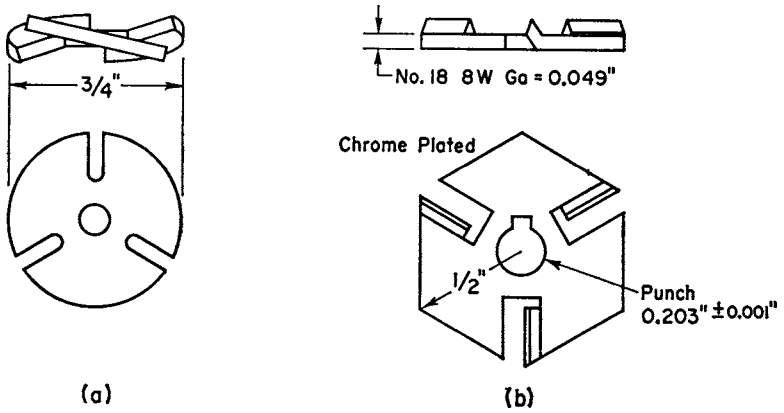
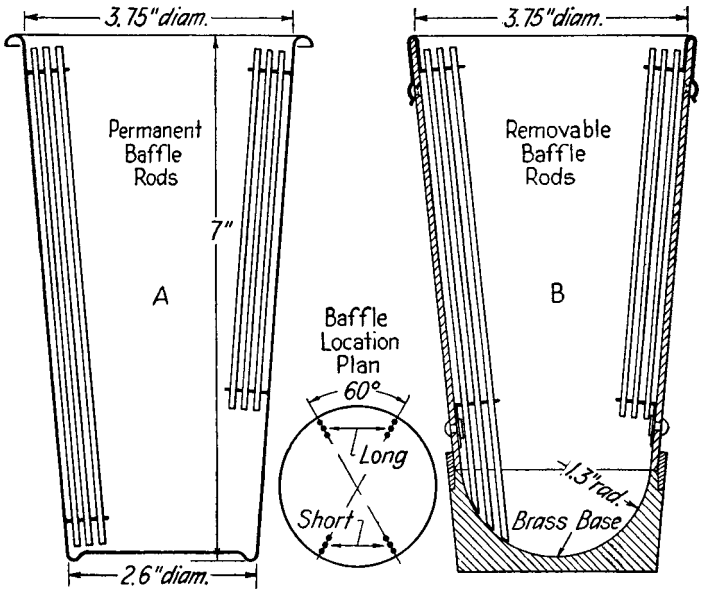


Metric Equivalents					
in.	0.001	0.049	0.203	½	¾
mm	0.03	1.24	5.16	12.7	19.0

FIG. 1 Detail of Stirring Paddles



Metric Equivalents			
in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus

3.6 *Sieves*—A series of sieves, of square-mesh woven-wire cloth, conforming to the requirements of Specification E11. A full set of sieves includes the following (Note 6):

3-in. (75-mm)
2-in. (50-mm)
1½-in. (37.5-mm)
1-in. (25.0-mm)
¾-in. (19.0-mm)
⅜-in. (9.5-mm)
No. 4 (4.75-mm)
No. 10 (2.00-mm)
No. 20 (850-µm)
No. 40 (425-µm)
No. 60 (250-µm)
No. 140 (106-µm)
No. 200 (75-µm)

NOTE 6—A set of sieves giving uniform spacing of points for the graph, as required in Section 17, may be used if desired. This set consists of the following sieves:

3-in. (75-mm)
1½-in. (37.5-mm)
¾-in. (19.0-mm)
⅜-in. (9.5-mm)
No. 4 (4.75-mm)
No. 8 (2.36-mm)
No. 16 (1.18-mm)
No. 30 (600-µm)
No. 50 (300-µm)
No. 100 (150-µm)
No. 200 (75-µm)

3.7 *Water Bath or Constant-Temperature Room*—A water bath or constant-temperature room for maintaining the soil suspension at a constant temperature during the hydrometer analysis. A satisfactory water tank is an insulated tank that maintains the temperature of the suspension at a convenient constant temperature at or near 68°F (20°C). Such a device is illustrated in Fig. 4. In cases where the work is performed in a room at an automatically controlled constant temperature, the water bath is not necessary.

3.8 *Beaker*—A beaker of 250-mL capacity.

3.9 *Timing Device*—A watch or clock with a second hand.

4. Dispersing Agent

4.1 A solution of sodium hexametaphosphate (sometimes called sodium metaphosphate) shall be used in distilled or demineralized water, at the rate of 40 g of sodium hexametaphosphate/litre of solution (Note 7).

NOTE 7—Solutions of this salt, if acidic, slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate. Bottles containing solutions should have the date of preparation marked on them.

4.2 All water used shall be either distilled or demineralized water. The water for a hydrometer test shall be brought to the temperature that is expected to prevail during the hydrometer test. For example, if the sedimentation cylinder is to be placed in the water bath, the distilled or demineralized water to be used shall be brought to the temperature of the controlled water bath; or, if the sedimentation cylinder is used in a room with controlled temperature, the water for the test shall be at the temperature of the room. The basic temperature for the

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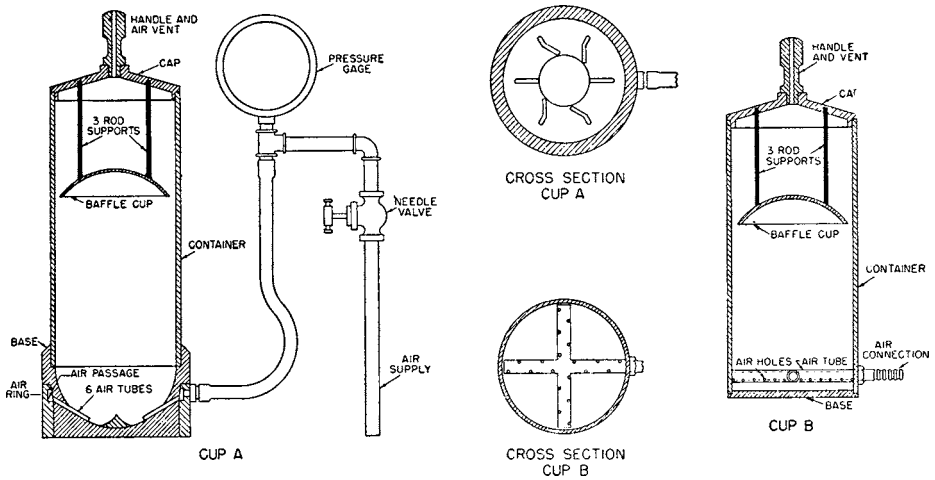
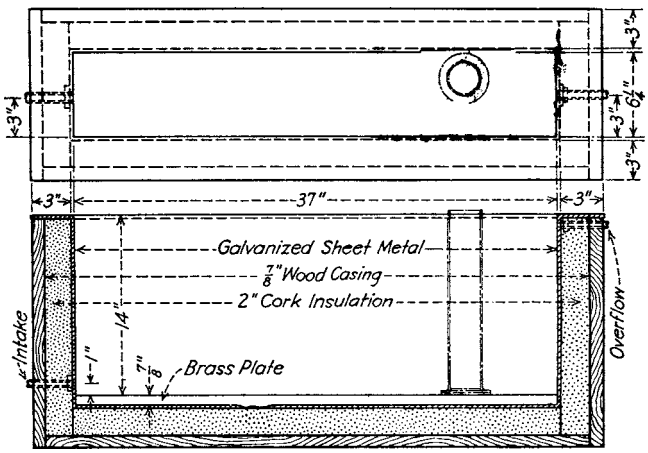


FIG. 3 Air-Jet Dispersion Cups of Apparatus B



Metric Equivalents						
in.	⅞	1	3	6¼	14	37
mm	22.2	25.4	76.2	158.2	356	940

FIG. 4 Insulated Water Bath

hydrometer test is 68°F (20°C). Small variations of temperature do not introduce differences that are of practical significance and do not prevent the use of corrections derived as prescribed.

5. Test Sample

5.1 Prepare the test sample for mechanical analysis as outlined in Practice D421. During the preparation procedure the sample is divided into two portions. One portion contains only particles retained on the No. 10 (2.00-mm) sieve while the other portion contains only particles passing the No. 10 sieve. The mass of air-dried soil selected for purpose of tests, as prescribed in Practice D421, shall be sufficient to yield quantities for mechanical analysis as follows:

5.1.1 The size of the portion retained on the No. 10 sieve shall depend on the maximum size of particle, according to the following schedule:

Nominal Diameter of Largest Particles, in. (mm)	Approximate Minimum Mass of Portion, g
⅜ (9.5)	500
¾ (19.0)	1000
1 (25.4)	2000
1½ (38.1)	3000
2 (50.8)	4000
3 (76.2)	5000

5.1.2 The size of the portion passing the No. 10 sieve shall be approximately 115 g for sandy soils and approximately 65 g for silt and clay soils.

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5.2 Provision is made in Section 5 of Practice D421 for weighing of the air-dry soil selected for purpose of tests, the separation of the soil on the No. 10 sieve by dry-sieving and washing, and the weighing of the washed and dried fraction retained on the No. 10 sieve. From these two masses the percentages retained and passing the No. 10 sieve can be calculated in accordance with 12.1.

NOTE 8—A check on the mass values and the thoroughness of pulverization of the clods may be secured by weighing the portion passing the No. 10 sieve and adding this value to the mass of the washed and oven-dried portion retained on the No. 10 sieve.

SIEVE ANALYSIS OF PORTION RETAINED ON NO. 10 (2.00-mm) SIEVE

6. Procedure

6.1 Separate the portion retained on the No. 10 (2.00-mm) sieve into a series of fractions using the 3-in. (75-mm), 2-in. (50-mm), 1½-in. (37.5-mm), 1-in. (25.0-mm), ¾-in. (19.0-mm), ⅜-in. (9.5-mm), No. 4 (4.75-mm), and No. 10 sieves, or as many as may be needed depending on the sample, or upon the specifications for the material under test.

6.2 Conduct the sieving operation by means of a lateral and vertical motion of the sieve, accompanied by a jarring action in order to keep the sample moving continuously over the surface of the sieve. In no case turn or manipulate fragments in the sample through the sieve by hand. Continue sieving until not more than 1 mass % of the residue on a sieve passes that sieve during 1 min of sieving. When mechanical sieving is used, test the thoroughness of sieving by using the hand method of sieving as described above.

6.3 Determine the mass of each fraction on a balance conforming to the requirements of 3.1. At the end of weighing, the sum of the masses retained on all the sieves used should equal closely the original mass of the quantity sieved.

HYDROMETER AND SIEVE ANALYSIS OF PORTION PASSING THE NO. 10 (2.00-mm) SIEVE

7. Determination of Composite Correction for Hydrometer Reading

7.1 Equations for percentages of soil remaining in suspension, as given in 14.3, are based on the use of distilled or demineralized water. A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.

7.1.1 Both soil hydrometers are calibrated at 68°F (20°C), and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings. The amount of the inaccuracy increases as the variation from the standard temperature increases.

7.1.2 Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of soil suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.

7.1.3 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

7.2 For convenience, a graph or table of composite corrections for a series of 1° temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

7.3 Prepare 1000 mL of liquid composed of distilled or demineralized water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cylinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used. When the temperature of the liquid becomes constant, insert the hydrometer, and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus formed on the stem. For hydrometer 151H the composite correction is the difference between this reading and one; for hydrometer 152H it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

8. Hygroscopic Moisture

8.1 When the sample is weighed for the hydrometer test, weigh out an auxiliary portion of from 10 to 15 g in a small metal or glass container, dry the sample to a constant mass in an oven at 230 ± 9°F (110 ± 5°C), and weigh again. Record the masses.

9. Dispersion of Soil Sample

9.1 When the soil is mostly of the clay and silt sizes, weigh out a sample of air-dry soil of approximately 50 g. When the soil is mostly sand the sample should be approximately 100 g.

9.2 Place the sample in the 250-mL beaker and cover with 125 mL of sodium hexametaphosphate solution (40 g/L). Stir until the soil is thoroughly wetted. Allow to soak for at least 16 h.

9.3 At the end of the soaking period, disperse the sample further, using either stirring apparatus A or B. If stirring apparatus A is used, transfer the soil-water slurry from the beaker into the special dispersion cup shown in Fig. 2, washing any residue from the beaker into the cup with distilled or demineralized water (Note 9). Add distilled or demineralized water, if necessary, so that the cup is more than half full. Stir for a period of 1 min.

NOTE 9—A large size syringe is a convenient device for handling the water in the washing operation. Other devices include the wash-water bottle and a hose with nozzle connected to a pressurized distilled water tank.

9.4 If stirring apparatus B (Fig. 3) is used, remove the cover cap and connect the cup to a compressed air supply by means of a rubber hose. A air gage must be on the line between the cup and the control valve. Open the control valve so that the

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gage indicates 1 psi (7 kPa) pressure (Note 10). Transfer the soil-water slurry from the beaker to the air-jet dispersion cup by washing with distilled or demineralized water. Add distilled or demineralized water, if necessary, so that the total volume in the cup is 250 mL, but no more.

NOTE 10—The initial air pressure of 1 psi is required to prevent the soil-water mixture from entering the air-jet chamber when the mixture is transferred to the dispersion cup.

9.5 Place the cover cap on the cup and open the air control valve until the gage pressure is 20 psi (140 kPa). Disperse the soil according to the following schedule:

Plasticity Index	Dispersion Period, min
Under 5	5
6 to 20	10
Over 20	15

Soils containing large percentages of mica need be dispersed for only 1 min. After the dispersion period, reduce the gage pressure to 1 psi preparatory to transfer of soil-water slurry to the sedimentation cylinder.

10. Hydrometer Test

10.1 Immediately after dispersion, transfer the soil-water slurry to the glass sedimentation cylinder, and add distilled or demineralized water until the total volume is 1000 mL.

10.2 Using the palm of the hand over the open end of the cylinder (or a rubber stopper in the open end), turn the cylinder upside down and back for a period of 1 min to complete the agitation of the slurry (Note 11). At the end of 1 min set the cylinder in a convenient location and take hydrometer readings at the following intervals of time (measured from the beginning of sedimentation), or as many as may be needed, depending on the sample or the specification for the material under test: 2, 5, 15, 30, 60, 250, and 1440 min. If the controlled water bath is used, the sedimentation cylinder should be placed in the bath between the 2- and 5-min readings.

NOTE 11—The number of turns during this minute should be approximately 60, counting the turn upside down and back as two turns. Any soil remaining in the bottom of the cylinder during the first few turns should be loosened by vigorous shaking of the cylinder while it is in the inverted position.

10.3 When it is desired to take a hydrometer reading, carefully insert the hydrometer about 20 to 25 s before the reading is due to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled or demineralized water.

NOTE 12—It is important to remove the hydrometer immediately after each reading. Readings shall be taken at the top of the meniscus formed by the suspension around the stem, since it is not possible to secure readings at the bottom of the meniscus.

10.4 After each reading, take the temperature of the suspension by inserting the thermometer into the suspension.

11. Sieve Analysis

11.1 After taking the final hydrometer reading, transfer the suspension to a No. 200 (75-μm) sieve and wash with tap water until the wash water is clear. Transfer the material on the No. 200 sieve to a suitable container, dry in an oven at 230 ± 9°F (110 ± 5°C) and make a sieve analysis of the portion retained, using as many sieves as desired, or required for the material, or upon the specification of the material under test.

CALCULATIONS AND REPORT

12. Sieve Analysis Values for the Portion Coarser than the No. 10 (2.00-mm) Sieve

12.1 Calculate the percentage passing the No. 10 sieve by dividing the mass passing the No. 10 sieve by the mass of soil originally split on the No. 10 sieve, and multiplying the result by 100. To obtain the mass passing the No. 10 sieve, subtract the mass retained on the No. 10 sieve from the original mass.

12.2 To secure the total mass of soil passing the No. 4 (4.75-mm) sieve, add to the mass of the material passing the No. 10 sieve the mass of the fraction passing the No. 4 sieve and retained on the No. 10 sieve. To secure the total mass of soil passing the ⅜-in. (9.5-mm) sieve, add to the total mass of soil passing the No. 4 sieve, the mass of the fraction passing the ⅜-in. sieve and retained on the No. 4 sieve. For the remaining sieves, continue the calculations in the same manner.

12.3 To determine the total percentage passing for each sieve, divide the total mass passing (see 12.2) by the total mass of sample and multiply the result by 100.

13. Hygroscopic Moisture Correction Factor

13.1 The hydroscopic moisture correction factor is the ratio between the mass of the oven-dried sample and the air-dry mass before drying. It is a number less than one, except when there is no hygroscopic moisture.

14. Percentages of Soil in Suspension

14.1 Calculate the oven-dry mass of soil used in the hydrometer analysis by multiplying the air-dry mass by the hygroscopic moisture correction factor.

14.2 Calculate the mass of a total sample represented by the mass of soil used in the hydrometer test, by dividing the oven-dry mass used by the percentage passing the No. 10 (2.00-mm) sieve, and multiplying the result by 100. This value is the weight W in the equation for percentage remaining in suspension.

14.3 The percentage of soil remaining in suspension at the level at which the hydrometer is measuring the density of the suspension may be calculated as follows (Note 13): For hydrometer 151H:

$$P = \left[(100\,000/W) \times G/\left(G - G_1\right)\right](R - G_1) \quad (1)$$

NOTE 13—The bracketed portion of the equation for hydrometer 151H is constant for a series of readings and may be calculated first and then multiplied by the portion in the parentheses.

For hydrometer 152H:

$$P = (Ra/W) \times 100 \quad (2)$$

where:

a = correction faction to be applied to the reading of hydrometer 152H. (Values shown on the scale are computed using a specific gravity of 2.65. Correction factors are given in Table 1),

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P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension,
R = hydrometer reading with composite correction applied (Section 7),
W = oven-dry mass of soil in a total test sample represented by mass of soil dispersed (see 14.2), g,
G = specific gravity of the soil particles, and
G_1 = specific gravity of the liquid in which soil particles are suspended. Use numerical value of one in both instances in the equation. In the first instance any possible variation produces no significant effect, and in the second instance, the composite correction for R is based on a value of one for G_1.

15. Diameter of Soil Particles

15.1 The diameter of a particle corresponding to the percentage indicated by a given hydrometer reading shall be calculated according to Stokes' law (Note 14), on the basis that a particle of this diameter was at the surface of the suspension at the beginning of sedimentation and had settled to the level at which the hydrometer is measuring the density of the suspension. According to Stokes' law: see Table 2

$$D = \sqrt{[30n/980(G - G_1)] \times L/T} \quad (3)$$

where:

D = diameter of particle, mm,
n = coefficient of viscosity of the suspending medium (in this case water) in poises (varies with changes in temperature of the suspending medium),
L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm. (For a given hydrometer and sedimentation cylinder, values vary according to the hydrometer readings. This distance is known as effective depth (see Table 2)),
T = interval of time from beginning of sedimentation to the taking of the reading, min,
G = specific gravity of soil particles, and
G_1 = specific gravity (relative density) of suspending medium (value may be used as 1.000 for all practical purposes).

TABLE 1 Values of Correction Factor, α, for Different Specific Gravities of Soil Particles[A]

Specific Gravity	Correction Factor[A]
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

[A] For use in equation for percentage of soil remaining in suspension when using Hydrometer 152H.

TABLE 2 Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specified Sizes[A]

Hydrometer 151H		Hydrometer 152H			
Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm
1.000	16.3	0	16.3	31	11.2
1.001	16.0	1	16.1	32	11.1
1.002	15.8	2	16.0	33	10.9
1.003	15.5	3	15.8	34	10.7
1.004	15.2	4	15.6	35	10.6
1.005	15.0	5	15.5		
1.006	14.7	6	15.3	36	10.4
1.007	14.4	7	15.2	37	10.2
1.008	14.2	8	15.0	38	10.1
1.009	13.9	9	14.8	39	9.9
1.010	13.7	10	14.7	40	9.7
1.011	13.4	11	14.5	41	9.6
1.012	13.1	12	14.3	42	9.4
1.013	12.9	13	14.2	43	9.2
1.014	12.6	14	14.0	44	9.1
1.015	12.3	15	13.8	45	8.9
1.016	12.1	16	13.7	46	8.8
1.017	11.8	17	13.5	47	8.6
1.018	11.5	18	13.3	48	8.4
1.019	11.3	19	13.2	49	8.3
1.020	11.0	20	13.0	50	8.1
1.021	10.7	21	12.9	51	7.9
1.022	10.5	22	12.7	52	7.8
1.023	10.2	23	12.5	53	7.6
1.024	10.0	24	12.4	54	7.4
1.025	9.7	25	12.2	55	7.3
1.026	9.4	26	12.0	56	7.1
1.027	9.2	27	11.9	57	7.0
1.028	8.9	28	11.7	58	6.8
1.029	8.6	29	11.5	59	6.6
1.030	8.4	30	11.4	60	6.5
1.031	8.1				
1.032	7.8				
1.033	7.6				
1.034	7.3				
1.035	7.0				
1.036	6.8				
1.037	6.5				
1.038	6.2				

[A] Values of effective depth are calculated from the equation:

$$L = L_1 + 1/2\,[L_2 - (V_B/A)] \quad (4)$$

where:

L = effective depth, cm,
L_1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm,
L_2 = overall length of the hydrometer bulb, cm,
V_B = volume of hydrometer bulb, cm^3, and
A = cross-sectional area of sedimentation cylinder, cm^2

L_2 = 14.0 cm
V_B = 67.0 cm^3
A = 27.8 cm^2

L_1 = 10.5 cm for a reading of 1.000
= 2.3 cm for a reading of 1.031

L_1 = 10.5 cm for a reading of 0 g/litre
= 2.3 cm for a reading of 50 g/litre

NOTE 14—Since Stokes' law considers the terminal velocity of a single sphere falling in an infinity of liquid, the sizes calculated represent the diameter of spheres that would fall at the same rate as the soil particles.

15.2 For convenience in calculations the above equation may be written as follows: see Table 3

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TABLE 3 Values of K for Use in Equation for Computing Diameter of Particle in Hydrometer Analysis

Temperature,° C	Specific Gravity of Soil Particles								
	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01530	0.01505	0.01481	0.01457	0.01435	0.01414	0.01394	0.01374	0.01356
17	0.01511	0.01486	0.01462	0.01439	0.01417	0.01396	0.01376	0.01356	0.01338
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.1323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01309	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01279	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01255	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01249	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01256	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149

$$D = K\sqrt{L/T} \quad (5)$$

where:

K = constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of K for a range of temperatures and specific gravities are given in Table 3. The value of K does not change for a series of readings constituting a test, while values of L and T do vary.

15.3 Values of D may be computed with sufficient accuracy, using an ordinary 10-in. slide rule.

NOTE 15—The value of L is divided by T using the A- and B-scales, the square root being indicated on the D-scale. Without ascertaining the value of the square root it may be multiplied by K, using either the C- or CI-scale.

16. Sieve Analysis Values for Portion Finer than No. 10 (2.00-mm) Sieve

16.1 Calculation of percentages passing the various sieves used in sieving the portion of the sample from the hydrometer test involves several steps. The first step is to calculate the mass of the fraction that would have been retained on the No. 10 sieve had it not been removed. This mass is equal to the total percentage retained on the No. 10 sieve (100 minus total percentage passing) times the mass of the total sample represented by the mass of soil used (as calculated in 14.2), and the result divided by 100.

16.2 Calculate next the total mass passing the No. 200 sieve. Add together the fractional masses retained on all the sieves, including the No. 10 sieve, and subtract this sum from the mass of the total sample (as calculated in 14.2).

16.3 Calculate next the total masses passing each of the other sieves, in a manner similar to that given in 12.2.

16.4 Calculate last the total percentages passing by dividing the total mass passing (as calculated in 16.3) by the total mass of sample (as calculated in 14.2), and multiply the result by 100.

17. Graph

17.1 When the hydrometer analysis is performed, a graph of the test results shall be made, plotting the diameters of the particles on a logarithmic scale as the abscissa and the percentages smaller than the corresponding diameters to an arithmetic scale as the ordinate. When the hydrometer analysis is not made on a portion of the soil, the preparation of the graph is optional, since values may be secured directly from tabulated data.

18. Report

18.1 The report shall include the following:

18.1.1 Maximum size of particles,

18.1.2 Percentage passing (or retained on) each sieve, which may be tabulated or presented by plotting on a graph (Note 16),

18.1.3 Description of sand and gravel particles:

18.1.3.1 Shape—rounded or angular,

18.1.3.2 Hardness—hard and durable, soft, or weathered and friable,

18.1.4 Specific gravity, if unusually high or low,

18.1.5 Any difficulty in dispersing the fraction passing the No. 10 (2.00-mm) sieve, indicating any change in type and amount of dispersing agent, and

18.1.6 The dispersion device used and the length of the dispersion period.

NOTE 16—This tabulation of graph represents the gradation of the sample tested. If particles larger than those contained in the sample were removed before testing, the report shall so state giving the amount and maximum size.

18.2 For materials tested for compliance with definite specifications, the fractions called for in such specifications shall be reported. The fractions smaller than the No. 10 sieve shall be read from the graph.

18.3 For materials for which compliance with definite specifications is not indicated and when the soil is composed almost entirely of particles passing the No. 4 (4.75-mm) sieve, the results read from the graph may be reported as follows:

(*1*) Gravel, passing 3-in. and retained on No. 4 sieve . . . %
(*2*) Sand, passing No. 4 sieve and retained on No. 200 sieve . . . %
  (*a*) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve . . . %
  (*b*) Medium sand, passing No. 10 sieve and retained on No. 40 sieve . . . %
  (*c*) Fine sand, passing No. 40 sieve and retained on No. 200 sieve . . . %
(*3*) Silt size, 0.074 to 0.005 mm . . . %
(*4*) Clay size, smaller than 0.005 mm . . . %
  Colloids, smaller than 0.001 mm . . . %

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18.4 For materials for which compliance with definite specifications is not indicated and when the soil contains material retained on the No. 4 sieve sufficient to require a sieve analysis on that portion, the results may be reported as follows (Note 17):

SIEVE ANALYSIS

Sieve Size	Percentage Passing
3-in.	. . . . .
2-in.	. . . . .
1½-in.	. . . . .
1-in.	. . . . .
¾-in.	. . . . .
⅜-in.	. . . . .
No. 4 (4.75-mm)	. . . . .
No. 10 (2.00-mm)	. . . . .
No. 40 (425-µm)	. . . . .
No. 200 (75-µm)	. . . . .

HYDROMETER ANALYSIS

0.074 mm	. . . . .
0.005 mm	. . . . .
0.001 mm	. . . . .

NOTE 17—No. 8 (2.36-mm) and No. 50 (300-µm) sieves may be substituted for No. 10 and No. 40 sieves.

19. Keywords

19.1 grain-size; hydrometer analysis; hygroscopic moisture; particle-size; sieve analysis

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Construction Material Testing Laboratory **SOP Number:SOP-GT-008**
Terracon **Effective: June, 1999**
Revision: August 4, 2011

STANDARD OPERATING PROCEDURE
FOR
ASTM D2216 – 10 Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

STEP 1
Complete all project information on the data sheet.

STEP 2
Collect 50 to 1500 gms of wet sample depending on particle size of sample and place in tare. Weigh wet soil and tare. Record weight on data sheet. Place tare and sample in drying oven. Maintain the drying oven at 110 +- 5 C unless otherwise specified. In most cases dry over night (about 12 to 16 h) is sufficient.

STEP 3
After the specimen has dried to a constant mass, remove the container from the oven Allow the container to cool to room temperature or until container can be handled comfortably with bare hands. Weigh the specimen.

STEP 4
Calculate the water content of the material.

NOTE: This specimen may be used for P-200, see SOP-GT-07.4.

Designation: D2216 – 10

Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass[1]

This standard is issued under the fixed designation D2216; 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 laboratory determination of the water (moisture) content by mass of soil, rock, and similar materials where the reduction in mass by drying is due to loss of water except as noted in 1.4, 1.5, and 1.7. For simplicity, the word "material" shall refer to soil, rock or aggregate whichever is most applicable.

1.2 Some disciplines, such as soil science, need to determine water content on the basis of volume. Such determinations are beyond the scope of this test method.

1.3 The water content of a material is defined in 3.2.1.

1.4 The term "solid material" as used in geotechnical engineering is typically assumed to mean naturally occurring mineral particles of soil and rock that are not readily soluble in water. Therefore, the water content of materials containing extraneous matter (such as cement etc.) may require special treatment or a qualified definition of water content. In addition, some organic materials may be decomposed by oven drying at the standard drying temperature for this method (110°C). Materials containing gypsum (calcium sulfate dihydrate) or other compounds having significant amounts of hydrated water may present a special problem as this material slowly dehydrates at the standard drying temperature (110°C) and at very low relative humidity, forming a compound (such as calcium sulfate hemihydrate) that is not normally present in natural materials except in some desert soils. In order to reduce the degree of dehydration of gypsum in those materials containing gypsum or to reduce decomposition in highly/fibrous organic soils, it may be desirable to dry the materials at 60°C or in a desiccator at room temperature. Thus, when a drying temperature is used which is different from the standard drying temperature as defined by this test method, the resulting water content may be different from the standard water content determined at the standard drying temperature of 110°C.

NOTE 1—Test Method D2974 provides an alternate procedure for determining water content of peat materials.

1.5 Materials containing water with substantial amounts of soluble solids (such as salt in the case of marine sediments) when tested by this method will give a mass of solids that includes the previously soluble dissolved solids. These materials require special treatment to remove or account for the presence of precipitated solids in the dry mass of the specimen, or a qualified definition of water content must be used. For example, see Test Method D4542 regarding information on marine sediments.

1.6 This test standard requires several hours for proper drying of the water content specimen. Test Methods D4643, D4944 and D4959 provide less time-consuming processes for determining water content. See Gilbert[2] for details on the background of Test Method D4643.

1.7 Two test methods are provided in this standard. The methods differ in the significant digits reported and the size of the specimen (mass) required. The method to be used may be specified by the requesting authority; otherwise Method A shall be performed.

1.7.1 *Method A*—The water content by mass is recorded to the nearest 1 %. For cases of dispute, Method A is the referee method.

1.7.2 *Method B*—The water content by mass is recorded to the nearest 0.1 %.

1.8 This standard requires the drying of material in an oven. If the material being dried is contaminated with certain chemicals, health and safety hazards can exist. Therefore, this standard should not be used in determining the water content of contaminated soils unless adequate health and safety precautions are taken.

1.9 *Units*—The values stated in SI units shall be regarded as standard excluding the Alternative Sieve Sizes listed in Table 1. No other units of measurement are included in this test method.

[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 July 1, 2010. Published August 2010. Originally approved in 1963. Last previous edition approved in 2005 as D2216–05. DOI: 10.1520/D2216-10.

[2] Gilbert, P.A., "Computer Controlled Microwave Oven System for Rapid Water Content Determination," Tech. Report GL-88–21, Department of the Army, Waterways Experiment Station, Corps of Engineers, Vicksburg, MS, November 1988 .

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

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TABLE 1 Minimum Requirements for Mass of Test Specimen, and Balance Readability[A]

Maximum Particle Size (100 % Passing)		Method A Water Content Recorded to ±1 %		Method B Water Content Recorded to ±0.1 %	
SI Unit Sieve Size	Alternative Sieve Size	Specimen Mass	Balance Readability (g)	Specimen Mass (g)	Balance Readability (g)
75.0 mm	3 in	5 kg	10	50 kg	10
37.5 mm	1-½ in.	1 kg	10	10 kg	10
19.0 mm	¾ in.	250 g	1	2.5 kg	1
9.5 mm	⅜ in.	50 g	0.1	500 g	0.1
4.75 mm	No. 4	20 g	0.1	100 g	0.1
2.00 mm	No. 10	20 g	0.1	20 g	0.01

[A]If water content data is to be used to calculate other relationships, such as moist or dry mass, wet or dry unit weight or total or dry density, then specimen mass up to 200 g must be determined using a balance accurate to 0.01 g.

1.10 Refer to Practice D6026 for guidance concerning the use of significant figures that shall determine whether Method, A or B is required. This is especially important if the water content will be used to calculate other relationships such as moist mass to dry mass or vice versa, wet unit weight to dry unit weight or vice versa, and total density to dry density or vice versa. For example, if four significant digits are required in any of the above calculations, then the water content must be recorded to the nearest 0.1 %. This occurs since 1 plus the water content (not in percent) will have four significant digits regardless of what the value of the water content is; that is, 1 plus 0.1/100 = 1.001, a value with four significant digits. While, if three significant digits are acceptable, then the water content can be recorded to the nearest 1 %.

1.11 *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:*[3]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D2974 Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D4220 Practices for Preserving and Transporting Soil Samples

D4318 Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils

D4542 Test Method for Pore Water Extraction and Determination of the Soluble Salt Content of Soils by Refractometer

D4643 Test Method for Determination of Water (Moisture) Content of Soil by Microwave Oven Heating

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D4944 Test Method for Field Determination of Water (Moisture) Content of Soil by the Calcium Carbide Gas Pressure Tester

D4959 Test Method for Determination of Water (Moisture) Content of Soil By Direct Heating

D5079 Practices for Preserving and Transporting Rock Core Samples

D6026 Practice for Using Significant Digits in Geotechnical Data

D7263 Test Methods for Laboratory Determination of Density (Unit Weight) of Soil Specimens

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Terminology

3.1 Refer to Terminology D653 for standard definitions of terms.

3.2 *Definitions:*

3.2.1 *water content by mass (of a material)*—the ratio of the mass of water contained in the pore spaces of soil or rock material, to the solid mass of particles in that material, expressed as a percentage. A standard temperature of 110 ± 5°C is used to determine these masses.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *constant dry mass (of a material)*—the state that a water content specimen has attained when further heating causes, or would cause, less than 1 % or 0.1 % additional loss in mass for Method A or B respectively. The time required to obtain constant dry mass will vary depending on numerous factors. The influence of these factors generally can be established by good judgement, and experience with the materials being tested and the apparatus being used.

4. Summary of Test Method

4.1 A test specimen is dried in an oven at a temperature of 110 ± 5°C to a constant mass. The loss of mass due to drying is considered to be water. The water content is calculated using the mass of water and the mass of the dry specimen.

5. Significance and Use

5.1 For many materials, the water content is one of the most significant index properties used in establishing a correlation between soil behavior and its index properties.

[3] 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.

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5.2 The water content of a material is used in expressing the phase relationships of air, water, and solids in a given volume of material.

5.3 In fine-grained (cohesive) soils, the consistency of a given soil type depends on its water content. The water content of a soil, along with its liquid and plastic limits as determined by Test Method D4318, is used to express its relative consistency or liquidity index.

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 D3740 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with Practice D3740 does not in itself ensure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

6. Apparatus

6.1 *Drying Oven*—Vented, thermostatically-controlled, preferably of the forced-draft type, meeting the requirements of Specification E145 and capable of maintaining a uniform temperature of 110 ± 5°C throughout the drying chamber.

6.2 *Balances*—All balances must meet the requirements of Specification D4753 and this section. A Class GP1 balance of 0.01 g readability is required for specimens having a mass of up to 200 g (excluding mass of specimen container) and a Class GP2 balance of 0.1 g readability is required for specimens having a mass over 200 g. However, the balance used may be controlled by the number of significant digits needed (see 1.10).

6.3 *Specimen Containers*—Suitable containers made of material resistant to corrosion and change in mass upon repeated heating, cooling, exposure to materials of varying pH, and cleaning. Unless a dessicator is used, containers with close-fitting lids shall be used for testing specimens having a mass of less than about 200 g; while for specimens having a mass greater than about 200 g, containers without lids may be used (see Note 3). One uniquely numbered (identified) container or number-matched container and lid combination as required is needed for each water content determination.

NOTE 3—The purpose of close-fitting lids is to prevent loss of moisture from specimens before initial mass determination, and to prevent absorption of moisture from the atmosphere following drying and before final mass determination.

6.4 *Desiccator (Optional)*—A desiccator cabinet or large desiccator jar of suitable size containing silica gel or anhydrous calcium sulfate. It is preferable to use a desiccant that changes color when it needs to be reconstituted.

NOTE 4—Anhydrous calcium sulfate is sold under the trade name Drierite.

6.5 *Container Handling Apparatus*, heat resistant gloves, tongs, or suitable holder for moving and handling hot containers after drying.

6.6 *Miscellaneous*, knives, spatulas, scoops, quartering cloth, wire saws, etc., as required.

7. Samples

7.1 Soil samples shall be preserved and transported in accordance with Practice D4220 Section 8 Groups B, C, or D soils. Rock samples shall be preserved and transported in accordance with Practice D5079 section 7.5.2, Special Care Rock. Keep the samples that are stored prior to testing in non-corrodible airtight containers at a temperature between approximately 3 and 30°C and in an area that prevents direct contact with sunlight. Disturbed samples in jars or other containers shall be stored in such a way as to minimize moisture condensation on the insides of the containers.

7.2 The water content determination should be done as soon as practicable after sampling, especially if potentially corrodible containers (such as thin-walled steel tubes, paint cans, etc.) or plastic sample bags are used.

8. Test Specimen

8.1 For water contents being determined in conjunction with another ASTM method, the specimen mass requirement stated in that method shall be used if one is provided. If no minimum specimen mass is provided in that method then the values given below shall apply. See Howard[4] for background data for the values listed.

8.2 The minimum specimen mass of moist material selected to be representative of the total sample is based on visual maximum particle size in the sample and the Method (Method A or B) used to record the data. Minimum specimen mass and balance readability shall be in accordance with Table 1.

8.3 Using a test specimen smaller than the minimum indicated in 8.2 requires discretion, though it may be adequate for the purposes of the test. Any specimen used not meeting these requirements shall be noted on the test data forms or test data sheets.

8.4 When working with a small (less than 200 g) specimen containing a relatively large gravel particle, it is appropriate not to include this particle in the test specimen. However, any discarded material shall be described and noted on the test data form/sheet.

8.5 For those samples consisting entirely of intact rock or gravel-size aggregate, the minimum specimen mass shall be 500 g. Representative portions of the sample may be broken into smaller particles. The particle size is dictated by the specimen mass, the container volume and the balance being used to determine constant mass, see 10.4. Specimen masses as small as 200 g may be tested if water contents of only two significant digits are acceptable.

9. Test Specimen Selection

9.1 When the test specimen is a portion of a larger amount of material, the specimen must be selected to be representative of the water condition of the entire amount of material. The manner in which the test specimen is selected depends on the purpose and application of the test, type of material being tested, the water condition, and the type of sample (from another test, bag, block, etc.).

[4] Howard, A. K., "Minimum Test Specimen Mass for Moisture Content Determination," *Geotechnical Testing Journal*, ASTM., Vol. 12, No. 1, March 1989, pp. 39-44.

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9.2 For disturbed samples such as trimmings, bag samples, etc; obtain the test specimen by one of the following methods (listed in order of preference):

9.2.1 If the material is such that it can be manipulated and handled without significant moisture loss and segregation, the material should be mixed thoroughly. Select a representative portion using a scoop of a size that no more than a few scoopfuls are required to obtain the proper size of specimen defined in 8.2. Combine all the portions for the test specimen.

9.2.2 If the material is such that it cannot be thoroughly mixed or mixed and sampled by a scoop, form a stockpile of the material, mixing as much as possible. Take at least five portions of material at random locations using a sampling tube, shovel, scoop, trowel, or similar device appropriate to the maximum particle size present in the material. Combine all the portions for the test specimen.

9.2.3 If the material or conditions are such that a stockpile cannot be formed, take as many portions of the material as practical, using random locations that will best represent the moisture condition. Combine all the portions for the test specimen.

9.3 Intact samples such as block, tube, split barrel, etc, obtain the test specimen by one of the following methods depending on the purpose and potential use of the sample:

9.3.1 Using a knife, wire saw, or other sharp cutting device, trim the outside portion of the sample a sufficient distance to see if the material is layered, and to remove material that appears more dry or more wet than the main portion of the sample. If the existence of layering is questionable, slice the sample in half. If the material is layered, see 9.3.3.

9.3.2 If the material is not layered, obtain the specimen meeting the mass requirements in 8.2 by: *(1)* taking all or one-half of the interval being tested; *(2)* trimming a representative slice from the interval being tested; or *(3)* trimming the exposed surface of one-half or from the interval being tested.

NOTE 5—Migration of moisture in some cohesionless soils may require that the entire sample be tested.

9.3.3 If a layered material (or more than one material type is encountered), select an average specimen, or individual specimens, or both. Specimens must be properly identified as to location, or what they represent, and appropriate remarks entered on the test data forms or test data sheets.

10. Procedure

10.1 Determine and record the mass of the clean and dry specimen container and its lid, if used along with its identification number.

10.2 Select representative test specimens in accordance with Section 9.

10.3 Place the moist test specimen in the container and, if used, set the lid securely in position. Determine the mass of the container and moist specimen using a balance (see 8.2 and Table 1) selected on the basis of the specimen mass or required significant digits. Record this value.

NOTE 6—To assist in the oven drying of large test specimens, they should be placed in containers having a large surface area (such as pans) and the material broken up into smaller aggregations.

10.4 Remove the lid (if used) and place the container with the moist specimen in the drying oven. Dry the specimen to a constant mass. Maintain the drying oven at 110 ± 5°C unless otherwise specified (see 1.4). The time required to obtain constant mass will vary depending on the type of material, size of specimen, oven type and capacity, and other factors. The influence of these factors generally can be established by good judgment and experience with the materials being tested and the apparatus being used.

10.4.1 In most cases, drying a test specimen overnight (about 12 to 16 h) is sufficient, especially when using forced draft ovens. In cases where there is doubt concerning the adequacy of drying to a constant dry mass, see 3.3.1 and check for additional loss in mass with additional oven drying over an adequate time period. A minimum time period of two hours should be used, increasing the drying time with increasing specimen mass. A rapid check to see if a relatively large specimen (> than about 100 g of material) is dry; place a small strip of torn paper on top of the material while it is in the oven or just upon removal from the oven. If the paper strip curls the material is **not** dry and requires additional drying time. Specimens of sand may often be dried to constant mass in a period of about 4 h, when a forced-draft oven is used.

10.4.2 Since some dry materials may absorb moisture from drying specimens that still retain moisture, dried specimens shall be removed before placing moist specimens in the same oven; unless they are being dried overnight.

10.5 After the specimen has dried to constant mass, remove the container from the oven (and replace the lid if used). Allow the specimen and container to cool to room temperature or until the container can be handled comfortably with bare hands and the operation of the balance will not be affected by convection currents or heat transmission or both. Determine the mass of the container and oven-dried specimen using the same type/capacity balance used in 10.3. Record this value. Tight fitting lids shall be used if it appears that the specimen is absorbing moisture from the air prior to determination of its dry mass.

10.5.1 Cooling in a desiccator is acceptable in place of tight fitting lids since it greatly reduces absorption of moisture from the atmosphere during cooling.

10.6 A copy of a sample data sheet is shown in Appendix X1. Any data sheet can be used, provided the form contains all the required data.

11. Calculation

11.1 Calculate the water content of the material as follows:

$$w = [(M_{cms} - M_{cds})/(M_{cds} - M_c)] \times 100 = (M_w/M_s) \times 100 \quad (1)$$

where:

w = water content, %,
M_{cms} = mass of container and moist specimen, g,
M_{cds} = mass of container and oven dry specimen, g,
M_c = mass of container, g,
M_w = mass of water ($M_w = M_{cms} - M_{cds}$), g, and
M_s = mass of oven dry specimen ($M_s = M_{cds} - M_c$), g.

12. Report: Test Data Form/Sheet

12.1 The method used to specify how data are recorded on the test data sheets or forms, as given below, is the industry

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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 method for engineering design.

12.1.1 Test data forms or test data sheets shall include the following:

12.1.2 Identification of the sample (material) being tested, such as boring number, sample number, test number, container number etc.

12.1.3 Water content of the specimen to the nearest 1 % for Method A or 0.1 % for Method B, as appropriate based on the minimum mass of the specimen. If this method is used in concert with another method, the water content of the specimen should be reported to the value required by the test method for which the water content is being determined. Refer to Practice D6026 for guidance concerning significant digits, especially if the value obtained from this test method is to be used to calculate other relationships such as unit weight or density. For instance, if it is desired to express dry unit weight, as determined by D7263 to the nearest 0.1 lbf/f^3 (0.02 kN/m^3), it may be necessary to use a balance with a greater readability or use a larger specimen mass to obtain the required significant digits the mass of water so that the water content can be determined to the required significant digits. Also, the significant digits in Practice D6026 may need to be increased when calculating phase relationships requiring four significant digits.

12.1.4 Indicate if test specimen had a mass less than the minimum indicated in 8.2.

12.1.5 Indicate if test specimen contained more than one material type (layered, etc.).

12.1.6 Indicate the drying temperature if different from 110 ± 5°C.

12.1.7 Indicate if any material (size and amount) was excluded from the test specimen.

12.2 When reporting water content in tables, figures, etc., any data not meeting the requirements of this test method shall be noted, such as not meeting the mass, balance, or temperature requirements or a portion of the material is excluded from the test specimen.

13. Precision and Bias

13.1 *Statements on Precision*[5]:

13.1.1 *Precision*—Test data on precision is not presented due to the nature of the soil or rock materials tested by this test method. It is either not feasible or too costly at this time to have ten or more laboratories participate in a round-robin testing program. Any variation observed in the data is just as likely to be due to specimen variation as to operator or laboratory testing variation.

13.1.2 Subcommittee D18.03 is seeking any data from the users of this test method that might be used to make a limited statement on precision.

13.1.3 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

14. Keywords

14.1 aggregate; consistency; index property; laboratory; moisture analysis; moisture content; soil; water content

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D13-1108.

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APPENDIX

(Nonmandatory Information)

X1. WATER CONTENT OF SOIL AND ROCK SAMPLE DATA SHEET

Project Name: _____ Project Number: _____

Test Method: __X__ Method A
_____ Method B

Laboratory Number	04-725-S	04-726-S	04-727-S		
Boring Number	B-1	B-2	B-2		
Field Number	SPT-1	SPT-2	SPT-2a		
Container / Lid Number	725	726	727		
Container Mass, g M_c	770.1	731.7	770.6		
Container+Moist Specimen Mass, g M_{cms}	1895.3	2008.4	1827.9		
Date / Time In Oven	8/20/2004 0700	8/20/2004 0700	8/20/2004 0700		
Initial Container+Oven Dry Specimen Mass, g	1721.4	1872.1	1707.6		
Date / Time Out of Oven	8/20/2004 1200	8/20/2004 1200	8/20/2004 1200		
Secondary Container+Oven Dry Specimen Mass, g	1721.4	1801.2	1660.8		
Date / Time Out of Oven	--	8/20/2004 1600	8/20/2004 1600		
Final Container+Oven Dry Specimen Mass, g, M_{cds}	1721.4	1801.2	1660.8		
Date / Time Out of Oven	--	8/21/2004 0700	8/21/2004 0700		
Mass of Water, g, $M_w = M_{cms} - M_{cds}$	173.9	207.2	167.1		
Mass of Solids, g, $M_s = M_{cds} - M_c$	951.3	1069.5	890.2		
Water Content, %, $w = (M_w/M_s) \times 100$	18	19	19		
Unified Soil Classification Group Symbol (Visual)	GC	GC	GC		
Bold Approximate Maximimum Grain Size (Visual)	3 in., 1½ in., **¾ in.**, ⅜ in., #4, #10, < #10	3 in., 1½ in., **¾ in.**, ⅜ in., #4, #10, < #10	3 in., 1½ in., **¾ in.**, ⅜ in., #4, #10, < #10	3 in., 1½ in., ¾ in., ⅜ in., #4, #10, < #10	3 in., 1½ in., ¾ in., ⅜ in., #4, #10, < #10
Oven Temperature if Other Than 110°C	—	—	—		

Remarks: _____

_____

Tested By: _____ Date: _____ Checked By: _____

Dry Mass By: _____ Date: _____ Spot Checked: _____

Calculated By: _____ Date: _____ Reviewed By: _____

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SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to these test methods since the last issue, D2216–05, that may impact the use of these test methods. (Approved July 1, 2010)

(*1*) Replaced “has to” with “must” in 1.10.
(*2*) Added the “heat resistant” to “gloves” in 6.5.
(*3*) Replaced “reduce” with “minimize” in 7.1.
(*4*) Added “or required significant digits” in 10.3.
(*5*) Revised 10.4.1 to clarify the process of obtaining and checking to determine if a specimen had reached constant mass.
(*6*) Added “that still retain moisture” in 10.4.2.
(*7*) Replaced “its being heated” with “heat transmission” in 10.5.
(*8*) Added “as determined by D7263” in 12.1.3.
(*9*) Added Footnote A to Table 1 reflecting balance requirements outlined in 6.2.

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Construction Material Testing Laboratory **SOP Number: SOP-GT-012**
Terracon **Effective: June, 1999**
Revision: August 4, 2011

STANDARD OPERATING PROCEDURE
FOR
PREPARING TEST SPECIMENS FROM UNDISTURBED SAMPLES FOR PERMEABILITY TESTING

Permeability Testing:

Remove Shelby Tube sample from holding area. Identify the sample to be tested. Measure approximate length of material inside the tube. Mark on the outside of the tube in no less than six inch intervals and secure tube in vise on the band saw. Cut each of the six inch markings and identify the cut section of the tube, ie Top 6", Middle 6", Bottom 6". Extrude the samples using the vertical extruder. Place samples into plastic bags sealed with a sample tag with all project and sample info including location of sample from the tube as described above.

For testing, select the most representative sample from the samples cut out of the Shelby Tube. After specimen selection has been made, using the Split Mitter Box, trim the specimen ends to a length of diameter ratio between 1.25 to 0.75:1. Measure the length and diameter at least three points and record the average on the worksheet. Weigh and record the weight on the worksheet.

Use the specimens trimmings for the initial moisture content in accordance with SOP-GT-008.

This specimen must be mounted into a cell immediately after all measurements are made and recorded on the worksheet. Record Station number or Station location on the worksheet.

STANDARD OPERATING PROCEDURE FOR MEASUREMENT OF HYDRAULIC CONDUCTIVITY OF SATURATED POROUS MATERIALS USING A FLEXIBLE WALL PERMEAMETER ASTM D5084 - 10

Prepare the specimen according to SOP-GT-011 or SOP-GT-012. Mount the specimen, membrane, stones and filter paper into the PERMEAMETER, assemble the cell and fill with water.
When the cell has been filled with water, close all valves that may be open.

Optional:

Calculate the estimated degree of saturation (EDS) by the following equation, Equation # 1:

$$EDS = \frac{\text{Initial Moisture Content, (IMC)}}{(Yw/DD)-(1/Gs)}$$

where, Yw = Unit weight of water, 62.4 pcf
DD = Dry Density of specimen, pcf

and, 1 = specific gravity of water
Gs = specific gravity of soil particles, 2.78 (ASSUMED)

and, using (Yw/DD) - (1/Gs) the Estimated Moisture Content (EMC) where saturation = 100%, the amount of water in mL, needed during the saturation phase to achieve 100% saturation can be calculated using the following equation, Equation # 2:

EMC - IMC = difference in percent = A
and, C/IMC X (A) = B

and, B - C = water needed during the saturation phase in mL.

where, EMC = Estimated Moisture Content
IMC = Initial Moisture Content
and, C = specimen wet mass
B = estimated wet mass of specimen at 100% saturation.

STEP 1 De-airing

Step 1.1
With the specimen mounted, cell filled with water, and all lines connected, apply a small confining pressure of 3 psi to the cell and open cell's panel valve to allow the cell to be pressurized.
Apply 1 psi pressure to the back pressure panel buret-accumulator and open the panel valve and corresponding valves at base of cell to allow water to flow to the specimen, top and bottom.
Step 1.2
Attach a 1/8" line to either of the cell's free valves located at base of cell.
Slowly open one valve at a time so that liquid will flow through the line and end cap.
Repeat Step 1.2 on the other free valve to complete de-airing of lines and end caps.

When all visible air has been removed, close all valves at cell's base.

NOTE: To aid in saturation, a **small** vacuum may be applied to the top and bottom of the specimen if the vacuum is less the target gradient used during the hydraulic conductivity test.

STEP 2 Saturation Phase

Adjust cell and Back pressure regulators to 3 and 0 psi respectfully.

If Equation # 1 and # 2 are used, adjust water levels in the inner burettes and record on perm worksheet and monitor during saturation phase.

STEP 2.1
Using the built-in Auto Load, adjust the Back pressure regulator to 5 psi.
(Cell pressure will automatically rise to 8 psi)
Approximately every 10 minutes rase the Back pressure an addition 5 psi until a total of 45 psi has been applied. (Approximately 80 minutes)

STEP 2.2

Once the Back pressure has reached 45 psi and, an additional 10 minutes has elapsed, rase back pressure to 50 psi. Check the cell pressure to ensure that 53 psi has been achieved. If not, adjust the cell pressure regulator either up or down until 53 psi is achieved.
Allow the specimen to saturate until the next working day or, if Equation # 1 and # 2 are utilized, continue until the target water in mL has been achieved.

STEP 3 Permeation

STEP 3.1

After the requirements in STEP 2 has been satisfied, close all valves and disconnect all lines to the cell. Remove cell from the saturation panel and place cell at a Constant Volume Panel for the permeation phase.

STEP 3.2

Check and adjust the Back pressure and cell pressure regulators on the Constant Volume Panel to the **exact** final pressures used during the saturation phase if needed.

Connect the cell line from the Constant Volume Panel to the quick connect on top of cell. Open panel's cell pressure valve to allow pressure into the cell.

Open panel's center value to **Both**. Check for trapped air in the Head and Tail lines coming from the panel and if present, open Head and Tail panel valves to allow water to flow through lines until all air has been removed from lines. Close Head and Tail valves at the panel.

Connect the **Head** line from the panel to the **Red** valve at cell's base.
Connect the **Tail** line from the panel to the **Black** valve at cell's base.
Open Head and Tail valves at the panel.
Open both the Red and Black valves simultaneously to allow flow into both the Head and Tail side, Top and Bottom of specimen. Allow Specimen to sit for about 10 minutes.

STEP 3.3

After 10 minutes, calculate the target elevation of mercury in center Buret to be used by the follow equation, Equation # 3:

$$I = \frac{\text{sample height} \times 20}{13.56 \times 2.54}$$

where: Sample height is the initial measured height,
20 = maximum target Gradients to be used
13.56 = specific gravity of water + specific gravity of mercury and,
2.54 = conversion of inches to centimeters
I = height of mercury, in cm. At start of test

Once the elevation of mercury in the panel's center buret has been adjusted, record on the worksheet elevations of both the height in the inner buret and outer buret of mercury. Record the height of mercury in the inner buret as Head and the outer buret as Tail.

Record a starting time on the worksheet in the Time column.

The elapsed time between readings is total dependent on the rate of flow through the

specimen, i.e., either 1, 5, 10, 15, 20 minutes or even 15, 30 or 45 seconds and so-on.

Regardless of which time interval is used, calculate permeability between each reading using the programmable calculator HP-32S II. See SOP-GT-13.4 for step-by-step instructions.

STEP 3.4

Continue readings and calculations until a minimum of five readings have been calculated.

Determine the average of the last four calculated perm (k) results and record on worksheet under the “AVG.” Section, right-hand side of worksheet.

Next, determine the Steady State as prescribed immediately above the “AVG.” Section on the worksheet.

Should the Steady State fall outside the prescribed limits, (>1E-08: +/- 25%, <1E-08: +/- 50% of mean) continue with this Step thoroughly until the requirements of this Step as been satisfied.

STEP 3.5 FAILING PERMEABILITY

In the event a specimen falls outside known project requirements (Specifications), the following provides a method for verification of results:

STEP 3.5.1

Do Not Erase Any Recorded Results!
Reset mercury in the center buret by turning the panel’s center valve to both.

Increase the Back pressure by 5 psi and allow the specimen to saturate an additional 24 hours.

After the additional 24 hours, repeat STEPS 3.3 and 3.4 in their entirety.

STEP 4

Once the above Step as been completed, the specimen may be removed from the cell.

Turn the panel’s center valve to both, slowly reduce the Back pressure regulator until all Back pressure as been released.

With the remaining 3 psi Cell pressure, place the drain line’s Quick Connect into the port at base of Cell and drain.

Turn Cell pressure regulator to vent and disconnect the Cell pressure line from top of Cell.

With all water removed from the Cell, disassemble Cell. Remove specimen from the Cell, Membrane, Stones and Filter paper.

Weigh specimen, measure height and record on worksheet.

Check for over testing that may be required of the specimen before disposing of the specimen.

Designation: D5084 – 10

Standard Test Methods for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter[1]

This standard is issued under the fixed designation D5084; 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 test methods cover laboratory measurement of the hydraulic conductivity (also referred to as *coefficient of permeability*) of water-saturated porous materials with a flexible wall permeameter at temperatures between about 15 and 30°C (59 and 86°F). Temperatures outside this range may be used; however, the user would have to determine the specific gravity of mercury and R_T (see 10.3) at those temperatures using data from *Handbook of Chemistry and Physics*. There are six alternate methods or hydraulic systems that may be used to measure the hydraulic conductivity. These hydraulic systems are as follows:

1.1.1 *Method A*—Constant Head

1.1.2 *Method B*—Falling Head, constant tailwater elevation

1.1.3 *Method C*—Falling Head, rising tailwater elevation

1.1.4 *Method D*—Constant Rate of Flow

1.1.5 *Method E*—Constant Volume–Constant Head (by mercury)

1.1.6 *Method F*—Constant Volume–Falling Head (by mercury), rising tailwater elevation

1.2 These test methods use water as the permeant liquid; see 4.3 and Section 6 on Reagents for water requirements.

1.3 These test methods may be utilized on all specimen types (undisturbed, reconstituted, remolded, compacted, etc.) that have a hydraulic conductivity less than about 1×10^{-6} m/s (1×10^{-4} cm/s), providing the head loss requirements of 5.2.3 are met. For the constant-volume methods, the hydraulic conductivity typically has to be less than about 1×10^{-7} m/s.

1.3.1 If the hydraulic conductivity is greater than about 1×10^{-6} m/s, but not more than about 1×10^{-5} m/s; then the size of the hydraulic tubing needs to be increased along with the porosity of the porous end pieces. Other strategies, such as using higher viscosity fluid or properly decreasing the cross-sectional area of the test specimen, or both, may also be possible. The key criterion is that the requirements covered in Section 5 have to be met.

1.3.2 If the hydraulic conductivity is less than about 1×10^{-11} m/s, then standard hydraulic systems and temperature environments will typically not suffice. Strategies that may be possible when dealing with such impervious materials may include the following: *(a)* controlling the temperature more precisely, *(b)* adoption of unsteady state measurements by using high-accuracy equipment along with the rigorous analyses for determining the hydraulic parameters (this approach reduces testing duration according to Zhang et al. (**1**)[2]), and *(c)* shortening the length or enlarging the cross-sectional area, or both, of the test specimen. Other items, such as use of higher hydraulic gradients, lower viscosity fluid, elimination of any possible chemical gradients and bacterial growth, and strict verification of leakage, may also be considered.

1.4 The hydraulic conductivity of materials with hydraulic conductivities greater than 1×10^{-5} m/s may be determined by Test Method D2434.

1.5 All observed and calculated values shall conform to the guide for significant digits and rounding established in Practice D6026.

1.5.1 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 should generally 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 this standard to consider significant digits used in analysis methods for engineering design.

1.6 This standard also contains a Hazards section about using mercury, see Section 7.

1.7 The time to perform this test depends on such items as the Method (A, B, C, D, E, or F) used, the initial degree of saturation of the test specimen and the hydraulic conductivity of the test specimen. The constant volume Methods (E and F) and Method D require the shortest period-of-time. Typically a test can be performed using Methods D, E, or F within two to three days. Methods A, B, and C take a longer period-of-time,

[1] This standard is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.04 on Hydrologic Properties and Hydraulic Barriers.

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[2] The boldface numbers in parentheses refer to the list of references appended to this standard.

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

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from a few days to a few weeks depending on the hydraulic conductivity. Typically, about one week is required for hydraulic conductivities on the order of 1×10^{-9} m/s. The testing time is ultimately controlled by meeting the equilibrium criteria for each Method (see 9.5).

1.8 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are mathematical conversions, which are provided for information purposes only and are not considered standard, unless specifically stated as standard, such as 0.5 mm or 0.01 in.

1.9 *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:*[3]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D698 Test Methods for Laboratory Compaction Characteristics of Soil Using Standard Effort (12 400 ft-lbf/ft^3(600 kN-m/m^3))

D854 Test Methods for Specific Gravity of Soil Solids by Water Pycnometer

D1140 Test Methods for Amount of Material in Soils Finer than No. 200 (75-µm) Sieve

D1557 Test Methods for Laboratory Compaction Characteristics of Soil Using Modified Effort (56,000 ft-lbf/ft^3(2,700 kN-m/m^3))

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

D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D2434 Test Method for Permeability of Granular Soils (Constant Head)

D2435 Test Methods for One-Dimensional Consolidation Properties of Soils Using Incremental Loading

D3550 Practice for Thick Wall, Ring-Lined, Split Barrel, Drive Sampling of Soils

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D4220 Practices for Preserving and Transporting Soil Samples

D4318 Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D4767 Test Method for Consolidated Undrained Triaxial Compression Test for Cohesive Soils

D5079 Practices for Preserving and Transporting Rock Core Samples

D6026 Practice for Using Significant Digits in Geotechnical Data

D6151 Practice for Using Hollow-Stem Augers for Geotechnical Exploration and Soil Sampling

D6169 Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 For common definitions of technical terms in this standard, refer to Terminology D653.

3.1.2 *head loss, Δh*—the change in total head of water across a given distance.

3.1.2.1 *Discussion*—In hydraulic conductivity testing, typically the change in total head is across the influent and effluent lines connected to the permeameter, while the given distance is typically the length of the test specimen.

3.1.3 *permeameter*—the apparatus (cell) containing the test specimen in a hydraulic conductivity test.

3.1.3.1 *Discussion*—The apparatus in this case is typically a triaxial-type cell with all of its components (top and bottom specimen caps, stones, and filter paper; membrane; chamber; top and bottom plates; valves; etc.).

3.1.4 *hydraulic conductivity, k*—the rate of discharge of water under laminar flow conditions through a unit cross-sectional area of porous medium under a unit hydraulic gradient and standard temperature conditions (20°C).

3.1.4.1 *Discussion*—In hydraulic conductivity testing, the term *coefficient of permeability* is often used instead of *hydraulic conductivity*, but *hydraulic conductivity* is used exclusively in this standard. A more complete discussion of the terminology associated with Darcy's law is given in the literature. (**2**, **3**)

3.1.5 *pore volume of flow—in hydraulic conductivity testing*, the cumulative quantity of flow into a test specimen divided by the volume of voids in the specimen.

4. Significance and Use

4.1 These test methods apply to one-dimensional, laminar flow of water within porous materials such as soil and rock.

4.2 The hydraulic conductivity of porous materials generally decreases with an increasing amount of air in the pores of the material. These test methods apply to water-saturated porous materials containing virtually no air.

4.3 These test methods apply to permeation of porous materials with water. Permeation with other liquids, such as chemical wastes, can be accomplished using procedures similar to those described in these test methods. However, these test methods are only intended to be used when water is the permeant liquid. See Section 6.

4.4 Darcy's law is assumed to be valid and the hydraulic conductivity is essentially unaffected by hydraulic gradient.

[3] 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.

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4.5 These test methods provide a means for determining hydraulic conductivity at a controlled level of effective stress. Hydraulic conductivity varies with varying void ratio, which changes when the effective stress changes. If the void ratio is changed, the hydraulic conductivity of the test specimen will likely change, see Appendix X2. To determine the relationship between hydraulic conductivity and void ratio, the hydraulic conductivity test would have to be repeated at different effective stresses.

4.6 The correlation between results obtained using these test methods and the hydraulic conductivities of in-place field materials has not been fully investigated. Experience has sometimes shown that hydraulic conductivities measured on small test specimens are not necessarily the same as larger-scale values. Therefore, the results should be applied to field situations with caution and by qualified personnel.

4.7 In most cases, when testing high swell potential materials and using a constant-volume hydraulic system, the effective confining stress should be about 1.5 times the swell pressure of the test specimen or a stress which prevents swelling. If the confining stress is less than the swell pressure, anomalous flow conditions my occur; e.g., mercury column(s) move in the wrong direction.

NOTE 1—The quality of the result produced by this standard is dependent of 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 this standard 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 *Hydraulic System*—Constant head (Method A), falling head (Methods B and C), constant rate of flow (Method D), constant volume-constant head (Method E), or constant volume-falling head (Method F) systems may be utilized provided they meet the following criteria:

5.1.1 *Constant Head*—The system must be capable of maintaining constant hydraulic pressures to ±5 % or better and shall include means to measure the hydraulic pressures to within the prescribed tolerance. In addition, the head loss across the permeameter must be held constant to ±5 % or better and shall be measured with the same accuracy or better. A pressure gage, electronic pressure transducer, or any other device of suitable accuracy shall measure pressures to a minimum of three significant digits. The last digit may be due to estimation, see 5.1.1.1.

5.1.1.1 Practice D6026 discusses the use or application of estimated digits. When the last digit is estimated and that reading is a function of the eye's elevation/location, then a mirror or another device is required to reduce the reading error caused by parallax.

5.1.2 *Falling Head*—The system shall allow for measurement of the applied head loss, thus hydraulic gradient, to ±5 % or better at any time. In addition, the ratio of initial head loss divided by final head loss over an interval of time shall be measured such that this computed ratio is accurate to ±5 % or better. The head loss shall be measured with a pressure gage, electronic pressure transducer, engineer's scale, graduated pipette, or any other device of suitable accuracy to a minimum of three significant digits. The last digit may be due to estimation, see 5.1.1.1. Falling head tests may be performed with either a constant tailwater elevation (Method B) or a rising tailwater elevation (Method C), see Fig. 1. This schematic of a hydraulic system presents the basic components needed to meet the objectives of Method C. Other hydraulic systems or schematics that meet these objectives are acceptable.

5.1.3 *Constant Rate of Flow*—The system must be capable of maintaining a constant rate of flow through the specimen to ±5 % or better. Flow measurement shall be by calibrated syringe, graduated pipette, or other device of suitable accuracy. The head loss across the permeameter shall be measured to a minimum of three significant digits and to an accuracy of ±5 % or better using an electronic pressure transducer(s) or other device(s) of suitable accuracy. The last digit may be due to estimation, see 5.1.1.1. More information on testing with a constant rate of flow is given in the literature (**4**).

5.1.4 *Constant Volume-Constant Head (CVCH)*—The system, with mercury to create the head loss, must be capable of maintaining a constant head loss cross the permeameter to ±5 % or better and shall allow for measurement of the applied head loss to ±5 % or better at any time. The head loss shall be measured to a minimum of three significant digits with an electronic pressure transducer(s) or equivalent device, (**5**) or based upon the pressure head caused by the mercury column, see 10.1.2. The last digit may be due to estimation, see 5.1.1.1.

5.1.4.1 Schematics of two CVCH systems are shown in Fig. 2 and Fig. 3. In each of these systems, the mercury-filled portion of the tubing may be continuous for constant head loss to be maintained. For the system showed in Fig. 2, the head loss remains constant provided the mercury column is vertical and is retained in only one half of the burette system (left burette in Fig. 2). In the system shown in Fig. 3, the head loss remains constant provided the water-mercury interface on the effluent end remains in the upper horizontal tube, and the water-mercury interface on the influent end remains in the lower horizontal tube. These schematics present the basic components needed to meet the objectives of Method E. Other hydraulic systems or schematics that meet these objectives are acceptable.

5.1.4.2 These types of hydraulic systems are typically not used to study the temporal or pore-fluid effect on hydraulic conductivity. The total volume of the specimen is maintained constant using this procedure, thereby significantly reducing effects caused by seepage stresses, pore fluid interactions, etc. Rather, these systems are intended for determining the hydraulic conductivity of a material as rapidly as possible.

5.1.4.3 *Hazards*—Since this hydraulic system contains mercury, special health and safety precautions have to be considered. See Section 7.

5.1.4.4 *Caution*—For these types of hydraulic systems to function properly, the separation of the mercury column has to be prevented. To prevent separation, the mercury and "constant head" tube have to remain relatively clean, and the inside diameter of this tube cannot be too large; typically a capillary tube is used. The larger diameter flushing tube (Fig. 2) is added

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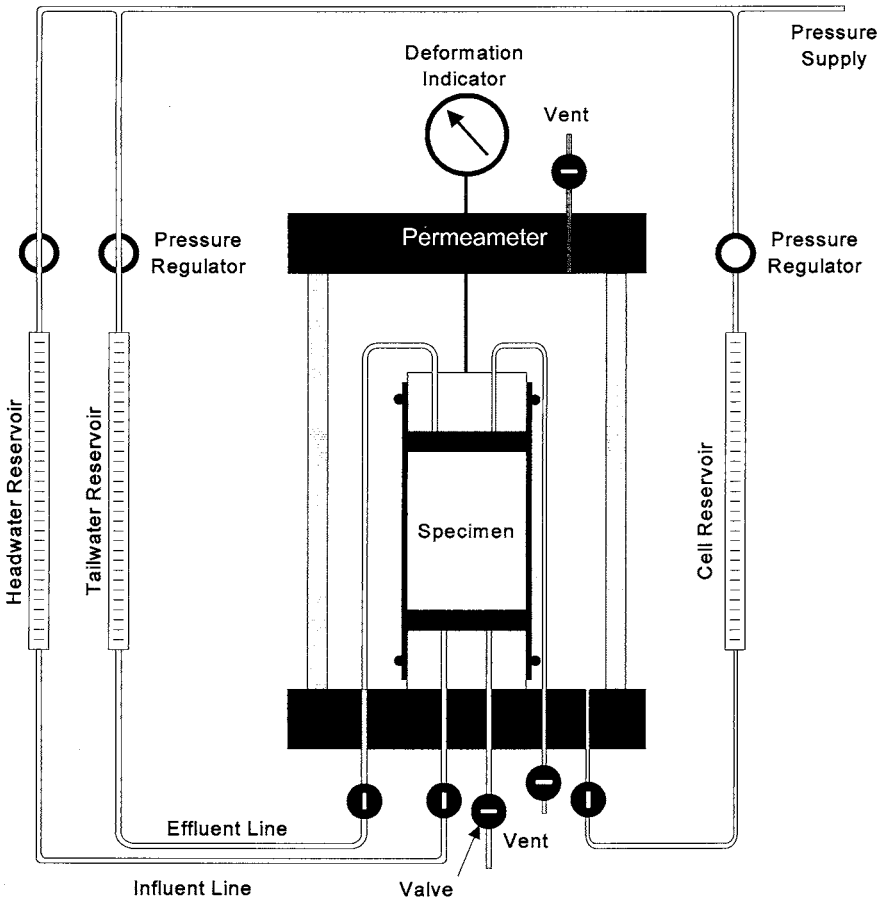

FIG. 1 Falling Head – Rising Tail System, Method C

to enable flushing clean water through the system without excessive mercury displacement. Traps to prevent the accidental flow of mercury out of the "Constant Head" tube or flushing tube are not shown in Fig. 2 and Fig. 3.

5.1.5 *Constant Volume-Falling Head (CVFH)*—The system, with mercury to create the head loss, shall meet the criteria given in 5.1.2. The head loss shall be measured to a minimum of three significant digits with an electronic pressure transducer(s) or equivalent device(s), (**5**) or based upon the differential elevation between the top surfaces of the mercury level in the headwater and tailwater tubes. The last digit may be due to estimation, see 5.1.1.1.

5.1.5.1 A schematic drawing of a typical CVFH hydraulic system is shown in Fig. 4 (**5**). Typically, the tailwater tube has a smaller area than the headwater tube to increase the sensitivity of flow measurements, and to enable flushing clean water through the system without excessive mercury displacement in the headwater tube. The schematic of the hydraulic system in Fig. 4 presents the basic components needed to meet the objectives of Method F. Other hydraulic systems or schematics that meet these objectives are acceptable. The development of the hydraulic conductivity equation for this type of system is given in Appendix X1.

5.1.5.2 See 5.1.4.2.

5.1.5.3 *Hazards*—Since this hydraulic system contains mercury, special health and safety precautions have to be considered. See Section 7.

5.1.5.4 *Caution*—For these types of hydraulic systems to function properly, the separation of the mercury column and entrapment of water within the mercury column have to be prevented. To prevent such problems, the mercury and tubes have to remain relatively clean. In addition, if different size headwater and tailwater tubes are used, capillary head might have to be accounted for, see Appendix X1, X1.2.3.2, and X1.4. Traps to prevent the accidental flow of mercury out of the tubes are not shown in Fig. 4.

5.1.6 *System De-airing*—The hydraulic system shall be designed to facilitate rapid and complete removal of free air bubbles from flow lines; e.g., using properly sized tubing and ball valves and fittings without pipe threads. Properly sized tubing, etc., means they are small enough to prevent entrapment of air bubbles, but not so small that the requirements of 5.2.3 cannot be met.

5.1.7 *Back Pressure System*—The hydraulic system shall have the capability to apply back pressure to the specimen to facilitate saturation. The system shall be capable of maintaining the applied back pressure throughout the duration of

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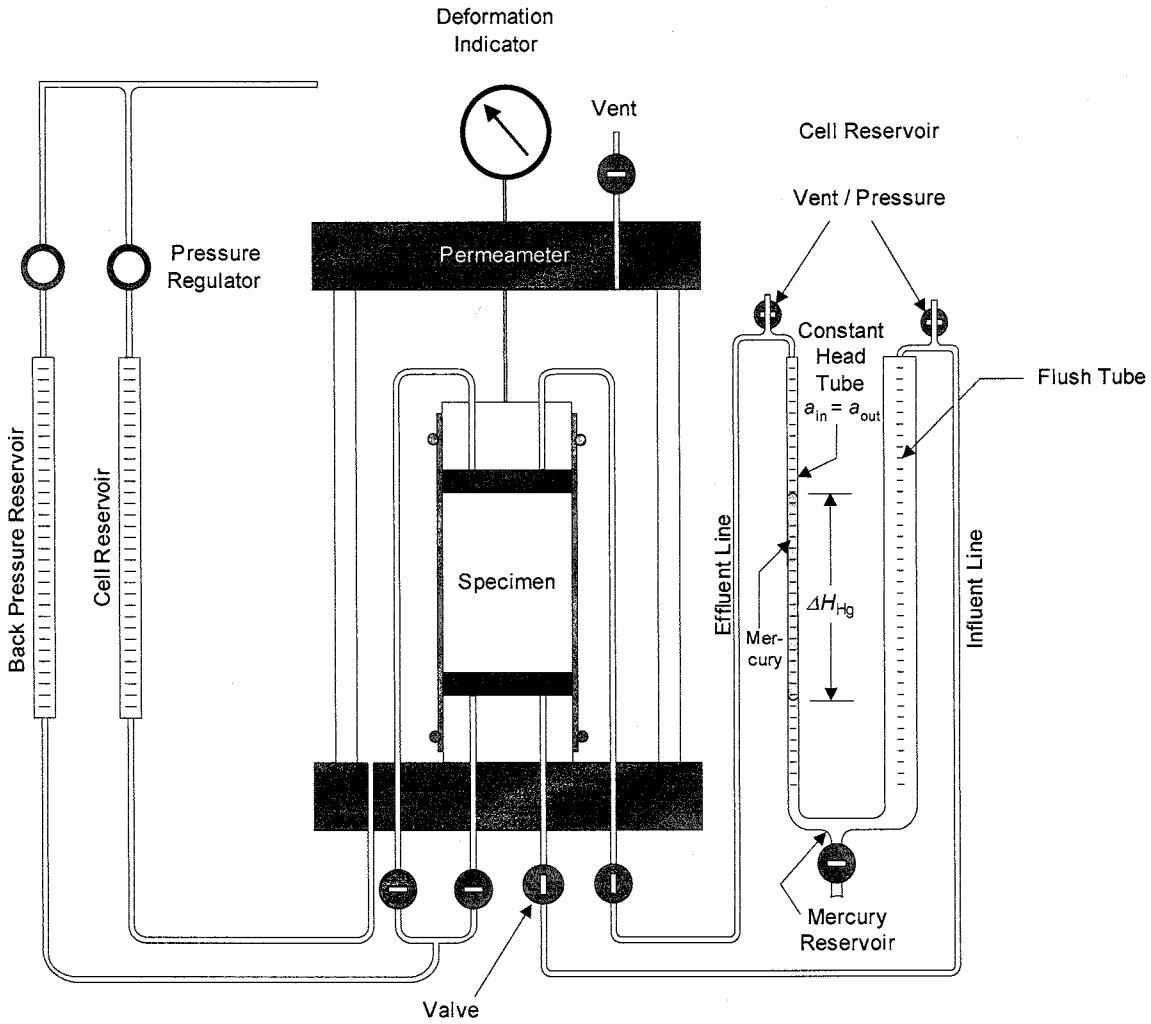

FIG. 2 Constant Volume – Constant Head System, Method E (5)

hydraulic conductivity measurements. The back pressure system shall be capable of applying, controlling, and measuring the back pressure to ±5 % or better of the applied pressure. The back pressure may be provided by a compressed gas supply, a deadweight acting on a piston, or any other method capable of applying and controlling the back pressure to the tolerance prescribed in this paragraph.

NOTE 2—Application of gas pressure directly to a fluid will dissolve gas in the fluid. A variety of techniques are available to minimize dissolution of gas in the back pressure fluid, including separation of gas and liquid phases with a bladder and frequent replacement of the liquid with de-aired water.

5.2 *Flow Measurement System*—Both inflow and outflow volumes shall be measured unless the lack of leakage, continuity of flow, and cessation of consolidation or swelling can be verified by other means. Flow volumes shall be measured by a graduated accumulator, graduated pipette, vertical standpipe in conjunction with an electronic pressure transducer, or other volume-measuring device of suitable accuracy.

5.2.1 *Flow Accuracy*—Required accuracy for the quantity of flow measured over an interval of time is ±5 % or better.

5.2.2 *De-airing and Compliance of the System*—The flow-measurement system shall contain a minimum of dead space and be capable of complete and rapid de-airing. Compliance of the system in response to changes in pressure shall be minimized by using a stiff flow measurement system. Rigid tubing, such as metallic or rigid thermoplastic tubing, or glass shall be used.

5.2.3 *Head Losses*—Head losses in the tubes, valves, porous end pieces, and filter paper may lead to error. To guard against such errors, the permeameter shall be assembled with no specimen inside and then the hydraulic system filled.

5.2.3.1 *Constant or Falling Head*—If a constant or falling head test is to be used, the hydraulic pressures or heads that will be used in testing a specimen shall be applied, and the rate of flow measured with an accuracy of ±5 % or better. This rate of flow shall be at least ten times greater than the rate of flow that is measured when a specimen is placed inside the permeameter and the same hydraulic pressures or heads are applied.

5.2.3.2 *Constant Rate of Flow*—If a constant rate of flow test is to be used, the rate of flow to be used in testing a

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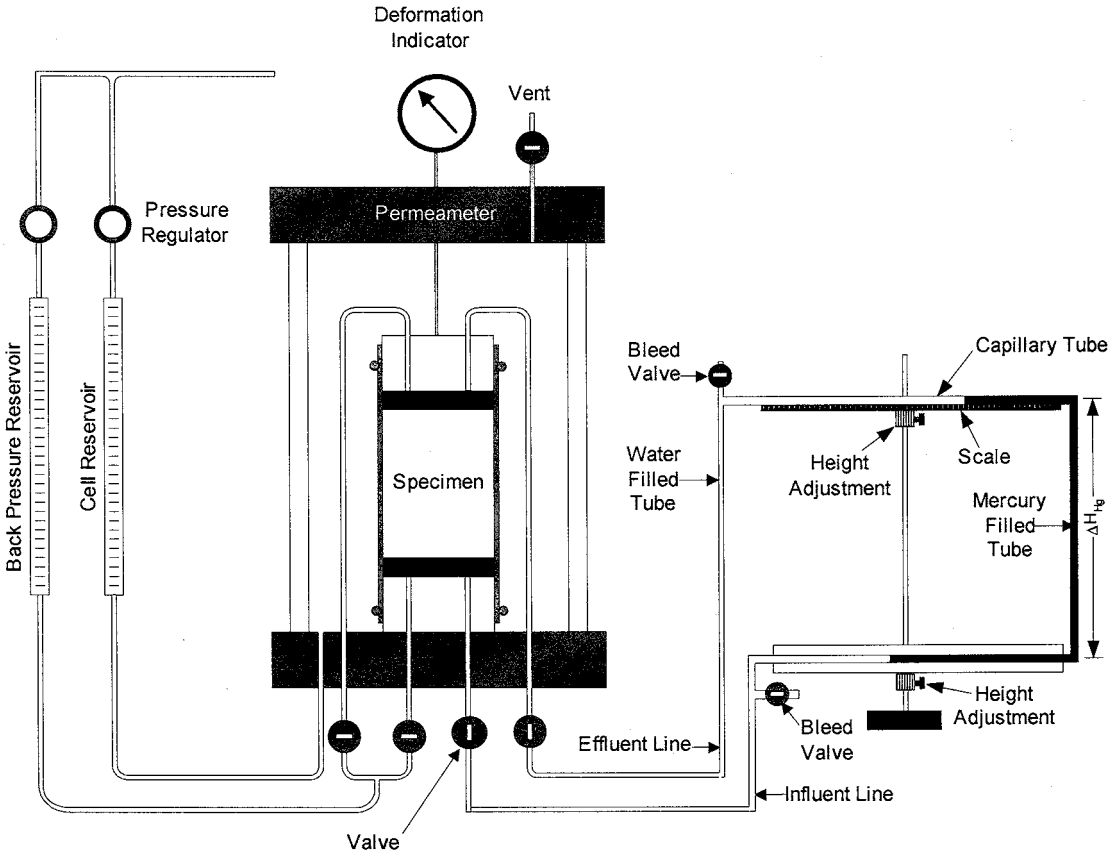

FIG. 3 Constant Volume—Constant Head System, Method E

specimen shall be supplied to the permeameter and the head loss measured. The head loss without a specimen shall be less than 0.1 times the head loss when a specimen is present.

5.3 *Permeameter Cell Pressure System*—The system for pressurizing the permeameter cell shall be capable of applying and controlling the cell pressure to ±5 % or better of the applied pressure. However, the effective stress on the test specimen (which is the difference between the cell pressure and the pore water pressure) shall be maintained to the desired value with an accuracy of ±10 % or better. The device for pressurizing the cell may consist of a reservoir connected to the permeameter cell and partially filled with de-aired water, with the upper part of the reservoir connected to a compressed gas supply or other source of pressure (see Note 3). The gas pressure shall be controlled by a pressure regulator and measured by a pressure gage, electronic pressure transducer, or any other device capable of measuring to the prescribed tolerance. A hydraulic system pressurized by deadweight acting on a piston or any other pressure device capable of applying and controlling the permeameter cell pressure within the tolerance prescribed in this paragraph may be used.

NOTE 3—De-aired water is commonly used for the cell fluid to minimize potential for diffusion of air through the membrane into the specimen. Other fluids that have low gas solubilities such as oils, are also acceptable, provided they do not react with components of the permeameter. Also, use of a long (approximately 5 to 7 m) tube connecting the pressurized cell liquid to the cell helps to delay the appearance of air in the cell fluid and to reduce the flow of dissolved air into the cell.

5.4 *Permeameter Cell*—An apparatus shall be provided in which the specimen and porous end pieces, enclosed by a membrane sealed to the cap and base, are subjected to controlled fluid pressures. A schematic diagram of a typical permeameter cell and falling head (raising tailwater) hydraulic system is shown in Fig. 1.

5.4.1 The permeameter cell may allow for observation of changes in height of the specimen, either by observation through the cell wall using a cathetometer or other instrument, or by monitoring of either a loading piston or an extensometer extending through the top plate of the cell bearing on the top cap and attached to a dial indicator or other measuring device. The piston or extensometer should pass through a bushing and seal incorporated into the top plate and shall be loaded with sufficient force to compensate for the cell pressure acting over the cross-sectional area of the piston where it passes through the seal. If deformations are measured, the deformation indicator shall be a dial indicator or cathetometer graduated to 0.5 mm or 0.01 in. or better and having an adequate travel range. Any other measuring device meeting these requirements is acceptable.

5.4.2 In order to facilitate gas removal, and thus saturation of the hydraulic system, four drainage lines leading to the specimen, two each to the base and top cap, are recommended. The drainage lines shall be controlled by no-volume-change valves, such as ball valves, and shall be designed to minimize dead space in the lines.

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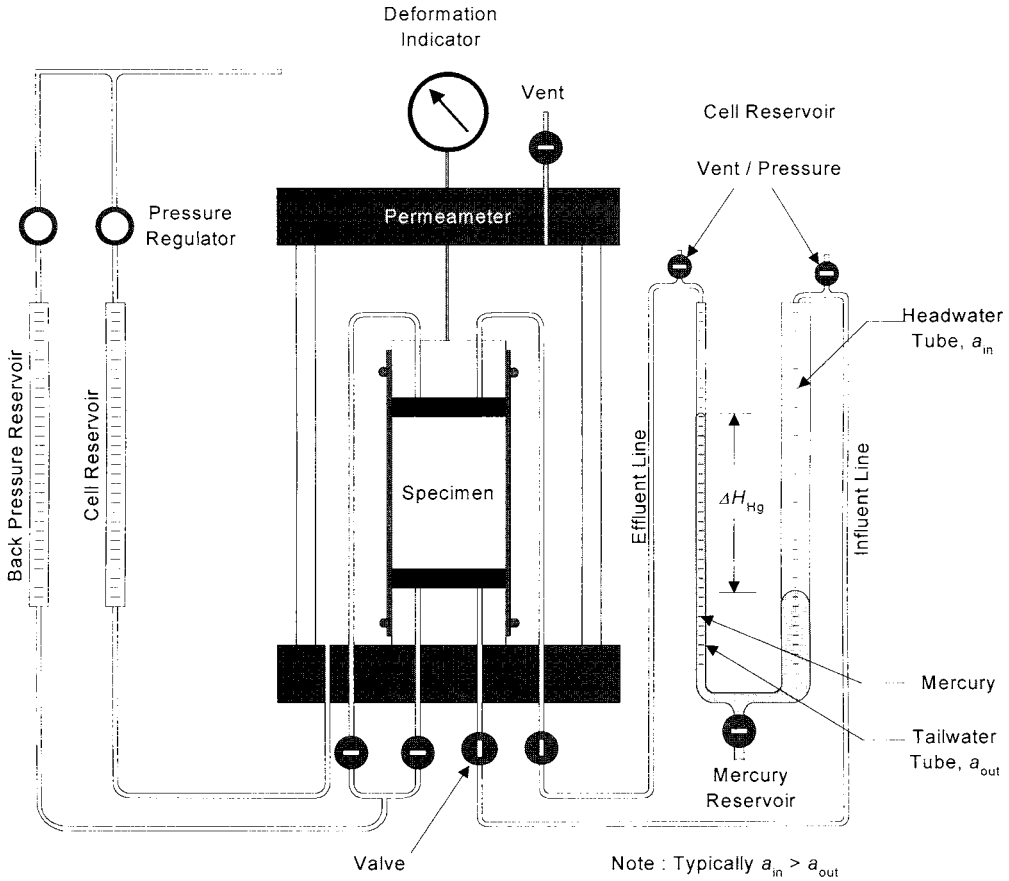

FIG. 4 Constant Volume – Falling Head System, Method F (5)

5.4.3 *Top Cap and Base*—An impermeable, rigid top cap and base shall be used to support the specimen and provide for transmission of permeant liquid to and from the specimen. The diameter or width of the top cap and base shall be equal to the diameter or width of the specimen to ±5 % or better. The base shall prevent leakage, lateral motion, or tilting, and the top cap shall be designed to receive the piston or extensometer, if used, such that the piston-to-top cap contact area is concentric with the cap. The surface of the base and top cap that contacts the membrane to form a seal shall be smooth and free of scratches.

5.4.4 *Flexible Membranes*—The flexible membrane used to encase the specimen shall provide reliable protection against leakage. The membrane shall be carefully inspected prior to use. If any flaws or pinholes are evident, the membrane shall be discarded. To minimize restraint to the specimen, the diameter or width of the non-stretched membrane shall be between 90 and 95 % of that of the specimen. The membrane shall be sealed to the specimen base and cap with rubber O-rings for which the unstressed, inside diameter or width is less than 90 % of the diameter or width of the base and cap, or by any other method that will produce an adequate seal.

NOTE 4—Membranes may be tested for flaws by placing them around a form sealed at both ends with rubber O-rings, subjecting them to a small air pressure on the inside, and then dipping them into water. If air bubbles come up from any point on the membrane, or if any visible flaws are observed, the membrane shall be discarded.

5.4.5 *Porous End Pieces*—The porous end pieces shall be of silicon carbide, aluminum oxide, or other material that is not attacked by the specimen or permeant liquid. The end pieces shall have plane and smooth surfaces and be free of cracks, chips, and discontinuities. They shall be checked regularly to ensure that they are not clogged.

5.4.5.1 The porous end pieces shall be the same diameter or width (±5 % or better) as the specimen, and the thickness shall be sufficient to prevent breaking.

5.4.5.2 The hydraulic conductivity of the porous end pieces shall be significantly greater than that of the specimen to be tested. The requirements outlined in 5.2.3 ensure this criterion is met.

5.4.6 *Filter Paper*—If necessary to prevent intrusion of material into the pores of the porous end pieces, one or more sheets of filter paper shall be placed between the top and bottom porous end pieces and the specimen. The paper shall have a negligibly small hydraulic impedance. The requirements outlined in 5.2.3 ensure that the impedance is small.

5.5 *Equipment for Compacting a Specimen*—Equipment (including compactor and mold) suitable for the method of compaction specified by the requester shall be used.

5.6 *Sample Extruder*—When the material being tested is a soil core, the soil core shall usually be removed from the sampler with an extruder. The sample extruder shall be capable

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of extruding the soil core from the sampling tube in the same direction of travel in which the sample entered the tube and with minimum disturbance of the sample. If the soil core is not extruded vertically, care should be taken to avoid bending stresses on the core due to gravity. Conditions at the time of sample extrusion may dictate the direction of removal, but the principal concern is to keep the degree of disturbance minimal.

5.7 *Trimming Equipment*—Specific equipment for trimming the specimen to the desired dimensions will vary depending on quality and characteristics of the sample (material). However, the following items listed may be used: lathe, wire saw with a wire about 0.3 mm (0.01 in.) in diameter, spatulas, knives, steel rasp for very hard clay specimens, cradle or split mold for trimming specimen ends, and steel straight edge for final trimming of specimen ends.

5.8 *Devices for Measuring the Dimensions of the Specimen*—Devices used to measure the dimensions of the specimen shall be capable of measuring to the nearest 0.5 mm or 0.01 in. or better (see 8.1.1) and shall be constructed such that their use will not disturb the specimen.

5.9 *Balances*—The balance shall be suitable for determining the mass of the specimen and shall be selected as discussed in Specification D4753. The mass of specimens less than 100 g shall be determined to the nearest 0.01 g. The mass of specimens between 100 g and 999 g shall be determined to the nearest 0.1 g. The mass of specimens equal to or greater than 1000 g shall be determined to the nearest gram.

5.10 *Equipment for Mounting the Specimen*—Equipment for mounting the specimen in the permeameter cell shall include a membrane stretcher or cylinder, and ring for expanding and placing O-rings on the base and top cap to seal the membrane.

5.11 *Vacuum Pump*—To assist with de-airing of permeant liquid (water) and saturation of specimens.

NOTE 5—For guidance or avoiding excessive consolidation in the use of vacuum for specimen saturation, consult 8.2 of Test Method D4767.

5.12 *Temperature Maintaining Device*— The temperature of the permeameter, test specimen, and reservoir of permeant liquid shall not vary more than ±3°C or ±6°F or better. Normally, this is accomplished by performing the test in a room with a relatively constant temperature. If such a room is not available, the apparatus shall be placed in a water bath, insulated chamber, or other device that maintains a temperature within the tolerance specified above. The temperature shall be periodically measured and recorded.

5.13 *Water Content Containers*—The containers shall be in accordance with Method D2216.

5.14 *Drying Oven*—The oven shall be in accordance with Test Method D2216.

5.15 *Time Measuring Device(s)*—Devices to measure the duration of each permeation trial, such as either a clock with a second hand or a stopwatch (or equivalent), or both.

6. Reagents

6.1 *Permeant Water*:

6.1.1 The permeant water is the liquid used to permeate the test specimen and is also the liquid used in backpressuring the specimen.

6.1.2 The type of permeant water should be specified by the requestor. If no specification is made, potable tap water shall be used for the permeant liquid. The type of water utilized shall be indicated in the test data sheet/form.

6.1.2.1 Chemical interactions between a permeant liquid and the porous material may lead to variations in hydraulic conductivity. Distilled water can significantly lower the hydraulic conductivity of clayey soils (**2**). For this reason, distilled water is not usually recommended as a permeant liquid. A permeant liquid used by some is a 0.01 molar $CaCl_2$ solution, which can be obtained for example, by dissolving 11.1 g of reagent-grade $CaCl_2$ in 10 L of de-aired, distilled water (commercial grade) or deionized water. This $CaCl_2$ solution is thought to neither increase nor decrease significantly the hydraulic conductivity of clayey soils. In areas with extremely hard or soft water, the $CaCl_2$ solution is recommended. Its use is also recommended when the flow of permeant water is significant (greater than about ⅓ to ½ times the volume of voids). Additional de-airing may modify the concentration of this solution slightly, but this should not affect the hydraulic conductivity.

6.1.3 *Deaired Water*—To aid in removing as much air from the test specimen as possible, deaired water shall be used. The water is usually deaired by boiling, by 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. If boiling is used, care shall be taken not to evaporate an excessive amount of water, which can lead to a larger salt concentration in the permeant water than desired. To prevent dissolution of air back into the water, deaired water shall not be exposed to air for prolonged periods.

7. Hazards

7.1 *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 into your state may be prohibited by state law.

7.1.1 Tubing composed of glass or other brittle materials may explode/shatter when under pressure, especially air. Therefore, such tubing should be enclosed. Establish allowable working pressures and make sure they are not exceeded.

7.2 *Precaution*—In addition to other precautions, store mercury in sealed shatterproof containers to control evaporation. When adding/subtracting mercury to/from the hydraulic system used in Method E or F, work in a well-ventilated area (preferably under a fume hood), and avoid contact with skin. Rubber gloves should be worn at all times when contact with mercury is possible.

7.2.1 Minimize uncontrolled flow of mercury out of the specialized hydraulic system by installing mercury traps or an inline check-valve mechanism. Minimize uncontrolled spills by using shatterproof materials or protective shields, or both.

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7.2.2 If mercury comes into contact with brass/copper fittings, valves, etc., such items may rapidly become leaky. Therefore, where-ever practical use stainless steel fittings, etc.

7.2.3 Clean up spills immediately using a recommended procedure explicitly for mercury.

7.2.4 Dispose of contaminated waste materials containing mercury in a safe and environmentally acceptable manner.

8. Test Specimens

8.1 *Size*—Specimens shall have a minimum diameter of 25 mm (1.0 in.) and a minimum height of 25 mm. The height and diameter of the specimen shall be measured to three significant digits or better (see 8.1.1). The length shall vary by no more than ±5 %. The diameter shall vary by no more than ±5 %. The surface of the test specimen may be uneven, but indentations must not be so deep that the length or diameter vary by more than ±5 %. The diameter and height of the specimen shall each be at least 6 times greater than the largest particle size within the specimen. If, after completion of a test, it is found based on visual observation that oversized particles are present, that information shall be indicated on the data sheet(s)/form(s).

8.1.1 If the density or unit weight needs to be determined/recorded to four significant digits, or the void ratio to three significant digits; then the test specimens dimensions need to have four significant digits; i.e., typically measured to the nearest 0.01 mm or 0.001 in.

8.1.2 Specimens of soil-cement and mixtures of cement, bentonite, and soils often have more irregular surfaces than specimens of soil. Thus, for these specimens the length and the diameter may vary by no more than ±10 %.

NOTE 6—Most hydraulic conductivity tests are performed on cylindrical test specimens. It is possible to utilize special equipment for testing prismatic test specimens, in which case reference to "diameter" in 8.1 applies to the least width of the prismatic test specimen.

8.2 *Undisturbed Specimens*—Undisturbed test specimens shall be prepared from a representative portion of undisturbed samples secured in accordance with Practice D1587, Practice D3550, Practice D6151, or Practice D2113. In addition, undisturbed samples may be obtained by "block sampling" (**6**). Additional guidance on other drilling and sampling methods is given in Guide D6169. Samples shall be preserved and transported in accordance with these requirements; for soils follow Group C in Practice D4220, while for rock follow either "special care" or "soil-like care," as appropriate in Practice D5079. Specimens obtained by tube sampling or coring may be tested without trimming except for cutting the end surfaces plane and perpendicular to the longitudinal axis of the specimen, provided soil characteristics are such that no significant disturbance results from sampling. Where the sampling operation has caused disturbance of the soil, the disturbed material shall be trimmed. Where removal of pebbles or crumbling resulting from trimming causes voids on the surface of the specimen that cause the length or diameter to vary by more than ±5 %, the voids shall be filled with remolded material obtained from the trimmings. The ends of the test specimen shall be cut and not troweled (troweling can seal off cracks, slickensides, or other secondary features that might conduct water flow). Specimens shall be trimmed, whenever possible, in an environment where changes in water content are minimized. A controlled high-humidity room is usually used for this purpose. The mass and dimensions of the test specimen shall be determined to the tolerances given in 5.8 and 5.9. The test specimen shall be mounted immediately in the permeameter. The water content of the trimmings shall be determined in accordance with Method D2216, to the nearest 0.1 % or better.

8.3 *Laboratory-Compacted Specimens*—The material to be tested shall be prepared and compacted inside a mold in a manner specified by the requester. If the specimen is placed and compacted in layers, the surface of each previously-compacted layer shall be lightly scarified (roughened) with a fork, ice pick, or other suitable object, unless the requester specifically states that scarification is not to be performed. Test Methods D698 and D1557 describe two methods of compaction, but any other method specified by the requester may be used as long as the method is described in the report. Large clods of material should not be broken down prior to compaction unless it is known that they will be broken in field construction, as well, or the requester specifically requests that the clod size be reduced. Neither hard clods nor individual particles of the material shall exceed ⅙ of either the height or diameter of the specimen. After compaction, the test specimen shall be removed from the mold, the ends scarified, and the dimensions and weight determined within the tolerances given in 5.8 and 5.9. After the dimensions and mass are determined, the test specimen shall be immediately mounted in the permeameter. The water content of the trimmings shall be determined in accordance with Method D2216 to the nearest 0.1 % or better.

8.4 *Other Preparation Methods*—Other methods of preparation of a test specimen are permitted if specifically requested. The method of specimen preparation shall be identified in the data sheet(s)/form(s).

8.5 After the height, diameter, mass, and water content of the test specimen have been determined, the dry unit weight shall be calculated. Also, the initial degree of saturation shall be estimated (this information may be used later in the back-pressure stage).

8.6 In some cases, the horizontal hydraulic conductivity of a sample needs to be determined. In that case, the specimen may be trimmed such that its longitudinal axis is perpendicular to the longitudinal axis of the sample. Obtaining a specimen having a diameter of 36 mm (1.4 in.) typically requires a cylindrical sample with a diameter equal to or greater than about 70 mm (2.8 in.) or a rectangular sample with a minimum dimension of about 40 mm (1.6 in.).

9. Procedure

9.1 *Specimen Setup*:

9.1.1 Cut two filter paper sheets to approximately the same shape as the cross section of the test specimen. Soak the two porous end pieces and filter paper sheets, if used, in a container of permeant water.

9.1.2 Place the membrane on the membrane expander. Apply a thin coat of silicon high-vacuum grease to the sides of the end caps. Place one porous end piece on the base and place one filter paper sheet, if used, on the porous end piece,

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followed by the test specimen. Place the second filter paper sheet, if used, on top of the specimen followed by the second porous end piece and the top cap. Place the membrane around the specimen, and using the membrane expander or other suitable O-ring expander, place one or more O-rings to seal the membrane to the base and one or more additional O-rings to seal the membrane to the top cap.

9.1.3 Attach flow tubing to the top cap, if not already attached, assemble the permeameter cell, and fill it with de-aired water or other cell fluid. Attach the cell pressure reservoir to the permeameter cell line and the hydraulic system to the influent and effluent lines. Fill the cell pressure reservoir with deaired water, or other suitable liquid, and the hydraulic system with deaired permeant water. Apply a small confining pressure of 7 to 35 kPa (1 to 5 psi) to the cell and apply a pressure less than the confining pressure to both the influent and effluent systems, and flush permeant water through the flow system. After all visible air has been removed from the flow lines, close the control valves. At no time during saturation of the system and specimen or hydraulic conductivity measurements shall the maximum applied effective stress be allowed to exceed that to which the specimen is to be consolidated.

9.2 *Specimen Soaking (Optional)*—To aid in saturation, specimens may be soaked under partial vacuum applied to the top of the specimen. Water under atmospheric pressure shall be applied to the specimen base through the influent lines, and the magnitude of the vacuum set to generate a hydraulic gradient across the specimen less than that which will be used during hydraulic conductivity measurements.

NOTE 7—Soaking under vacuum is applicable when there are continuous air voids in the specimen e.g., specimens having a degree of saturation of less than about 85%. The specimen may swell when exposed to water; the effective stress will tend to counteract the swelling. However, for materials that tend to swell, unless the applied effective stress is greater than or equal to the swell pressure, the specimen will swell. In addition, see Note 5.

9.3 *Back-Pressure Saturation*—To saturate the specimen, back pressuring is usually necessary. Fig. 5 (**7**) provides guidance on back pressure required to attain saturation. Additional guidance on the back-pressure process is given by Black and Lee (**8**) and Head (**9**).

NOTE 8—The relationships presented in Fig. 5 are based on the assumption that the water used for back pressuring is deaired and that the only source for air to dissolve into the water is air from the test specimen. If air pressure is used to control the back pressure, pressurized air will dissolve into the water, thus reducing the capacity of the water used for back pressure to dissolve air located in the pores of the test specimen. The problem is minimized by using a long (>5 m) tube that is impermeable to air between the air-water interface and test specimen, by separating the back-pressure water from the air by a material or fluid that is relatively impermeable to air, by periodically replacing the back-pressure water with deaired water, or by other means.

9.3.1 During the saturation process, any change in the volume (swelling or compression of the void ratio, density, etc.) of the test specimen should be minimized. The easiest way to verify that volume changes are minor is to measure the height of the specimen during the back-pressuring process. Volume changes are considered minor if the resulting change in hydraulic conductivity is less than about one-half the acceptable error of 25 % given in 9.5.4, unless more stringent control on density or hydraulic conductivity, or both, is required. For this to occur the axial strain should be less than about 0.4 % for normally consolidated soils, or about 0.1 % for overconsolidated soils. See Appendix X2.

9.3.2 Take and record an initial reading of specimen height, if being monitored. Open the flow line valves and flush out of the system any free air bubbles using the procedure outlined in 9.1.3. If an electronic pressure transducer or other measuring device is to be used during the test to measure pore pressures or applied hydraulic gradient, bleed any trapped air from the device.

9.3.3 Adjust the applied confining pressure to the value to be used during saturation of the specimen. Apply back pressure by simultaneously increasing the cell pressure and the influent

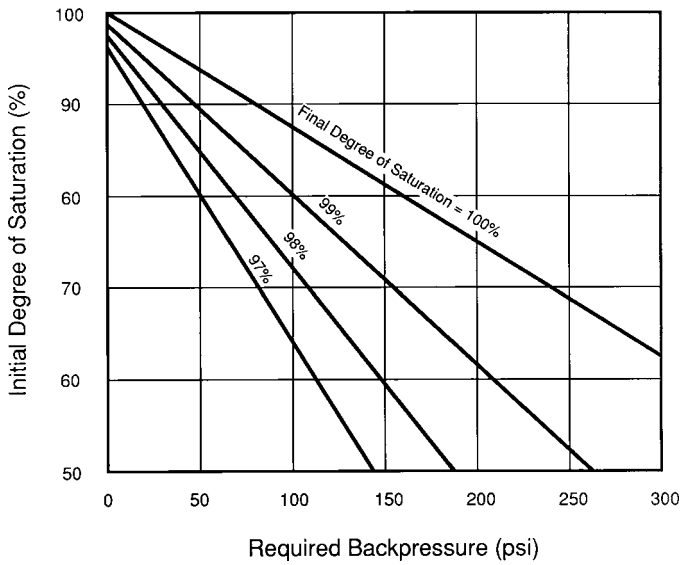

FIG. 5 Back Pressure to Attain Various Degrees of Saturation (7)

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and effluent pressures in increments. The maximum value of an increment in back pressure shall be sufficiently low such that no point in the specimen is exposed to an effective stress in excess of that to which the specimen will be subsequently consolidated. At no time shall a head be applied such that the effective confining stress is <7 kPa (1 psi) because of the danger of separation of the membrane from the test specimen. Maintain each increment of pressure for a period of a few minutes to a few hours, depending upon the characteristics of the specimen. To assist in removal of trapped air, a small hydraulic gradient may be applied across the specimen to induce flow.

9.3.4 Saturation shall be verified with one of the three following techniques:

9.3.4.1 Saturation may be verified by measuring the *B* coefficient as described in Test Method D4767 (see Note 9). The test specimen shall be considered to be adequately saturated if the *B* value is ≥0.95, or for relatively incompressible materials, for example, rock, if the *B* value remains unchanged with application of larger values of back pressure. The *B* value may be measured prior to or after completion of the consolidation phase (see 9.4). An accurate *B*-value determination can only be made if no gradient is acting on the specimen and all pore-water pressure induced by consolidation has dissipated. That is, conform completion of primary consolidation before this determination; see Test Method D2435 or D4767 on how to confirm completion of primary consolidation.

NOTE 9—The *B* coefficient is defined for this type of test as the change in pore-water pressure in the porous material divided by the change in confining pressure. Compressible materials that are fully saturated with water will have a *B* value of 1.0. Relatively incompressible, saturated materials have *B* values that are somewhat less than 1.0 (**10**).

9.3.4.2 Saturation of the test specimen may be confirmed at the completion of the test by calculation of the final degree of saturation. The final degree of saturation shall be 100 ± 5 %. However, measurement of the *B* coefficient as described in 9.3.4.1 or use of some other technique (9.3.4.3) is strongly recommended because it is much better to confirm saturation prior to permeation than to wait until after the test to determine if the test was valid.

9.3.4.3 Other means for verifying saturation, such as observing the flow of water into the specimen when the back pressure is increased, can be used for verifying saturation provided data are available for similar materials to establish that the procedure used confirms saturation as required in 9.3.4.1 or 9.3.4.2.

9.4 *Consolidation*—The specimen shall be consolidated to the effective stress specified by the requester. Consolidation shall be accomplished in stages, with the increase in cell pressure minus back pressure (effective stress) in each new stage equal to or less than the effective stress in the previous stage i.e., consolidation increment ratio of one or less.

NOTE 10—The test specimen may be consolidated prior to application of back pressure. Also, the back pressure and consolidation phases may be completed concurrently if back pressures are applied sufficiently slowly to minimize potential for overconsolidation of the specimen.

9.4.1 Record the specimen height, if being monitored, prior to application of consolidation pressure and periodically during consolidation.

9.4.2 Increase the cell pressure to the level necessary to develop the desired effective stress, and begin consolidation. Drainage may be allowed from the base or top of the specimen, or simultaneously from both ends.

9.4.3 (Optional) Record outflow volumes to confirm that primary consolidation has been completed prior to initiation of the hydraulic conductivity test. Alternatively, measurements of the change in height of the test specimen can be used to confirm completion of consolidation.

NOTE 11—The procedure in 9.4.3 is optional because the requirements of 9.5 ensure that the test specimen is adequately consolidated during permeation because if it is not, inflow and outflow volumes will differ significantly. However, for accurate *B*-value determination, saturation should be confirmed at the completion of consolidation (see 9.3.4.1). Recording outflow volumes or height changes is recommended as a means for verifying the completion of consolidation prior to initialization of permeation. Also, measurements in the change in height of the test specimen, coupled with knowledge of the initial height, provide a means for checking the final height of the specimen.

9.5 *Permeation*:

9.5.1 *Hydraulic Gradient*—When possible, the hydraulic gradient ($i = \Delta h/L$, for definitions of notation see 10.1) used for hydraulic conductivity measurements should be similar to that expected to occur in the field. In general, hydraulic gradients from <1 to 5 cover most field conditions. However, the use of small hydraulic gradients can lead to very long testing times for materials having low hydraulic conductivity (less than about 1×10^{-8} m/s). Somewhat larger hydraulic gradients are usually used in the laboratory to accelerate testing, but excessive gradients must be avoided because high seepage pressures may consolidate the material, material may be washed from the specimen, or fine particles may be washed downstream and plug the effluent end of the test specimen. These effects could increase or decrease hydraulic conductivity. If no gradient is specified by the requestor, the following guidelines may be followed:

Hydraulic Conductivity, m/s	Recommended Maximum Hydraulic Gradient
1×10^{-5} to 1×10^{-6}	2
1×10^{-6} to 1×10^{-7}	5
1×10^{-7} to 1×10^{-8}	10
1×10^{-8} to 1×10^{-9}	20
less than 1×10^{-9}	30

9.5.1.1 A higher gradient than given above may be used if the higher gradient can be shown not to change the hydraulic conductivity. For example, on a representative specimen, perform a hydraulic conductivity determination at $i = 30$ than at $i = 50$ or 100, or more. Determine which, if any, of the hydraulic conductivities (k) determined at these gradients are similar (i.e., within the acceptable steady-state range given for the Method (A, B, C, D, E, or F). Any gradient equal to or less than the highest gradient yielding a similar hydraulic conductivity may be used for testing.

NOTE 12—Seepage pressures associated with large hydraulic gradients can consolidate soft, compressible specimens and reduce their hydraulic conductivity. Smaller hydraulic gradients (<10) may be necessary for such specimens.

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9.5.2 *Initialization*—Initiate permeation of the specimen by increasing the influent (headwater) pressure (see 9.3.3). The effluent (tailwater) pressure shall not be decreased because air bubbles that were dissolved by the specimen water during backpressuring may come out of solution if the pressure is decreased. The back pressure shall be maintained throughout the permeation phase.

9.5.2.1 The maximum increase in headwater pressure cannot exceed 95 % of the effective consolidation stress. Alternatively, the difference between the cell pressure and the total headwater pressure cannot be less than 5 % of the effective consolidation stress.

9.5.2.2 At the start and end of each permeation trial, at t_1 and t_2, read and record the test temperature to the nearest 0.1°C. See Section 10. If the number of significant digits in the calculation of hydraulic conductivity at 20°C can be one, then the test temperature can be measured to the nearest degree Celsius.

9.5.3 *Time Measurements*—Measure and record the time at the start and end of each permeation trial (or its interval) to two or more significant digits. That is the time interval has to be greater than 9 s unless the time is recorded to the nearest 0.1 s.

9.5.4 *Constant Head Tests*:

9.5.4.1 *(Method A)*—Measure and record the required head loss across the tolerances and significant digits stated in 5.1.1 and 5.2.3 at the start and end of each permeation trial (as a minimum). The head loss across the permeameter shall be kept constant to ±5 % or better. Measure and record periodically the quantity of inflow as well as the quantity of outflow to a minimum of three significant digits. Also measure and record any changes in height of the test specimen, if being monitored (see Note 12). Continue permeation until at least four values of hydraulic conductivity are obtained over an interval of time in which: (*1*) the ratio of outflow to inflow rate is between 0.75 and 1.25, and (*2*) the hydraulic conductivity is steady. The hydraulic conductivity shall be considered steady if four or more consecutive hydraulic conductivity determinations fall within ±25 % or better of the mean value for $k \geq 1 \times 10^{-10}$ m/s or within ±50 % or better for $k < 1 \times 10^{-10}$ m/s, and a plot or tabulation of the hydraulic conductivity versus time shows no significant upward or downward trend.

9.5.4.2 *Method E (Constant Volume)*—Measure and record the required head loss across the permeameter to the tolerances and significant digits stated in 5.1.4. The head loss across the permeameter shall be kept constant to ±5 % or better. Measure and record, to a minimum of three significant digits, the quantity of either inflow (influent) or outflow (effluent). In this measurement the last digit may be due to estimation, see 5.1.1.1. In addition, measure and record any changes in the height of the test specimen, if being monitored (see Note 12). Continue permeation until at least two or more values of hydraulic conductivity (k) are steady. The hydraulic conductivity shall be considered steady if two or more consecutive k determinations fall within ±15 % or better of the mean value (two or more determinations) for $k \geq 1 \times 10^{-10}$ m/s or within ±50 % or better for $k < 1 \times 10^{-10}$ m/s.

9.5.5 *Falling-Head Tests (Methods B, C, and F)*—Measure and record the required head loss across the permeameter to the tolerances and significant digits stated in 5.1.2. Measure and record these head losses at the start and end of each permeation trial (as a minimum). At no time shall the applied head loss across the specimen be less than 75 % of the initial (maximum) head loss during the hydraulic conductivity determination (see Note 13). At the start and end of each trial, as a minimum, measure and record any changes in the height of the test specimen, if being monitored. To meet these requirements, especially for Method F, the initial head loss in each trial will most likely have to be reset to the same value (±5 %) used in the first trial. In addition, the "75 % criterion" mentioned above has to be adhered to closely.

9.5.5.1 *Methods B and C*—The volumes of outflow and inflow shall be measured and recorded to three significant digits (the last digit may be due to estimation, see 5.1.1.1). Measure and record these volumes at the start and end of each permeation trial (as a minimum). Continue permeation until at least four values of hydraulic conductivity are obtained over an interval of time in which: the ratio of outflow to inflow rate is between 0.75 and 1.25, and the hydraulic conductivity is steady (see 9.5.4.1).

NOTE 13—When the water pressure in a test specimen changes and the applied total stress is constant, the effective stress in the test specimen changes, which can cause volume changes that can invalidate the test results. The requirement that the head loss not decrease very much is intended to keep the effective stress from changing too much. For extremely soft, compressible test specimens, even more restrictive criteria may be needed. Also, when the initial and final head losses across the test specimen do not differ by much, great accuracy is needed to comply with the requirement of 5.1.2 that the ratio of initial to final head loss be determined with an accuracy of ±5 % or better. When the initial and final head loss over an interval of time do not differ very much, it may be possible to comply with the requirements for a constant head test (9.5.4) in which the head loss must not differ by more than ±5 % and to treat the test as a constant head test.

9.5.5.2 *Method F (Constant Volume)*—Continue permeation until at least two or more values of hydraulic conductivity (k) meet the requirements stated in 9.5.4.2.

9.5.6 *Constant Rate of Flow Tests (Method D)*—Initiate permeation of the specimen by imposing a constant flow rate. Choose the flow rate so the hydraulic gradient does not exceed the value specified, or if none is specified, the value recommended in 9.5.1. Periodically measure the rate of inflow, the rate of outflow, and head loss across the test specimen to the tolerances and significant digits given in 5.1.3. Also, measure and record any changes in specimen height, if being monitored. Continue permeation until at least four values of hydraulic conductivity are obtained over an interval of time in which the ratio of inflow to outflow rates is between 0.75 and 1.25, and hydraulic conductivity is steady (see 9.5.4.1).

9.6 *Final Dimensions of the Specimen*—After completion of permeation, reduce the applied confining, influent, and effluent pressures in a manner that does not generate significant volume change of the test specimen. Then carefully disassemble the permeameter cell and remove the specimen. Measure and record the final height, diameter, and total mass of the specimen. Then determine the final water content of the specimen by the procedure of Method D2216. Dimensions and mass of the test specimen shall be measured to the tolerances specified in 5.8 and 5.9.

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NOTE 14—The specimen may swell after removal of back pressure as a result of air coming out of solution. A correction may be made for this effect, provided that changes in the length of the specimen are monitored during the test. The strain caused by dismantling the cell is computed from the length of the specimen before and after dismantling the cell. The same strain is assumed to have occurred in the diameter. The corrected diameter and actual length before the back pressure was removed are used to compute the volume of the test specimen prior to dismantling the cell. The volume prior to dismantling the cell is used to determine the final dry density and degree of saturation.

10. Calculation

10.1 *Constant Head and Constant Rate of Flow Tests*:

10.1.1 *Methods A and D*—Calculate the hydraulic conductivity, *k*, as follows:

$$k = \frac{\Delta Q \cdot L}{A \cdot \Delta h \cdot \Delta t} \quad (1)$$

where:

k = hydraulic conductivity, m/s,
ΔQ = quantity of flow for given time interval Δt, taken as the average of inflow and outflow, m^3,
L = length of specimen, m,
A = cross-sectional area of specimen, m^2,
Δt = interval of time, *s*, over which the flow ΔQ occurs $(t_2 - t_1)$,
t_1 = time at start of permeation trial, date: hr:min:sec,
t_2 = time at end of permeation trial, date: hr:min:sec,
Δh = average head loss across the permeameter/specimen $((\Delta h_1 + \Delta h_2)/2)$, m of water,
Δh_1 = head loss across the permeameter/specimen at t_1, m of water, and
Δh_2 = head loss across the permeameter/specimen at t_2, m of water.

NOTE 15—The interval of time, Δt, can be measured directly using a stop watch or equivalent device, see 11.5.1. Units other than second(s), meters (m), etc., may be used providing an appropriate unit conversion factor (UCF) is used so *k* is in m/s or other units, if requested or customary (see Section 11).

10.1.2 *Method E*—Use the above Eq 1. If the height of the mercury column in the "Constant Head" tube is used to determine the head loss, Δh, use the following equation.

$$\Delta h = \Delta H_{Hg} \cdot \left(\frac{\rho_{Hg}}{\rho_w} - 1\right) = \Delta H_{Hg} \cdot (G_{Hg} - 1) \quad (2)$$

where:

ΔH_{Hg} = the peak to peak height of mercury column (see Fig. 2), m, and
ρ_{Hg} = the density of mercury, g/cm^3,
ρ_{wg} = the density of water, g/cm^3,
G_{Hg} = the ratio of the density of mercury to the density of water (specific gravity of mercury) at the test/trial temperature. See Table 1.

NOTE 16—For the constant-volume hydraulic systems, there is no head loss across the permeameter/specimen due to elevation head. Units other than seconds (s), meters (m), etc., may be used providing an appropriate UCF is used so *k* is in m/s or other units, if requested or customary (see Section 11).

10.2 *Falling-Head Tests*:

TABLE 1 Specific Gravity of Mercury (G_{Hg})

Temperature (°C)	$G_{Hg} = (\rho_{Hg}/\rho_w)$
15	13.570
16	13.570
17	13.570
18	13.570
19	13.570
20	13.570
21	13.571
22	13.571
23	13.572
24	13.573
25	13.574
26	13.575
27	13.576
28	13.577
29	13.579
30	13.580

10.2.1 *Constant Tailwater Pressure (Method B)*—Calculate the hydraulic conductivity, *k*, as follows:

$$k = \frac{a \cdot L}{A \cdot \Delta t} ln\left(\frac{\Delta h_1}{\Delta h_2}\right) \quad (3)$$

where:

a = cross-sectional area of the reservoir containing the influent liquid, m^2, and
ln = natural logarithm (base e = 2.71828).

See Note 15.

10.2.2 *Increasing Tailwater Pressure (Method C)*—Calculate the hydraulic conductivity, *k*, as follows:

$$k = \frac{a_{in} \cdot a_{out} \cdot L}{(a_{in} + a_{out}) \cdot A \cdot \Delta t} ln\left(\frac{\Delta h_1}{\Delta h_2}\right) \quad (4)$$

where:

a_{in} = cross-sectional area of the reservoir containing the influent/inflow liquid, m^2, and
a_{out} = cross-sectional area of the reservoir containing the effluent/outflow liquid, m^2.

See Note 15.

NOTE 17—For the case in which $a_{out} = a_{in} = a$, the equation for calculating *k* for a falling head test with a rising tailwater level is:

$$k = \frac{a \cdot L}{2 \cdot A \cdot \Delta t} ln\left(\frac{\Delta h_1}{\Delta h_2}\right) \quad (5)$$

where:

a = area of the reservoirs containing either the influent/inflow or effluent/outflow liquid, m^2

10.2.3 *Constant-Volume System (Method F)*— Calculate the hydraulic conductivity, *k*, as follows:

$$k = \left(\frac{a_{in} \cdot a_{out}}{(a_{out} + a_{in})} \cdot \frac{1}{(G_{Hg} - 1)}\right) \cdot \frac{L}{A} \cdot \frac{1}{\Delta t} \cdot ln\left(\frac{\Delta h_1}{\Delta h_2}\right) \quad (6)$$

10.2.3.1 If the differential elevation between the top surfaces of the mercury level in the headwater and tailwater tubes is used to determine the head loss, Δh, use the following equations.

a) For the head loss at the start of the permeation trial, h_1:

$$h_1 = (\Delta H_{Hg,1} + \Delta H_{Hg,c}) \cdot \left(\frac{\rho_{Hg}}{\rho_w} - 1\right) = (\Delta H_{Hg,1} + \Delta H_{Hg,c}) \cdot (G_{Hg} - 1) \quad (7)$$

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where:

$\Delta H_{Hg,1}$ = difference in elevation between the top surfaces of the mercury level in the tailwater and headwater tubes at the start of the permeation trial, t_1(see Fig. 3), m, and

$\Delta H_{Hg,c}$ = difference in elevation of mercury in the headwater and tailwater tubes of the manometer with equal pressures applied to both tubes, m. This value is positive if the inside diameter (ID) of the headwater tube is larger than the ID of the tailwater tube, and negative if the opposite is true. A discussion on capillary head is given in Appendix X1, X1.2.3.2 and X1.4. See Note 16.

b) For the head loss at the end of the permeation trial, Δh_2,:

$$\Delta h_2 = \Delta h_1 + \left((-\Delta Hg_{tail}) \cdot \left(\frac{a_{out}}{a_{in}} + 1 \right) \cdot (G_{Hg} - 1) \right) \quad (8)$$

where:

$-\Delta Hg_{tail}$ = the negative change in elevation of the mercury levels in the tailwater tube during the permeation trial, m.

The reason why ΔHg_{tail} is used instead of $\Delta Hg_{Hg,2}$ (difference in mercury levels at end of trial) is explained in Appendix X1, X1.3.2.1.

10.3 *Hydraulic Conductivity at Standard Temperature*—Correct the hydraulic conductivity to that for 20°C (68°F), k_{20}, by multiplying k by the ratio of the viscosity of water at test temperature to the viscosity of water at 20°C (68°F), R_T:

$$k_{20} = R_T \cdot K \quad (9)$$

with

$$R_T = 2.2902\,(0.9842^{T})/T^{0.1702} \quad (10)$$

where:

k_{20} = hydraulic conductivity corrected to 20°C, m/s

R_T = ratio of the viscosity of water at test temperature to the viscosity of water at 20°C

T = average test temperature during the permeation trial ((T_1+ T_2)/2), to the nearest 0.1°C.

T_1 = test temperature at start of permeation trial, to nearest 0.1 °C, and

T_2 = test temperature at end of permeation trial, to nearest 0.1 °C

10.3.1 The equation for R_T is only accurate to three significant digits between 5 and 50°C (41 and 122°F), see 1.1.

10.3.2 If the number of significant digits in the calculation of hydraulic conductivity at 20°C can be one, then the test temperature can be measured to the nearest °C.

11. Report: Test Data Sheet(s)/Form(s)

11.1 The methodology used to specify how data are recorded on the test data sheet(s)/form(s), as given below, is covered in 1.5.

11.2 Record as a minimum the following general information (data):

11.2.1 Sample/specimen identifying information, such as Project No., Boring No., Sample No., Depth, etc.

11.2.2 Any special selection and preparation process, such as removal of gravel or other materials, or identification of their presence, if "undisturbed" specimen.

11.2.3 If the specimen is reconstituted, remolded or trimmed in a specialized manner (determine horizontal hydraulic conductivity, see 8.6), provide information on method of reconstitution, remolding, etc.

11.3 Record as a minimum the following test specimen data:

11.3.1 The measured specific gravity test (Test Method D854) or assumed value.

11.3.2 The initial mass, dimensions (length and diameter), area, and volume of the specimen, to either three or four significant digits (see 8.1 and 8.1.1).

11.3.3 The initial water content (nearest 0.1 percent), dry unit weight (three or four significant digits, see 8.1.1) and saturation (nearest percent) of the test specimen.

11.3.4 The final mass, dimensions (length and diameter), area, and volume of the specimen, to either three or four significant digits (see 8.1 and 8.1.1).

11.3.5 The final water content (nearest 0.1 percent), dry unit weight (three or four significant digits, see 8.1.1) and saturation (nearest percent) of the test specimen.

11.4 Record as a minimum the following test boundary conditions:

11.4.1 The type of permeant liquid used.

11.4.2 The magnitude of total back pressure (two significant digits or three if used in the head loss determination).

11.4.3 The effective consolidation stress (two or more significant digits).

11.4.4 The area of the headwater and tailwater tubes (such as burettes, reservoirs, U-tube manometers, etc.), as applicable (three or more significant digits).

11.4.5 The length (L) and area (A) of the test specimen during permeation (minimum of three significant digits).

11.4.5.1 These values can be determined based on either *a)* the initial dimensions of specimen plus any length/height and volume changes occurring during saturation and consolidation; or *b)* final dimensions of the test specimen, see 11.3.4.

11.5 Record as a minimum the following permeation data:

11.5.1 The date, time (or start and elapsed time), temperature (nearest 0.1°C see 10.3.2), head loss reading(s), flow reading(s) (if applicable), and deformation gage (if applicable) at the start and end of each trial/determination. Applicable measurements/readings and any averages/differences calculated using measurements/readings obtained shall have two or more significant digits, unless specified differently in Section 9.

11.5.2 The calculated initial hydraulic gradient and ending value if falling head Method B, C, or F is being used, and the hydraulic conductivity to two or more significant digits.

11.5.3 The average corrected hydraulic conductivity (k_{20}, see 10.3) for the values meeting the applicable requirements in 9.5.4 to 9.5.6. Record this value to two or three significant digits in units of m/s or other units, if requested or customary, for example, 7.1×10^{-10} or 7.13×10^{-10}m/s.

11.5.4 A graph or table of hydraulic conductivity versus time or pore volumes of flow is recommended, unless a constant-volume hydraulic system is used.

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12. Precision and Bias

12.1 *Precision*—The precision of this test method is based on an interlaboratory study of D5084, Standard Test Methods for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter, conducted in 2008. Each of twelve laboratories tested three different soil types:

ML-1: Vicksburg silt (ASTM Reference Soil ML-1)
CH-1: Vicksburg clay (ASTM Reference Soil CH-1)
CL-1: Annapolis clay (ASTM Reference Soil CL-1)

All three soils are from the D18 ISR Reference Soils and Testing Program. Index properties for the soils are shown in Table 2. These properties are from the ASTM Reference Soils and Testing Program.

Every "test result" represents an individual determination. Each laboratory reported three replicate test results for the analyses. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:D18-D1018.[4]

12.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "r" value for that material; "r" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

12.1.1.1 Repeatability limits are listed in Table 3.

12.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "R" value for that material; "R" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

12.1.2.1 Reproducibility limits are listed in Table 3.

12.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

12.1.4 Any judgment in accordance with statements 12.1.1 and 12.1.2 would have an approximately 95 % probability of being correct.

12.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method. Therefore no statement on bias is being made.

12.3 The precision statement was determined through statistical examination of 104 results, from twelve laboratories, on the three soils described in 12.1.

13. Keywords

13.1 coefficient of permeability; constant head; constant rate of flow; constant volume; falling head; hydraulic barriers; hydraulic conductivity; liner; permeability; permeameter

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D18-1018.

TABLE 2 Index Properties for ASTM Reference Soils Used in Interlaboratory Study on Saturated Hydraulic Conductivity

ASTM Reference Soil ID	Liquid Limit per D4318 (%)	Plasticity Index per D4318 (%)	Specific Gravity of Soils per D854 (-)	Percent Finer than No. 200 Sieve per D1140 (%)
ML-1	27.3 ± 2.5	3.9 ± 4.5	2.725 ± 0.043	99.0 ± 0.3
CL-1	33.2 ± 1.4	13.4 ± 3.7	2.675 ± 0.030	88.5 ± 0.8
CH-1	59.7 ± 2.8	39.3 ± 7.0	2.726 ± 0.032	98.8 ± 0.4

TABLE 3 Hydraulic Conductivity Statistics from ILS Report RR:D18-D1018 (All Units in cm/s)

ILS Soil	Average $\bar{x}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
ML-1	1.2×10^{-6}	3.3×10^{-7}	4.4×10^{-7}	9.3×10^{-7}	1.2×10^{-6}
CL-1	3.8×10^{-8}	4.4×10^{-9}	6.2×10^{-9}	1.2×10^{-8}	1.8×10^{-8}
CH-1	3.6×10^{-9}	2.9×10^{-9}	4.7×10^{-9}	8.2×10^{-9}	1.3×10^{-8}

APPENDIXES

(Nonmandatory Information)

X1. DEVELOPMENT OF HYDRAULIC CONDUCTIVITY EQUATION FOR THE MERCURY CONSTANT VOLUME-FALLING HEAD HYDRAULIC SYSTEM

X1.1 *Introduction*—A schematic of a mercury constant volume-falling head hydraulic system is given in Fig. 4. In this figure, the falling head is applied by the difference in elevation between the mercury levels in the tailwater and headwater tubes of the mercury U-tube manometer. In designing this type of hydraulic system, the area of the tailwater tube (a_{out}) is made significantly smaller than that of the headwater tube (a_{in}). This is done for three reasons:

First, to increase the sensitivity of the flow/volume measurement;

Second, to decrease the time required to measure the hydraulic conductivity; and,

Third, so one can clean the hydraulic system by flushing water through the tailwater tube and out the headwater tube of the mercury U-tube manometer without loosing mercury.

X1.1.1 The tubing lines leading from the test specimen to mercury U-tube manometer are filled with water, as well as the spaces above the mercury in the manometer. Therefore, the volume of the saturated test specimen remains constant during permeation. This occurs because the components (water, tubing, and manometer) of the hydraulic system are relatively incompressible compared to soil. In addition, there is continuity of inflow and outflow of permeant water during permeation.

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X1.1.2 The presentation of determining heads and head losses in a mercury constant volume-falling head hydraulic system is presented before the development of the hydraulic conductivity equation. This allows one to become familiar with the notation and required parameters before addressing that relatively complicated equation development process. The heads involved are the total head (H), pressure head (H_p), elevation head (H_e), capillary head (H_c), velocity head (H_v), and total head loss (Δh).

X1.2 *Determination of Total Head and Total Head Loss*

X1.2.1 *Total Head*—The total head is equal to the sum of the pressure head (H_p), elevation head (H_e), velocity head (H_v), and capillary head (H_c). Usually it is expressed in height of water, such as m or cm of water. In addition, the velocity head is assumed to be zero/insignificant providing the conditions specified in 4.4 through 5.2.3.2 are met. The pressure head is the height of a vertical column of static water that can be supported by the static pressure (p) at a given point. It may be expressed as

$$H_p = \text{UCF} \cdot \frac{p}{\rho_w \cdot g} \quad \text{(X1.1)}$$

where:

H_p = the pressure head at given point (units of vertical height of water column, m),
UCF = a unit conversion factor,
p = the static pressure at a given point (units of force per unit area, kPa),
ρ_w = the density of the water (units of mass per unit volume, Mg/m^3), and
g = the acceleration of gravity, convert mass to force (9.80665 m/s^2 or 980.665 cm/s^2).

X1.2.2 The static pressure at any point within a confined fluid may be calculated as shown in Fig. X1.1, assuming there is no drop in pressure due to velocity head loss. This figure shows that fluid pressure for Point O at elevation El. O on the "out" or tailwater side may be expressed as:

$$p_{o,\,out} \approx \text{UCF} \cdot (\Delta H_{Hg,OB} \cdot \rho_{Hg} \cdot g + \Delta p_{c,out} + \Delta H_{w,BD} \cdot \rho_w \cdot g + U_b) \quad \text{(X1.2)}$$

or

$$p \approx \text{UCF} \cdot (\Delta H_{Hg} \cdot \rho_{Hg} \cdot g + \Delta p_c + \Delta H_w \cdot \rho_w \cdot g + U_b)$$

where:

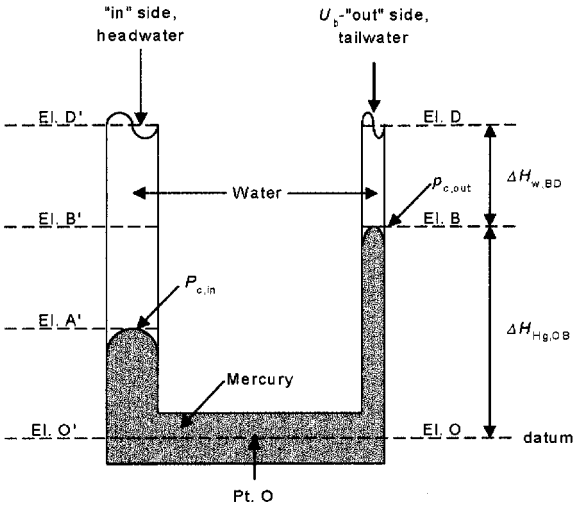
ΔH_{Hg} = the differential height of the mercury column, m,
ρ_{Hg} = the density of the mercury, Mg/m^3,
ΔH_w = the differential height of the water column, m,
Δp_c = the change in pressure due to capillarity at the tube-water-mercury interface, kPa, and
U_b = the applied back pressure, kPa.

X1.2.3 For the above case, the pressure head (in height of water) is

$$H_p \approx \text{UCF} \cdot \left(H_{Hg} \times \frac{\rho_{Hg} \cdot g}{\rho_w \cdot g} + H_w \times \frac{\rho_w \cdot g}{\rho_w \cdot g} + \frac{\Delta p_c}{\rho_w \cdot g} + \frac{U_b}{\rho_w \cdot g} \right) \quad \text{(X1.3)}$$

or

$$H_p \approx \text{UCF} \cdot \left(H_{Hg} \times G_{Hg} + \Delta H_w + \Delta H_c + \frac{U_b}{\rho_w \cdot g} \right)$$



Mercury U-tube Manometer

Determination of pressure for Pt. O at EL. O on the "out" or tailwater side, $p_{O,out}$.

$$p_{O,out} = \text{UCF} \cdot (OB \cdot \rho_{Hg} \cdot g + \Delta p_{c,out} + BD \cdot \rho_w \cdot g + U_b)$$

or

$$= \text{UCF} \cdot (\Delta H_{Hg,OB} \cdot \rho_{Hg} \cdot g + \Delta p_{c,out} + \Delta H_{w,BD} \cdot \rho_w \cdot g + U_b)$$

where:

UCF = a unit conversion factor,
ρ_{Hg} = the density of mercury, g/cm^3 or Mg/m^3,
ρ_w = the density of water, g/cm^3 or Mg/m^3,
g = the acceleration of gravity, m/s^2 or cm/s^2,
$\Delta p_{c,out}$ = the change in pressure due to capillarity at the tube-water-mercury interface in the outflow or tailwater tube, and
U_b = the applied back pressure, kPa or kN/m^2

FIG. X1.1 Static Pressure Calculations

where:

G_{Hg} = the specific gravity of mercury at a given temperature, and
ΔH_c = the change in head due to capillarity, m of water, see X1.2.3.2

X1.2.3.1 *Velocity Head*—In most cases, the velocity head or velocity head loss is assumed equal to zero or insignificant, providing the requirements specified in 5.2.3 are met.

X1.2.3.2 *Capillary Head*—In most cases, the capillary head or capillary head loss is assumed equal to zero. However, in some mercury U-tube manometers, the difference in capillary head between the headwater and tailwater tubes; i.e., the capillary head loss, ΔH_c is significant. Therefore, it has to be accounted for as shown in Fig. X1.2. To help one understand the derivation of ΔH_c in this figure, one has to remember to account for the "water leg" in the mercury U-tube manometer containing water instead of air. Subtracting away the "water leg" pressure does this. Also, the pressure difference measured by the manometer has to be converted to a pressure head by dividing it by $\rho_w \cdot g$. As shown in Fig. X1.2, the capillary head loss is

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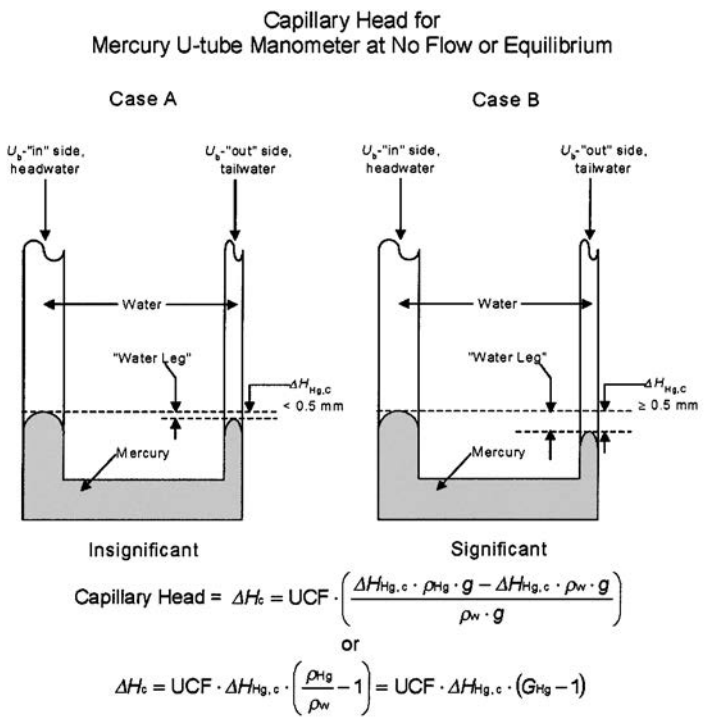

NOTE—For this case capillary head loss is a positive value since the total head on the head water side would have to be increased to make the mercury levels equal.

FIG. X1.2 Difference in Capillary Head in Mercury U-tube Manometer

$$\Delta H_c = \Delta H_{Hg,c} \cdot (G_{Hg} - 1) \quad \text{(X1.4)}$$

where:

ΔH_c = capillary head loss in hydraulic system, m of water,

$\Delta H_{Hg,c}$ = differential height of mercury in the tailwater/outflow and headwater/inflow tubes of the manometer with equal pressure applied to each tube, m of mercury, and

G_{Hg} = specific gravity of mercury at test/trial temperature, see Table 1.

X1.2.3.2.1 As shown in Fig. X1.2, the application of either ΔH_c or $\Delta H_{Hg,c}$ is only necessary when $\Delta H_{Hg,c}$ is equal to or greater than 0.0005 m or 0.5 mm (0.02 in.). An explanation of how to measure ΔH_c is given in X1.4.

X1.2.4 *Total Head*—As stated above, the total head equals the sum of the pressure, elevation, velocity, and capillary heads. Since the change in velocity head is assumed to be zero and the change in capillary head is included in the pressure head calculation given above (Eq X1.3), the total head relationships at various points/elevations, as shown in Fig. X1.3, may be expressed as follows.

X1.2.4.1 Assuming there are no head losses in the tubing, the pressure head at Point Z ($H_{p,Z}$) equals the pressure head just above Point B ($H_{p,B}$), before the effect of capillary head; therefore

$$H_{p,Z} = H_{p,B} \text{ and } H_{p,X} = H_{p,B'}$$

X1.2.4.2 By definition, the total head just above Point B equals the pressure plus elevation heads at that point, therefore

$$H_B = H_{p,B} + H_{e,OB} = \frac{U_b}{\rho_w \cdot g} + H_{e,OB}$$

and

$$H_{B'} = H_{p,B'} + H_{e,O'B'}$$

Assuming continuity in hydraulics, the pressure head at Point B′ equals

$$H_{p,B'} = \frac{U_b}{\rho_w \cdot g} + \Delta H_{p,c,B} + H_{p,BA} + H_{p,AO} - H_{p,O'A'} - \Delta H_{p,c,A'} - H_{p,A'B'}$$

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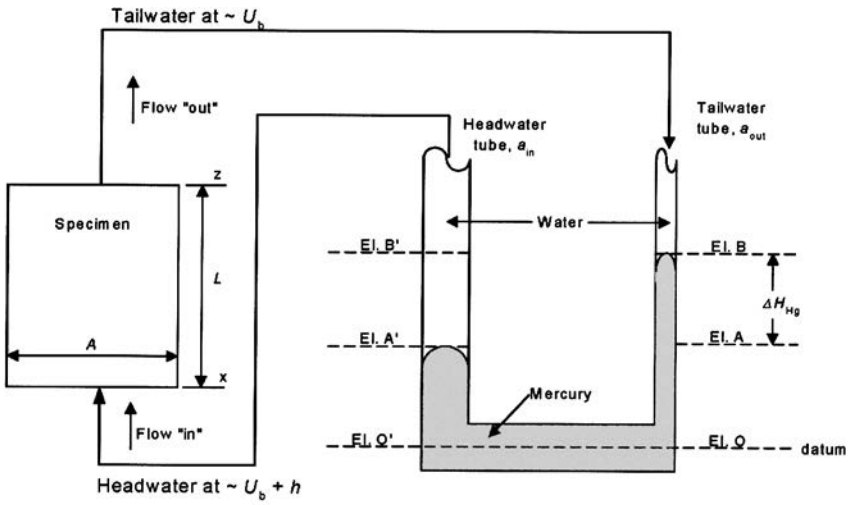

FIG. X1.3 Schematic of Mercury Constant Volume – Falling Head Hydraulic System for Head and Head Loss Equations

where:

$\Delta H_{p,c,B}$ = the change in capillary pressure head going from just above Point B to just below it, and

$\Delta H_{p,c,A'}$ = the change in capillary pressure head going from just below Point A′ to just above it.

X1.3 *Total Head Loss*—Based on a detailed review of the hydraulic systems shown in Fig. X1.1, Fig. X1.2, and Fig. X1.3, one can come to the conclusion that the flow of fluid (permeant) will only occur when the difference in the mercury heights in the U-tube manometer is greater then equilibrium value, as shown in Fig. X1.2.

X1.3.1 *Initial Head Loss*—Using the total head discussion given above and the notation given in Fig. X1.3; the initial total-head loss (Δh_1) across the specimen is

$$\Delta h_1 = H_X - H_Z = H_{B'} - H_B \quad \text{(X1.5)}$$

or

$$\Delta h_1 = H_{p,B'} - H_{p,B} =$$
$$= \frac{U_b}{\rho_w \cdot g} + \Delta H_{p,c,B} + H_{p,BA} + H_{p,AO} - H_{p,O'A'} - \Delta H_{p,c,A'} - H_{p,A'B'} - H_{p,B}$$

Since

$$H_{p,b} = \frac{U_b}{\rho_w \cdot g}, \quad \text{(X1.6)}$$

$$H_{p,AO} = H_{p,O'A'}, \text{ and}$$
$$\Delta H_c = \Delta H_{p,c,B} - \Delta H,$$

therefore

$$\Delta h_1 = H_{p,BA} - H_{p,A'B'} + \Delta H_c \quad \text{(X1.7)}$$

Using generic notation instead of specific notation as given in Fig. X1.1 to Fig. X1.4, therefore

$$\Delta h_1 = \frac{\Delta H_{Hg,1} \cdot g}{\rho_W} - \Delta H_{Hg,1} + \Delta H_{Hg,c} \times \left(\frac{\rho_{Hg}}{\rho_W} - 1 \right) \quad \text{(X1.8)}$$

and rearranging, therefore

$$\Delta h_1 = (\Delta H_{Hg} + \Delta H_{Hg,c}) \cdot \left(\frac{\rho_{Hg}}{\rho_w} - 1 \right) \quad \text{(X1.9)}$$

$$\Delta h_1 = (\Delta H_{Hg,1} + \Delta H_{Hg,c}) \cdot (G_{Hg} - 1)$$

where:

Δh_{Hg} = the initial total-head loss at the start (t_1) of a given permeation trial, in m of water,

ΔH_{Hg} = the initial differential height of mercury in the tailwater and headwater tubes of the manometer at the start (t_1) of a given permeation trial, in m,

$\Delta H_{Hg,c}$ = the positive differential height of mercury in the tailwater and headwater tubes of the manometer with equal pressures applied to both tubes, in m. This height differential is caused by the difference in capillary pressure heads within the two tubes making up the mercury U-tube manometer, see X1.2.3.2 and X1.4

ΔH

X1.3.2 *Final Head Loss*—The final total-head loss (Δh_2) across the specimen is

$$\Delta h_2 = (\Delta H_{Hg,2} + \Delta H_{Hg,c}) \cdot (G_{Hg} - 1) \quad \text{(X1.10)}$$

where:

Δh_2 = the final total-head loss at the end (t_2) Of a given permeation trial, in m of water,

$\Delta H_{Hg,2}$ = the final differential height of mercury in the tailwater and headwater tubes of the manometer at the end (t_2) of a given permeation trial, in m.

X1.3.2.1 The determination of $\Delta H_{Hg,2}$ requires two readings; i.e., the elevation of the top surfaces of the mercury (meniscus) in the tailwater and headwater tubes. Each of these readings will have some error, especially the headwater reading. In addition, the change in the headwater readings between t_1 and t_2 is typically very small and at about the sensitivity to which readings can be made/estimated. Because of these factors, it is assumed that the accuracy of Δh_2 can increased by just measuring the change in elevation of the top surface

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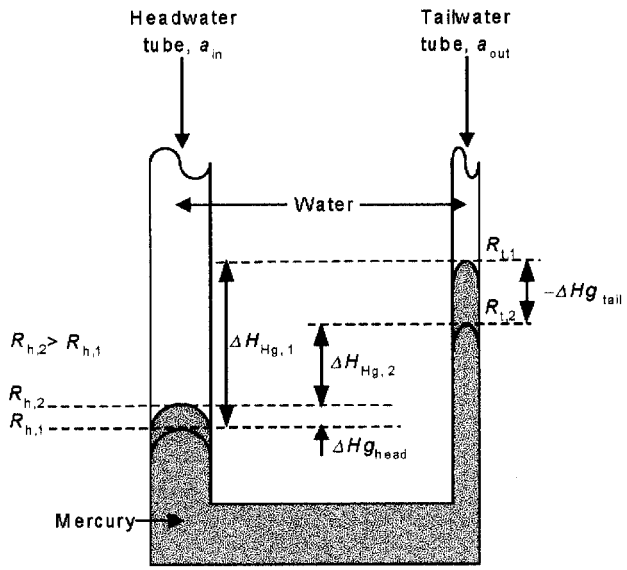

"Flow" Functions:

Flow "in" = $\Delta Q_{in} = \Delta Hg_{head} \cdot a_{in}$

Flow "out" = $-\Delta Q_{out} = -\Delta Hg_{tail} \cdot a_{out}$

Based on continuity, $\Delta Q_{in} = -\Delta Q_{out}$

Therefore, $\Delta Hg_{head} \cdot a_{in} = -\Delta Hg_{tail} \cdot a_{out}$

or

$$\Delta Hg_{head} = -\Delta Hg_{tail} \cdot \frac{a_{out}}{a_{in}}$$

"Head" Functions:

$$\Delta H_{Hg,2} = \Delta H_{Hg,1} + (-\Delta Hg_{tail}) - \Delta Hg_{head}$$

Substituting for ΔHg_{head},

$$\Delta H_{Hg,2} = \Delta H_{Hg,1} + (-\Delta Hg_{tail}) + \left(-\Delta Hg_{tail} \cdot \frac{a_{out}}{a_{in}}\right)$$

$$\Delta H_{Hg,2} = \Delta H_{Hg,1} + (-\Delta Hg_{tail}) \cdot \left(\frac{a_{out}}{a_{in}} + 1\right)$$

Substituting for $\Delta H_{Hg,2}$ in the final head loss equation (X1.10) and rearranging,

$$\Delta h_2 = \left(\Delta H_{Hg,1} + \Delta H_{Hg,c} + \left(-\Delta Hg_{tail}\right) \cdot \left(\frac{a_{out}}{a_{in}} + 1\right)\right) \cdot (G_{Hg} - 1)$$

or

$$\Delta h_2 = \Delta h_1 + \left(\left(-\Delta Hg_{tail}\right) \cdot \left(\frac{a_{out}}{a_{in}} + 1\right) \cdot (G_{Hg} - 1)\right)$$

FIG. X1.4 Relationship Between Change in Flow and Total Head Loss

mercury level in the tailwater tube and calculating what $\Delta H_{Hg,2}$ should be based on the area relationships between the tailwater and headwater tubes. As shown in Fig. X1.4, the following flow relationships can be established.

$$\Delta Q_{in} = \Delta Hg_{head} \cdot a_{in}$$

,

$$-\Delta Q_{out} = -\Delta Hg_{tail} \cdot a_{out}$$

NOTE X1.1—the symbol for height (H) has been omitted to keep the notation simpler, and

ΔQ_{in} = inflow of permeant water for given time interval (positive units of volume),

$-\Delta Q_{out}$ = outflow of permeant water for given time interval (negative units of volume),

ΔHg_{head} = positive change in elevation of the mercury level (top of meniscus) in the headwater tube (units of distance)

$-\Delta Hg_{tail}$ = negative change in elevation of the mercury level (top of meniscus) in the tailwater tube (units of distance),

a_{in} = area of the headwater/inflow tube containing mercury (units of area), and

a_{out} = area of the tailwater/outflow tube containing mercury (units of area)

Based on continuity of flow in a saturated specimen at constant volume,

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$$\Delta Q\text{in} = -\Delta Q\text{out}$$

therefore,

$$\Delta Hg_{\text{head}} \cdot a_{\text{in}} = -\Delta Hg_{\text{tail}} \cdot a_{\text{out}} \quad \text{(X1.11)}$$

or

$$\Delta Hg_{\text{head}} = -\Delta Hg_{\text{tail}} \cdot \frac{a_{\text{out}}}{a_{\text{in}}}$$

therefore,

$$\Delta H_{\text{Hg},2} = \Delta H_{Hg,1} + (-\Delta Hg_{\text{tail}}) + \left(\Delta Hg_{\text{tail}} \cdot \frac{a_{\text{out}}}{a_{\text{in}}} \right) \quad \text{(X1.12)}$$

or

$$\Delta H_{\text{Hg},2} = \Delta H_{\text{Hg},1} + (-\Delta Hg_{\text{tail}}) \cdot \left(\frac{a_{\text{out}}}{a_{\text{in}}} + 1 \right)$$

Substituting for $\Delta H_{\text{Hg},2}$ and rearranging

$$\Delta h_2 = \left(\Delta H_{\text{Hg},1} + \Delta H_{\text{Hg,c}} + (-\Delta Hg_{\text{tail}}) \cdot \left(\frac{a_{\text{out}}}{a_{\text{in}}} + 1 \right) \right) \cdot (G_{\text{Hg}} - 1) \quad \text{(X1.13)}$$

or

$$\Delta h_2 = \Delta h_1 + \left((-\Delta Hg_{\text{tail}}) \cdot \left(\frac{a_{\text{out}}}{a_{\text{in}}} + 1 \right) \cdot (G_{\text{Hg}} - 1) \right)$$

X1.4 *Capillary Head Measurements*—The key to measuring the difference in capillary head (ΔH_c) between the headwater and tailwater tubes of the mercury U-tube manometer is to ensure that an equal water pressure is applied to both tubes. In addition, flow of water can occur under that equal water pressure. This can be accomplished by individually connecting the tailwater and headwater tubing lines to clean burettes containing water at equal elevation. These lines can not have any air bubbles in them. Then, apply the same air pressure to these two burettes. This air pressure should be similar to the back pressure applied during testing. Finally, adjust the height of one burette (typically the one connected to the headwater line) so the water level within each burette is equal. In making this height adjustment, make sure the water level in the headwater burette starts out below that of the tailwater burette. This simulates the direction of fluid flow during the test.

X1.4.1 Once the water level in the two burettes are level, determine the difference in elevation of the two mercury columns at the tops of their meniscuses. The mercury level in the tailwater tube (one with a smaller ID) should be below that in the headwater tube. If it is not, there is an error in applying equal pressures to the two tubes of the U-tube manometer, check for air in the lines, external pressure source, etc..

X1.4.2 If the mercury U-tube manometer being used is the version in which the tailwater tube is contained within the headwater tube, a different approach has to be used. A different approach is required since the mercury level in the tailwater tube is not visible at equilibrium. One approach would be to raise the headwater burette until the mercury levels (top of menisci) in the U-tube manometer (headwater and tailwater columns) are equal. Then determine the difference in elevation of the water levels in the headwater and tailwater burettes in m of water,

Next, convert ΔH_c to $\Delta H_{\text{Hg,c}}$ with

$\Delta H_{\text{Hg,c}} = \Delta H_c/(G_{\text{Hg}} - 1) = \Delta H_c/12.74$, in m of mercury. The value of 12.57 is good for temperatures ranging between 15°C and 25°C.

X1.5 *Falling-Head Hydraulic Conductivity Equation*—Darcy's law for hydraulic conductivity in a saturated medium requires that:

$$q = k \cdot i \cdot A = k \cdot \frac{\Delta h}{L} \cdot A \quad \text{(X1.14)}$$

or

$$\Delta Q = k \cdot \frac{\Delta h}{L} \cdot A \cdot \Delta t$$

where:

- q = rate of flow of the fluid (units of volume over time, m^3/s),
- k = hydraulic conductivity or coefficient of permeability (units of length over time, m/s),
- i = hydraulic gradient (no unit),
- Δh = total head loss across a given length/test specimen (unit of height of water, m),
- L = given length (test specimen) over which the total head loss occurs (unit of distance, m),
- ΔQ = volume of flow for a given time interval (unit of volume, m^3), and
- Δt = time interval (unit of time, s).

X1.5.1 For a differential volume of flow and time period, this equation becomes

$$d\Delta Q = k \cdot \frac{\Delta h}{L} \cdot A \cdot d\Delta t \quad \text{(X1.15)}$$

where:

- $d\Delta Q$ = differential volume of flow in a differential time period, and
- $d\Delta t$ = differential time period.

X1.5.2 It can be demonstrated that the differential volume of flow is a function of the differential head loss, as shown below.

From Fig. X1.4 or X1.3.2.1;

$$-\Delta Q = -\Delta Hg_{\text{tail}} \cdot a_{\text{out}} \quad \text{(X1.16)}$$

or

$$-d\Delta Q = -d\Delta Hg_{\text{tail}} \cdot a_{\text{out}}$$

and from Fig. X1.4,

$$-d\Delta Hg_{\text{tail}} = \frac{dh}{1} \cdot \frac{a_{\text{in}}}{a_{\text{out}} + a_{\text{in}}} \cdot \frac{1}{(G_{\text{Hg}} - 1)} \quad \text{(X1.17)}$$

By substituting for $-d\Delta Q$ and $-d\Delta Hg_{\text{tail}}$ from the above equations in Eq X1.15 we get,

$$-\frac{dh}{1} \cdot \frac{a_{\text{in}} \cdot a_{\text{out}}}{a_{\text{out}} + a_{\text{in}}} \cdot \frac{1}{(G_{\text{Hg}} - 1)} = k \cdot \frac{\Delta h}{L} \cdot A \cdot d\Delta t \quad \text{(X1.18)}$$

or

$$d\Delta t = -\frac{L}{A} \cdot \frac{1}{k} \cdot \frac{1}{\Delta h} \cdot \frac{1}{(G_{\text{Hg}} - 1)} \cdot \frac{a_{\text{in}} \cdot a_{\text{out}}}{a_{\text{out}} + a_{\text{in}}} \cdot dh$$

By integrating between times t_1 and t_2 and h_1 and h_2 we get,

$$\int_{t_1}^{t_2} d\Delta t = \int_{\Delta h_1}^{\Delta h_2} -\frac{L}{A} \cdot \frac{1}{k} \cdot \frac{1}{\Delta h} \cdot \frac{1}{(G_{\text{Hg}} - 1)} \cdot \frac{a_{\text{in}} \cdot a}{a_{out} + a_{in}} \cdot dh$$

which yields the general constant volume-falling head equation,

$$\Delta t = -\frac{L}{A} \cdot \frac{1}{k} \cdot \frac{1}{(G_{\text{Hg}} - 1)} \cdot \frac{a_{\text{in}} \cdot a_{out}}{a_{\text{out}} + a_{\text{in}}} ln \left(\frac{\Delta h_2}{\Delta h_1} \right) \quad \text{(X1.19)}$$

or

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$$k = -\frac{L}{A}\cdot\frac{1}{\Delta t}\cdot\frac{1}{(G_{Hg}-1)}\cdot\frac{a_{in}\cdot a_{out}}{a_{out}+a_{in}}\,ln\left(\frac{\Delta h_2}{\Delta h_1}\right)$$

Noting that,

$$-\,ln\frac{\Delta h_2}{\Delta h_1} = ln\frac{\Delta h_1}{\Delta h_2}$$

the equation becomes,

$$k = \left(\frac{a_{in}\cdot a_{out}}{(a_{out}+a_{in})}\cdot\frac{1}{(G_{Hg}-1)}\right)\cdot\frac{L}{A}\cdot\frac{1}{\Delta t}\cdot ln\left(\frac{\Delta h_1}{\Delta h_2}\right) \quad \text{(X1.20)}$$

where:

k = hydraulic conductivity of the test specimen at the test temperature, m/s,
Δh_1 = total head loss across length L at the start of a permeation trial; i.e., initial total head loss, m of water,
Δh_2 = final total head loss across length L at the end of a permeation trial; i.e., final total head loss, m of water,
Δt = elapsed time during a permeation trial; i.e., $\Delta t = t_2 - t_1$, s,
a_{out} = area of the tailwater tube (tube with smaller ID), m^2,
a_{in} = area of the headwater tube, in m^2,
G_{Hg} = specific gravity of mercury (ρ_{Hg}/ρ_w) at the test temperature,
ρ_{Hg} = density of mercury at the test temperature, in Mg/m^3,
ρ_W = density of water at the test temperature, Mg/m^3,
$(G_{Hg}–1)$ = constant equal to 12.57 between 15 and 25°C,
L = length/height of the test specimen, in m,
A = area of the test specimen, in m^2,
ln = natural logarithm (base e),

and head loss equations from X1.3.1 and X1.3.2 are

$$\Delta h_1 = (\Delta H_{Hg,1} + \Delta H_{Hg,c})\cdot(G_{Hg}-1) \quad \text{(X1.21)}$$

$$\Delta h_2 = \Delta h_1 + \left((-\Delta Hg_{tail})\cdot\left(\frac{a_{out}}{a_{in}}+1\right)\cdot(G_{Hg}-1)\right) \quad \text{(X1.22)}$$

where for Δh_1 and Δh_2:

$\Delta H_{Hg,1}$ = the initial difference in height of mercury in the tailwater and headwater tubes of the manometer at the start (t_1) of a given permeation trial, m;
$\Delta H_{Hg,c}$ = the positive difference in height of mercury in the tailwater and headwater tubes of the manometer with equal pressure applied to both tubes, m, (This height differential is caused by the difference in capillary pressure of the two tubes making up the mercury U-tube manometer); and,
$-\Delta Hg_{tail}$ = the negative change in height of the mercury level in the tailwater tube of the manometer during a given permeation trial, m.

X2. RELATIONSHIP BETWEEN CHANGE IN AXIAL STRAIN AND HYDRAULIC CONDUCTIVITY OF TEST SPECIMEN

X2.1 *Introduction*—It is important to understand how hydraulic conductivity (k) changes with changes in void ratio, dry unit weight, or volume change of a given test specimen. With this understanding, one can thereby know the accuracy to which volume changes need to be controlled while testing. For instance, if k is very sensitive to volume changes then the effective consolidation stress needs to be accurately controlled; also, any volume changes during the back-pressuring process would have to be minimized.

X2.1.1 The relationships presented in this appendix are for relatively plastic clays, CL or CH, with a plasticity index greater than about 10, but less than about 50.

X2.2 *Change in Void Ratio versus Hydraulic Conductivity*—A graphical representation of how hydraulic conductivity (k) varies with void ratio (e) is presented in Fig. X2.1. This figure graphically shows that the k decreases as e decreases. In addition, for a given change in void ratio (Δe) the rate of change in k is much more dramatic in the overconsolidated range than in the normally consolidated range. The definition for the terms/notation presented in this figure are:

k= given hydraulic conductivity, m/s,
k_L= lower bound k for given percent change in k, m/s,
k_U= upper bound k for given percent change in k, m/s,
Δk_L= lower bound change in k for given percent change (decimal form) in k, m/s/
% Change = the percent change (decimal form) in k,
Δe_L= the compressive change in void ration (decreasing change)
e_U= the swelling change in void ratio (increasing change), and
m = the ratio of Δe to $\Delta log\ k = \Delta e/log\ (k/k_L)$, 1/(m/s).

X2.3 Mathematical Relationship Between e and k—It can be shown that:

$$k_L = k + (-\Delta k_L) = k - \%Change \times k = (1 - \%Change)\times k \quad \text{(X2.1)}$$

or

$$k_U = (1 + \%Change)\times k$$
$$-\Delta k_L = log(1 - \%Change) \quad \text{(X2.2)}$$

or

$$\Delta k_U = log(1 + \%Change)$$
$$e_L = e + (-\Delta e_L) \quad \text{(X2.3)}$$

or

$$e_U = e + \Delta e_U$$
$$-\Delta e_L = m \times -\Delta k_L = m \times log(1 - \%Change), \quad \text{(X2.4)}$$

or

$$\Delta e_U = m \times \Delta k_U = m \times log(1 + \%Change)$$

X2.4 Based upon the theory of elasticity, the following relationships between axial strain (ε_a) and volumetric strain (ε_v), and volumetric strain and change in void ratio ($\Delta\varepsilon$) are:

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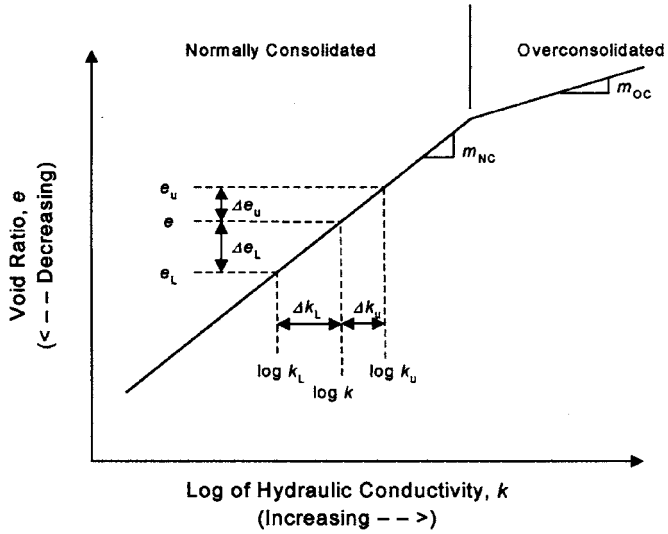

FIG. X2.1 Typical Relationship Between Void Ratio and Logarithm of Hydraulic Conductivity

$$\Delta\varepsilon_a = \frac{1}{3}\cdot E_c\cdot \Delta\varepsilon_v = \left(\frac{1}{3}\cdot E_c\cdot -\Delta\varepsilon_L\right)/(1-e_o) \qquad \text{(X2.5)}$$

$\Delta\varepsilon_a$ = the change in compressive axial strain ($\Delta L/L_o$), m/m.
E_c = a constant to correct for the non-elastic response of hydraulic-conductivity test specimens. For test specimens having a height to diameter ratio of about one, this value is about 0.8 for normally consolidated (NC) specimens and 0.6 for overconsolidated (OC) specimens,
$\Delta\varepsilon_v$ = the change in compressive volumetric strain ($\Delta V/V_o$), m^3/m^3.
$-\Delta e$ = the compressive change in void ratio; i.e., decreasing change,
e_o = the initial void ratio.

X2.5 Combining the above equations and rearranging:

$$\Delta\varepsilon_{a,NC} = \frac{-1}{3\cdot E_c}\cdot\frac{m_{NC}}{1+e_o}\cdot log(1-\%Change) \qquad \text{(X2.6)}$$

or

$$\Delta\varepsilon_{a,OC} = \frac{-1}{3\cdot E_c}\cdot\frac{m_{OC}}{1+e_o}\cdot log(1-\%Change)$$

also

$$-\%Change = 10^{-\left(3\cdot E_c\cdot\left(\frac{1+e_o}{m}\right)\cdot\Delta\varepsilon_{a,L}\right)} \qquad \text{(X2.7)}$$

$$+\%Change = 10^{-\left(3\cdot E_c\cdot\left(\frac{1+e_o}{m}\right)\cdot-\Delta\varepsilon_{a,U}\right)}$$

where:

m_{NC} = m value in normally consolidated region, 1/[m/s], and
m_{OC} = m value in the overconsolidated region, 1/[m/s].

X2.5.1 Tavenas, et al. (**10**) indicates that m is about (⅓ to ½) × e_o for normally consolidated clays. For overconsolidated clay, it is assumed that m in the overconsolidated range is reduced by the same ratio that the compression index (C) is when going from the normally consolidated region (C_{NC}) to the overconsolidated region (C_{OC}). Therefore,

$$m_{OC}/m_{NC} = C_{OC}/C_{NC} \sim 0.185 + 0.002 \times PI$$

where PI = plasticity index. This assumption is based on limited data, and in some cases, there was not any significant difference in m between the normally consolidated and overconsolidated regions. Based on the above, an initial void ratio of 0.8 and a plasticity index (PI) of 30, m_{NC} is about 0.33; while m_{OC} is about 0.082.

X2.5.2 Using the above m values and equations, and a %Change equal to 12.5 %, one could assume excess axial strains caused by a poor testing protocol should be less than the following values:

For normally consolidated soils:

$$\Delta\varepsilon_{a,NC} = \frac{-1}{3\cdot 0.8}\cdot\frac{0.33}{1.8}\cdot log\ 0.875 = 0.0044 = 0.4\ \%$$

For overconsolidated consolidated soils:

$$\Delta\varepsilon_{a,OC} = \frac{-1}{3\cdot 0.6}\cdot\frac{0.082}{1.8}\cdot log\ 0.875 = 0.00147 = 0.1\ \%$$

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SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to these test methods since the last issue, D5084–03, that may impact the use of these test methods. (Approved July 1, 2010)

(*1*) Replaced "*h*" with "Δ*h*" throughout. Deleted part of X1.1.2 and Note X1.2.

(*2*) Revised 1.8 to comply with the D18 Standards Preparation Manual.

(*3*) Revised 3.1.1 to comply with the D18 Standards Preparation Manual.

(*4*) Revised the "Mercury Warning" in 7.1 with the wording in the new ASTM "Mercury Caveat."

(*5*) Revised 8.1 to clarify the statement regarding dimensions of specimens.

(*6*) Revised 11.5.1 to ensure that any differences/averages calculated using measured values comply with the significant digit requirements in the standard.

(*7*) Rewrote Section 12 Precision and Bias, including new Tables 2 and 3.

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TEST AMERICA LAB SOPs

Title: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHODS 8260C, 8260B, AND 8260A

[Method: EPA Methods 8260C, 8260B, and 8260A]

Approvals (Signature/Date):

Technology Specialist	02/14/11 Date	Health & Safety Coordinator	02/01/11 Date
Quality Assurance Manager	02/01/11 Date	Technical Director	02/04/11 Date
Laboratory Director	02/09/11 Date		

This SOP was previously identified as SOP No. NC-MS-019, Rev 1, dated 01/07/09

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1.0 SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges, and other solid matrices.

1.2 This SOP is applicable to Methods 8260B and 8260C. It may also be used for analysis following Method 8260A.

1.3 This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 5 to 200 µg/L for 5 mL waters, 1 to 40 µg/L for low-level waters, 5 to 200 µg/kg for low-level soils, and 250 to 10,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1 and 3.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2.0 SUMMARY OF METHOD

2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2. Aqueous samples are purged directly. Soils are preserved by extracting the volatile analytes into methanol. Soil samples may also be preserved with sodium bisulfate or by freezing and purging directly.

2.3. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40°C (40°C required for low-level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components, which are detected with a mass spectrometer.

2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified

component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3.0 DEFINITIONS

3.1 Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014. The use of ultra high purity gases, prepurged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases, the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination. Refer to SOP NC-QA-020 for additional information on holding blanks.

4.3. Matrix interferences may be caused by non-target contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially on an autosampler. Whenever an unusually concentrated sample is analyzed, it must be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered, the sample must be diluted.

5. SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately. Cut-resistant gloves MUST be worn when opening VOA vials and when doing any other task that presents a strong possibility of getting cut.

5.3. Primary Materials Used

5.3.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium bisulfate	Irritant	None	Causes mild to severe irritation to the eyes. Prolonged exposure may cause burn if not flushed with water. May cause mild irritation to skin. Prolonged exposure may cause burn if not flushed with water.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. It is recommended that analysts break up work tasks to avoid repetitive motion tasks, such as opening a large number of vials or containers in one time period.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with a sticker that reads "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made. MS VOA samples may be prepared outside of the hood, unless it is known that concentrations are high.

5.6. The preparation of standards and reagents must be conducted in a fume hood with the sash closed as far as the operations will permit. MS VOA standards may be prepared outside of the hood due to low concentrations of analytes.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the laboratory Group Leader.

5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the TestAmerica Corporate Environmental Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by annual refresher training.

5.9. Specific Safety Concerns or Requirements

5.9.1. The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.9.2. The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.9.3. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.9.4. Sodium bisulfate creates Sulfuric Acid when mixed with water.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 µL and larger

6.2. Syringe: 5, 25, or 50 mL glass with luerlok tip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.01 g

6.4. Glassware

6.4.1. Vials: 20 and 40 mL with screw caps and Teflon® liners.

6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipettes: Pasteur, 5 ¾ in.

6.7. pH paper: Wide range, pH 0-14.

6.8. Gases

6.8.1. Helium: Ultra high purity, gr. 5, 99.999%.

6.8.2. Nitrogen: Ultra high purity from cylinders or gas generators may be used as an alternative to helium for purge gas.

6.9. Purge and Trap Device. The purge and trap device consists of the sample purger, trap, and desorber.

6.9.1. Sample Purger. The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low-level soils are purged directly from a VOA vial.

6.9.2. Trap. A variety of traps may be used, depending on the target analytes required. One of the traps used is the Vocarb 3000 trap. Other traps such as the OI 10 may be used if the Quality Control criteria are met. Refer also to instrument operating manuals located within the laboratory.

6.9.3. Desorber. The desorber must be capable of rapidly heating the trap to at least 180°C. Many such devices are commercially available.

6.9.4. Sample Heater. A heater capable of maintaining the purge device at 40°C is necessary for low-level soil analysis.

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6.10. Gas Chromatograph/Mass Spectrometer System

6.10.1. Gas Chromatograph. The gas chromatograph (GC) system must be capable of temperature programming.

6.10.2. Gas Chromatographic Columns. Capillary columns are used. Some typical columns are listed below:

6.10.2.1. Column 1. 20m x 0.18 ID DB-624 with 1 µm film thickness.

6.10.2.2. Mass Spectrometer. The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.10.3. GC/MS Interface. In general, direct introduction to the mass spectrometer is used but any interface that achieves all acceptance criteria may be used.

6.10.4. Data System. A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The NIST/EPA mass spectral library must be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methanol. Purge and Trap grade, high purity

7.1.2. Reagent Water. High purity water that meets the requirements for a method blank when analyzed (see Section 9.4). Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.1.3. Hydrochloric Acid – (1:1 v/v). Reagent grade or equivalent

7.1.4. Sodium bisulfate. Reagent grade or equivalent

7.2. Standards

7.2.1. Calibration Standard

7.2.1.1. Stock Solutions. Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon®-sealed screw-cap bottles with minimal headspace at -10° to -20°C. Note that standard/spiking concentrations or vendors are subject to change.

7.2.1.2. Working standards. A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20% then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem, then a new initial calibration must be performed.

7.2.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.1.4. If stock or secondary dilution standards are purchased in sealed ampoules, they may be used up to the manufacturer's expiration date.

7.2.1.5. Additional information can be found in SOP NC-QA-017.

7.2.2. Internal Standards. Internal standards are added to all samples, standards, and blank analyses. Refer to Table 5 for internal standard components.

7.2.3. Surrogate Standards. Refer to Table 6 for surrogate standard components and spiking levels.

7.2.4. Laboratory Control Sample Spiking Solutions. Refer to Table 7 for LCS components and spiking levels.

7.2.5. Matrix Spiking Solutions. The matrix spike contains the same components as the LCS. Refer to Table 7.

7.2.6. Tuning Standard. A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 50 ng/µL of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

7.2.7. All standard preparation information is detailed in the Standard Logbook.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection to analysis.

8.2. For DoD samples, water samples are normally preserved at pH $\leq$ 2 with 1:1 hydrochloric acid. Unpreserved water samples must be analyzed within seven days of sampling.

8.3. Solid samples are field preserved with sodium bisulfate solution or by freezing upon receipt at the laboratory for low-level analysis, or with methanol for medium-level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Analysis must be completed 14 days from sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore™ sample. (The 5g or 25g sampler can be used, depending on client preference). Following shipment back to the lab, the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes), then it will be necessary to use two additional 5g EnCore™ samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore™ samplers

8.5.1. Ship one 5g (or 25g) EnCore™ sampler per field sample position.

8.5.2. An additional 2 oz plastic bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25g sample, 5 µL for a nominal 5g sample.) Refer to Section 17.2 for Michigan project criteria.

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 500 µL of 50 µg/mL solution for a nominal 25 g sample, 100 µL for a nominal 5g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25g sample or 5 mL methanol for a nominal 5g sample. Refer to Section 17.2 for Michigan project criteria.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500 µL of spike to 25 mL methanol or 100 µL spike to 5 mL methanol). Refer to Section 17.2 for Michigan project criteria.

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6. Sample collection for medium-level analysis using field methanol preservation

8.6.1. Prepare a 2-oz sample container by adding 25 mL purge and trap grade methanol. (If a 5g sample is to be used, add 5 mL methanol to a 2 oz container or VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g, and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional 2 oz plastic bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25g sample, 5 µL for a nominal 5g sample.) Refer to Section 17.2 for Michigan project criteria.

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 500 µL of 50 µg/mL solution for a nominal 25g sample, 100 µL for a nominal 5g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25g sample or 5 mL methanol for a nominal 5g sample. Refer to Section 17.2 for Michigan project criteria.

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8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500 µL of spike to 25 mL methanol or 100 µL spike to 5 mL methanol). Refer to Section 17.2 for Michigan project criteria.

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.7. Low-level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg), low-level soil preservation must be used. However, it is also necessary to take a sample for the medium-level (field methanol preserved or using the EnCore™ sampler) procedure in case the concentration of analytes in the soil is above the calibration range of the low-level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore™ sampling device and returned to the lab. It is recommended that two EnCore™ samplers be used for each field sample position to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials for sodium bisulfate preservation by adding a magnetic stir bar, approximately 1g of sodium bisulfate, and 5 mL of reagent water. Prepare vials for preservation by freezing by adding a stir bar and 5 mL reagent water.

8.7.5. Seal and label the vial. It is strongly recommended that the vial is labeled with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction. The label absolutely must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.01g, and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore™ sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil, and note on the label.

Note: Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at <-10°C within 48 hours. The sample must be analyzed within 14 days after sampling and stored at a 45 degree angle in the freezer.

8.7.8. Alternatively the sodium bisulfate preservation may be performed in the field. This is not recommended because of the many problems that can occur in the field setting.

Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional 2 oz plastic bottle with no preservative for percent moisture determination, and an additional VOA vial preserved with methanol for the medium level procedure. Depending on the type of soil, it may also be necessary to ship vials with no or extra preservative.

8.8. Unpreserved Soils

8.8.1. At specific client request, unpreserved soils packed into glass jars or brass tubes may be accepted and sub-sampled in the lab. This is the old procedure based on Method 5030A and Method 8260A. It is no longer included in SW846 and is likely to generate results that are biased low, possibly by more than an order of magnitude.

8.9. Aqueous samples are stored in glass containers with Teflon®-lined septa at $4^{o}C \pm 2^{o}C$ with minimum headspace.

8.10. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14-day holding time. However, they should be analyzed as soon as possible. The lack of preservation must be addressed in the case narrative). Maximum holding time for the EnCore™ sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.11. A holding blank is stored with the samples. This is analyzed weekly. It is replaced every seven days.

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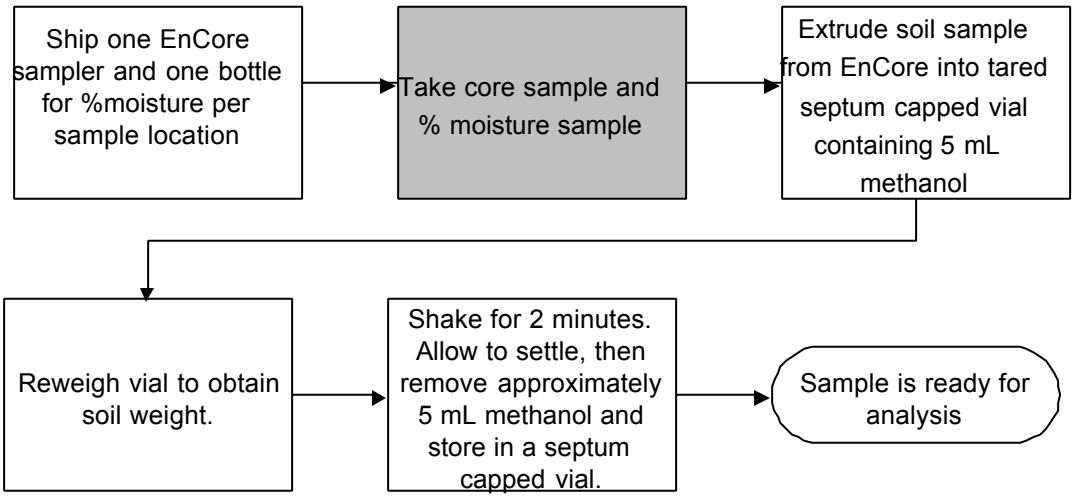
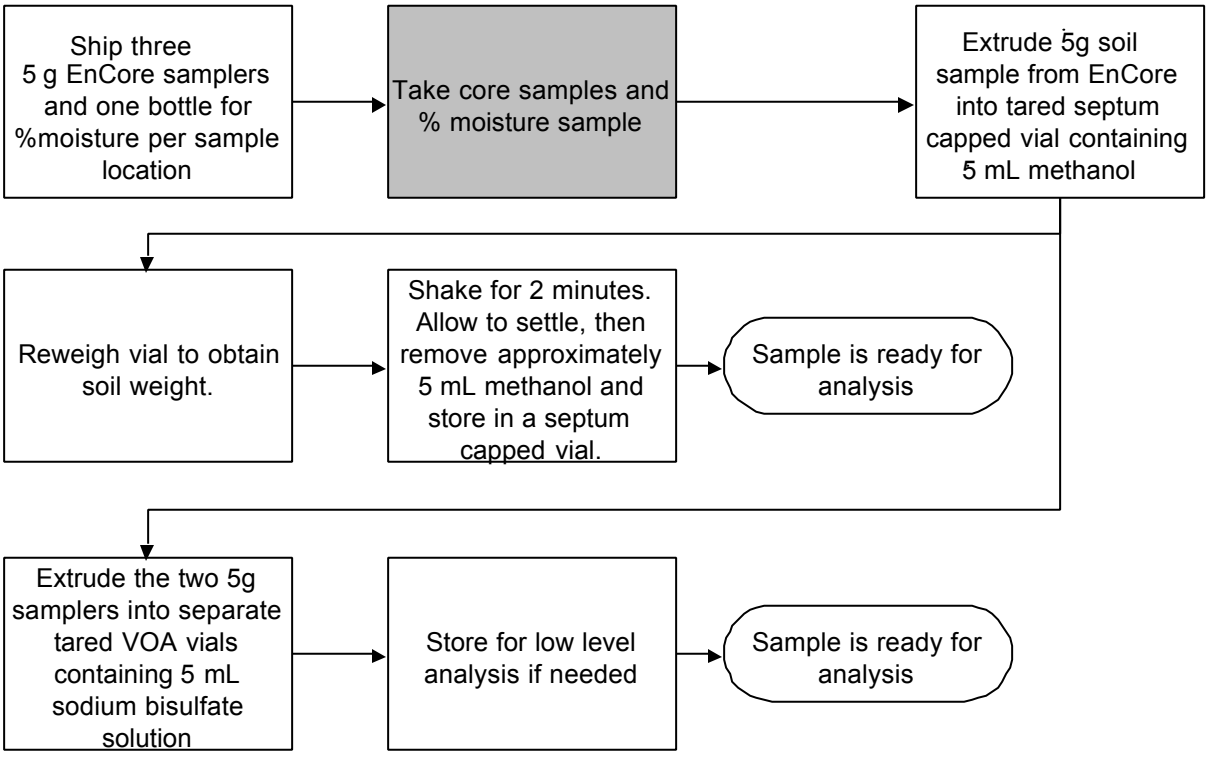
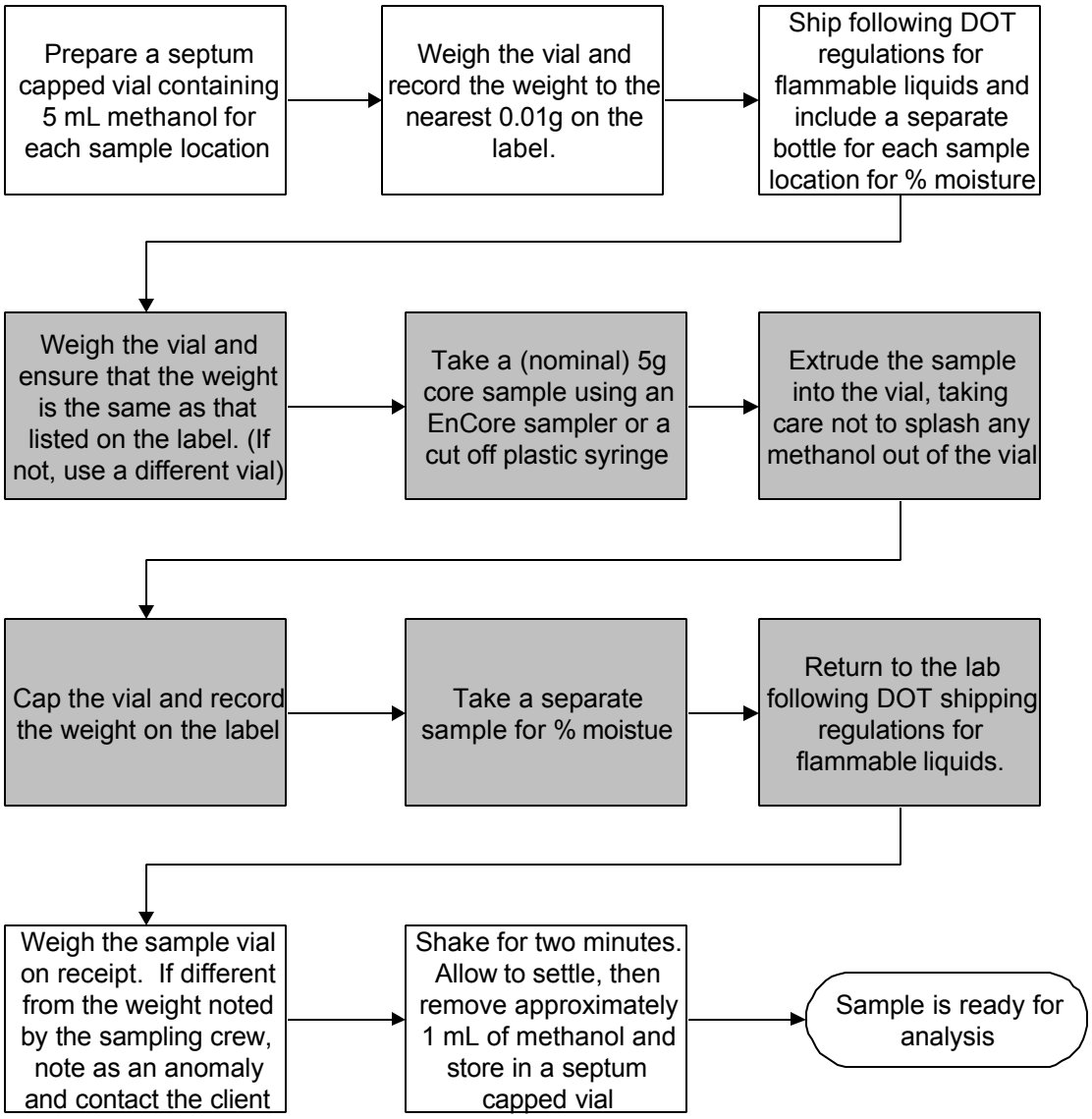
EnCore procedure when low level is not required (field steps in gray)



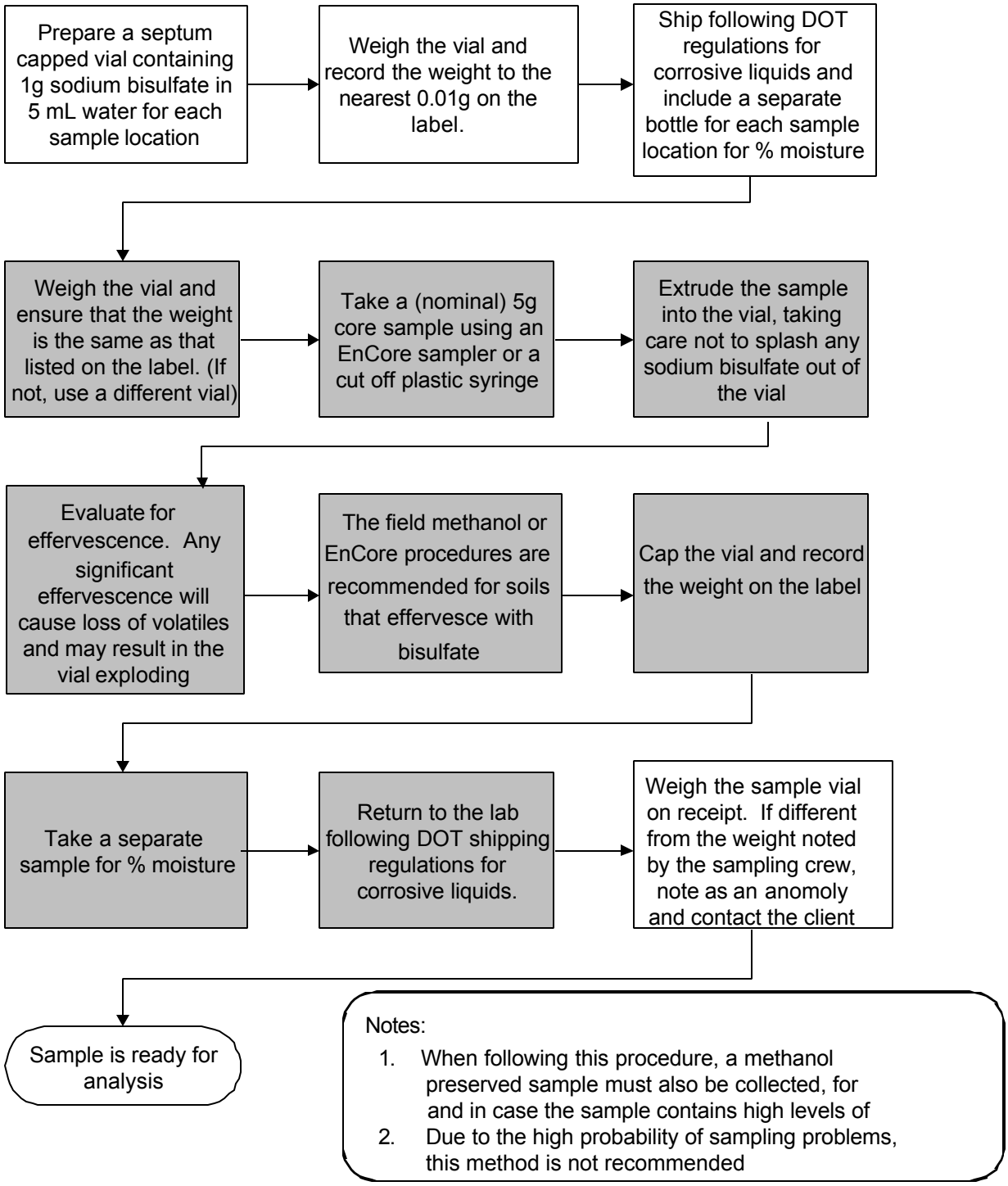
EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



Field bisulfate preservation procedure (field steps in gray)



9. QUALITY CONTROL

9.1. Batch

9.1.1. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort must be made to keep the samples together.

9.1.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Refer to the TestAmerica QC program document (QA-003) for further details of the batch definition.

9.2. Control Limits

9.2.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.2.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs (QC Browser program).

9.3. Surrogates

9.3.1. Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 6. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure instrument performance is acceptable.
- Recalculate the data and/or re-analyze if either of the above checks reveal a problem.
- Reprepare and re-analyze the sample if there is sufficient volume. If there is insufficient volume, the surrogate is narrated.

It is only necessary to reprepare/re-analyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

9.3.2 If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then re-analysis or flagging of the data is required. For Ohio VAP samples, all surrogates must be in control, or samples must be reprepared and re-analyzed.

Note: For Ohio VAP and DoD samples, all surrogates must be within acceptance criteria. The exceptions for Ohio VAP are as follows:
(a) insufficient sample for re-extraction, or (b) the surrogates are biased high and the samples are non-detect.

9.3.3 For concrete matrix, Dibromofluoromethane may have poor recovery in samples and matrix spikes. If the surrogate does not meet criteria, no further action is required due to matrix.

9.3.4 Refer to the TestAmerica QC Program document (QA-003) for further details of the corrective actions.

9.4 Method Blanks

9.4.1 For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles, the method blank consists of reagent water. For medium-level volatiles, the method blank consists of the same volume of methanol that was used to prepare the samples. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below). The method blank is acceptable if any compound detected in the blank is present in the associated samples at ten times the blank level. For Ohio VAP work, there can be no target analyte greater than the RL in the method blank unless the sample result is ND. All samples associated with an unacceptable blank will be reprepared and re-analyzed.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone), the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

- Re-analysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers.

9.4.2 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client must take place. For Ohio VAP samples, all surrogates must be in control, or repreparation of the batch is required.

9.4.3 If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.4.4 Refer to the TestAmerica QC Program document, Policy QA-003, for further details of the corrective actions.

9.4.5 Refer to SOP NC-QA-016 for further details concerning DoD Project Work.

9.5 Laboratory Control Samples (LCS)

9.5.1 For each batch of samples, analyze an LCS. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS contains a representative subset of the analytes of interest (see Table 7), and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and re-analysis of the batch. For Ohio VAP samples, all surrogates must be in control on the LCS, or repreparation and re-analysis of the batch is required. The exceptions are as follows: (a) insufficient sample for repreparation, (b) expired holding times, or (c) the LCS is biased high and the samples are non-detect for those analytes.

- If the batch is not re-extracted and re-analyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.

- If re-extraction and re-analysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.2 Refer to the TestAmerica QC Program document (Policy QA-003) for further details of the corrective action.

9.5.3 If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be

expected statistically. These requirements must be negotiated with the client. n-Hexane must be spiked and reported for the LCS for Ohio VAP samples.

9.5.4 If full analyte spike lists are used at the client request, it is possible some compounds in the LCS may interfere with each other. In that case, the lab will quantitate those compounds in the LCS with a secondary ion which is free from interferences.

9.6 Matrix Spikes

9.6.1 For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 7. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory-specific, historically-generated limits.

9.6.2 If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

9.6.2.1 If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-analysis of the batch.

9.6.2.2 If an MS/MSD is not possible due to limited sample, then an LCS duplicate may be analyzed, if required by specific clients or program.

9.6.2.3 The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7 Nonconformance and Corrective Action

9.7.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluoro-

benzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system.

10.1.2. General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	To give at least 5 scans/peak, but not to exceed 2 seconds/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 0.4 – 0.6 mL/minute

10.2 Gas chromatograph suggested temperature program

10.2.1 BFB Analysis

Initial Temperature:	100°C
Initial Hold Time:	0.1 minute
Temperature Program:	20°C/minute
Final Temperature:	200°C

10.2.2 Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	2minutes
Temperature Program:	15°C/minute
Final Temperature:	200°C
Final Hold Time:	3 minutes

10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 8 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each 12-hour time period. The 12-hour time period begins at the moment of injection of BFB.

10.4. Initial Calibration

10.4.1. A series of at least five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Six standards must be used for a quadratic least squares calibration. Suggested calibration levels for a 5 mL purge are: 5, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Suggested calibration levels for a low level 5mL purge are 1, 5, 10, 20, and 40 µg/L. Again, some analytes are prepared at higher levels. Tables 2, 2A, and 4 list the calibration levels for each analyte. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. (For example, adequate sensitivity can be obtained by using a 5 mL purge volume to reach the same reporting limits that once required a 25 mL purge. The calibration levels will still be the same 1, 5, 10, 20, 40 µg/L.) However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

NOTE: For Method 8260C. Historically the surrogate compounds have been included in the multi-point initial calibration at variable concentrations in order to evaluate the linear response as with any target analyte. However, with improvements in instrumentation and more reliance on the autosampler, an option is available depending on the project-specific data quality requirements for allowing the autosampler (or using a manual technique) to spike the initial calibration standards with surrogates in the same manner as the samples are spiked. With this option the surrogate standards in the initial calibration can be
averaged to develop a response factor and an effective one point calibration with the sole purpose to measure the surrogate recovery using the same concentration for each sample analysis. For this calibration option the surrogate linear response is less important, since multiple concentrations of surrogates are not being measured. Instead, the surrogate concentration remains constant throughout and the recovery of this known concentration can easily be attained without demonstrating if the response is linear.
Under a second calibration option, the surrogates can be calibrated in the same manner as the target analytes, however, the laboratory should have the latitude to employ either option given the instrument system limitations and the ability to meet the project's data quality objectives.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests.

10.4.3. Internal standard calibration is used. The internal standards are listed in Table 5. Target compounds must reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area

response of the characteristic ions against the concentration for each compound and internal standard. See Table 12 for a list of characteristic ions. See Equation 1, Section 12, for calculation of response factor.

10.4.4. For Method 8260B, the % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 11 for the CCCs. This criteria must be met before sample analysis begins.

10.4.4.1. Calibration Check Compound (CCC) (Method 8260B only)

10.4.4.1.1. CCCs are a representative group of compounds, which are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

10.4.4.2. System Performance Check Compounds (SPCC) (Method 8260B only)

10.4.4.2.1 SPCCs are compounds, which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 9 for the SPCC compounds for Method 8260B and required minimum response factors. Refer to Table 10 for the recommended minimum relative response factor criteria for initial and continuing calibration verification for Method 8260C.

10.4.6. Weighting of Data Points

10.4.6.1. In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and must be used if the data system has this capability. The Y-intercept is evaluated to determine calibration acceptability.

10.4.7. For any analyte with % RSD >15%, linear or quadratic curve fits may be used if the compounds have historically exhibited a non-linear response. The analyst must consider instrument maintenance to improve the linearity of response. Nonlinear calibration models cannot be used to extend the calibration range for compounds that normally exhibit a linear response, but in a narrower calibration range. If the % RSD is > 15%, the analyst may drop the low or high in the ICAL , as long as a minimum of five points are maintained (six points for quadratic) and the quantitation range is adjusted accordingly. Otherwise, the coefficient of determination r^2 must be $\geq$ 0.990. For Method 8260C, % RSD is ± 20% for each target analyte.

10.4.8. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9. The calibration standards for the initial five-point calibration for low-level soils that are not preserved in sodium bisulfate (i.e., are preserved by freezing or not preserved) must be heated to 40°C for purging. Using this calibration curve for water samples is acceptable as long as all calibration, QC, and samples are also heated to 40°C. A separate five-point calibration must be prepared for analysis of low level soils that are preserved with sodium bisulfate. Low-level soils analysis requires the use of a closed vial autosampler such as the Varian Archon, O.I. 4552 or Tekmar Precept. Each standard for analysis of sodium bisulfate preserved samples is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water and 1g sodium bisulfate. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging.

10.4.10. Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five-point initial calibration. If the analyte is detected in any of the samples, a five-point initial calibration must be generated and the sample(s) re-analyzed for quantitation. However, if the analyte is not detected, the non-detect must be reported and no further action is necessary.

Note: This procedure must not be used for Ohio VAP samples.

10.4.11. Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after the initial calibration. For Method 8260B, the recovery for CCC compounds must be ≤ 20%. The recovery for non-CCC compounds must be ≤ 50% with an allowance of up to six compounds > 50%.

10.4.11.1 For Method 8260C, the acceptance criteria is 70-130% for each target analyte.

10.5. Continuing Calibration. The initial calibration must be verified every 12 hours.

10.5.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. A midpoint calibration standard is used as the continuing calibration.

10.5.2. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in Equation 4, Section 12.3.4.

10.5.3. For Method 8260B, the % drift of the CCCs must be $\leq$ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 9. In addition, the percent drift of all analytes must be $\leq$ 50% with allowance for up to six target analytes to have percent drift > 50%.

10.5.3.1. For Method 8260C, all compounds of interest must be verified at 20%.

10.5.3.2. Refer to Table 11 for specific Ohio VAP analytes.

10.5.4. If the CCCs and/or the SPCCs do not meet the criteria in Section 10.5.3 and Table 9, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action such as a different type of column will require a new initial calibration. For Method 8260C, any sample non-detects for an analyte that fails the SOP criteria low, must have a low level CCV (CCV at the RL) in the batch as a sensitivity demonstration. The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detect samples to be reported without flagging.

10.5.5. Once the above criteria have been met, sample analysis may begin. **Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs.** Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

11. PROCEDURE

11.1. Procedural Variations

11.1.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation must be completely documented using a Nonconformance Memo and approved by a Supervisor or Group

Leader and QA Manager. The Nonconformance Memo must be filed in the project file.

11.1.2. Any unauthorized deviations from this procedure must also be documented as a non-conformance with a cause and corrective action described. The laboratory may not deviate from the method for Ohio VAP samples.

11.2. Preliminary Evaluation

11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.3. Sample Analysis Procedure

11.3.1. All analysis conditions for samples must be the same as for the continuing calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain an MS/MSD, an LCS, and a method blank. See Section 9.4 for method blank preparation.

11.3.2.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. However, if any re-tuning of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new batch must be started. For medium-level soils, the batch is defined at the sample preparation stage.

11.3.2.2. It is not necessary to re-analyze batch QC with re-analyses of samples. However, any reruns must be part of a valid batch.

11.3.3 Dilutions must be done just prior to the GC/MS analysis of the sample. Dilutions are made in a Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 1 µL of sample, then serial dilutions must be made in volumetric flasks. Dilutions may also be prepared in a 40 mL vial. An appropriate amount of water is added to the vial. The sample is added using an appropriate syringe.

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11.3.3.1 The diluted concentration is to be estimated to be in the upper half of the calibration range.

11.4. Methanol Extract Soils

11.4.1 Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Sections 8.5 or 8.6) to the syringe. If less than 1µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1µL will be added to the water in the syringe. Refer to Section 17.2 for Michigan project requirements.

11.5. Liquid wastes that are soluble in methanol and insoluble in water.

11.5.1 Pipette 1 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1g.

11.5.2 Quickly add 4 mL of methanol, then add 5µL of a 2500 µg/mL surrogate spiking solution to bring the final volume to 5 mL. Cap the vial and shake for two minutes to mix thoroughly. For an MS/MSD or LCS, 4.9 mL of methanol, 5µL of a 2500 µg/mL surrogate spiking solution, and 0.1 mL of matrix spike solution is used.

11.5.3 Rinse a gas-tight syringe with organic-free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Sections 8.5 or 8.6) to the syringe. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1µL will be added to the water in the syringe.

11.6. Aqueous and low-level soil sample analysis (Purge and Trap units that sample directly from the VOA vial)

11.6.1 Units which sample from the VOA vial must be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.6.2 If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise, the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to be analyzed as soils.

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11.6.3 Soil samples, which are preserved with sodium bisulfate, must be quantitated against a curve prepared with standards containing about the same amount of sodium bisulfate as the samples (1g in 5 mL).

11.6.4 Soil samples, which are preserved by freezing, must be allowed to thaw completely before sample analysis begins.

11.6.5 Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.7 Water Samples Not Directly Sampled from VOA Vials

11.7.1. All samples and standard solutions must be at ambient temperature before analysis.

11.7.2. Fill a syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is $\geq$ 5 µL. Check and document the pH of the remaining sample.

11.7.3. Add 50 ng of each internal and surrogate standard. The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 10 µg/L solution for a 5 mL sample). Inject the sample into the purging chamber. The internal and surrogate standards can be added automatically by the autosampler.

11.7.3.1. For TCLP samples, use 1 mL of TCLP leachate with 4 mL reagent water. (Note: TCLP reporting limits will be five times higher than the corresponding aqueous limits.)

11.7.4. Purge the sample for 11 minutes (trap must be below 35°C).

11.7.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for approximately 3-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.7.6. Desorb and bake time and temperature are optimized for the type of trap in use. The same conditions must be used for samples and standards.

11.8. Low-Level Solids Analysis using discrete autosamplers, Methods 8260A and 5030A

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated soil/sediment sample mixed with reagent water containing the surrogates and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.8.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.8.2. Weigh out 5g (or other appropriate aliquot) of sample into a 40 mL vial. Record the weight to the nearest 0.1g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 0.5g. If the sample is contaminated with analytes such that a purge amount less than 0.5g is appropriate, use the medium level method. For the medium level method, add 5g soil to 5 mL methanol containing the surrogates, mix for two minutes, allow to settle, then remove a portion of the methanol, and store in a clean Teflon®-capped vial at 4°C until analysis. Analyze as described in Section 11.5.

11.8.3. Add 5 mL of organic free water to the VOA vial. Add surrogate/internal standard (and matrix spike solutions if required.). Add directly to the sample from Section 11.5.1.

11.8.4. The above steps must be performed rapidly and without interruption to avoid loss of volatile organics.

11.9 Medium-Level Soil/Sediment and Waste Samples

11.9.1. Sediments/soils and waste that are insoluble in methanol.

11.9.1.1 Weigh 5 g (wet weight) into a tared vial. Use a top-loading balance. Record the weight to 0.1 gram. Do not discard any supernatant liquids.

11.9.1.2 Quickly add 5 mL of methanol, and 5µL of 2500 µg/mL surrogate spiking solution to bring the final volume of methanol to 5 mL. For an LCS or MS/MSD sample, add 4.9 mL of methanol, 5µL of surrogate spike solution, and 0.1 mL of matrix spike solution. Cap the vial and shake or vortex to mix thoroughly.

Note: Sections 11.9.1.1 and 11.9.1.2 must be performed rapidly and without interruption to avoid the loss of volatile organics.

11.10. Initial review and corrective actions

11.10.1. If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Re-analysis of samples analyzed while the system was malfunctioning is required.

11.10.2. If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Re-analysis of samples analyzed while the system was malfunctioning is required. Re-analysis must be undiluted if matrix interference is not observed.

11.10.2.1. Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. If the change in sensitivity is a matrix effect, the sample is re-analyzed to confirm. If the change in sensitivity is due to instrumental problems, all affected samples must be re-analyzed after the problem is corrected. For Ohio VAP projects, the laboratory will re-analyze any sample where the internal standard fails, and there is no evidence of matrix interference. If there is no matrix interference, the sample must be reanalyzed at the original dilution. If the internal standard is within criteria, report the second analysis. If the internal standard is still outside of criteria, the sample must be analyzed at a second dilution. If the internal standard still does not meet criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required..

11.11. Dilutions

11.11.1 If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution must be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be re-analyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.11.2 Guidance for Dilutions Due to Matrix

11.11.2.1 If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample

must be re-analyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement.

11.11.3 Reporting Dilutions

11.11.3.1 The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component, and (2) correspondence of the sample component and the standard component characteristic ions. See Table 12 for a list of the characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

12.1.1.1 The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same 12 hours as the sample.

12.1.1.2 The relative intensities of ions must agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst must report that identification and proceed with quantitation.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) must be present in the sample spectrum.

12.2.1.2. The relative intensities of the major ions must agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3. Molecular ions present in the reference spectrum must be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of coeluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum must be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6. Computer-generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches must the analyst assign a tentative identification.

12.3. Calculations

12.3.1. Response Factor (RF)

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard, ng

C_x = Concentration of the compound being measured, ng

12.3.2. Standard Deviation (SD)

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

Where:

X_i = Value of X at i through N

N = Number of points

X = Average value of X_I

12.3.3. Percent Relative Standard Deviation (%RSD)

Equation 3

$$\%RSD = \frac{\text{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$ = **Mean of RF values in the curve**

12.3.4. Percent Drift Between the Initial Calibration and the Continuing Calibration

Equation 4

$$\% \text{ Drift} = \frac{C_{expecte} - C_{found}}{C_{expecte}} \times 100$$

Where:

$C_{expecte}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.5. Target Compound and Surrogate Concentrations

12.3.5.1 Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average

response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is $\leq$ 15%.

12.3.5.2 Calculation of Concentration Using Average Response Factors

Equation 5

$$\textbf{Concentration } mg/L = \frac{x}{RF}$$

12.3.5.3 Calculation of Concentration using Linear Fit

Equation 6

$$\textbf{Concentration } mg/L = A + Bx$$

12.3.5.4. Calculation of Concentration Using Quadratic Fit

Equation 7

$$\textbf{Concentration } mg/L = A + Bx + Cx^2$$

Where:

x is defined in Equations 8, 9, and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

12.3.5.5. Calculation of ***x*** for Water and water-miscible waste:

Equation 8

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

X = ug/L

A_x = Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion)

A_{is} = Area of the characteristic ion for the internal standard

I_s = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

V_o = Volume of water purged, mL

12.3.5.6. Calculation of ***x*** for Medium level soils:

Equation 9

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

X = ug/kg

A_x, I_s, D_f, A_{is}, same as for water

V_t = Volume of total extract, mL

V_a = Volume of extract added for purging, µL

W_s = Weight of sample extracted, g

$$D = \frac{100 - \%\text{moisture}}{100}$$

12.3.5.7. Calculation of ***x*** for Low level soils:

Equation 10

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

X = ug/kg

A_x, I_s, A_{is}, same as for water

D = as for medium level soils

W_s = Weight of sample added to the purge vessel, g

12.3.5.8. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area in the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

In other words, the concentration is equal to *x* as defined in Equations 8, 9, and 10.

12.3.6. MS/MSD Recovery

Equation 11

$$\text{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spike Sample result

SR = Sample Result

SA = Spiked amount

12.3.7. Relative % Difference calculation for the MS/MSD:

Equation 12

$$\text{RPD} = \frac{|\text{MSR - MSDR}|}{\frac{1}{2}(\text{MSR + MSDR})} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

12.4 Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002.

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.1.1. Generally, each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is defined in QA SOPs NC-QA-021 and CA-Q-S-006. When non-standard compounds are analyzed at client request, lesser requirements are possible with client agreement. At a minimum, a standard at the reporting limit must be analyzed to demonstrate the capability of the method. The non-standard compound must be detected in the reporting limit standard to be acceptable.

13.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of a standard at the reporting limit and a single point calibration.

13.2. Initial Demonstration

13.2.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.3. Training Qualification

13.3.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.3.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

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14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

15.2. All waste will be disposed of in accordance with Federal, State, and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.3. The following waste streams are produced when this method is carried out.

15.3.1. **Acidic material from the auto-sampler**: Waste stream must be collected and neutralized before discharge to a sewer system if the pH is less than 5.

15.3.2. **Methanol waste from rinses and standards:** Methanol waste is discarded as a flammable liquid in a solvent waste container identified as "Flammable Liquid Waste".

15.3.3. **All samples including purged and extracted soils and waters:** Samples are collected in boxes and removed from the lab to storage. The Waste Coordinator handles crushing the vials and proper disposal.

15.3.4. **Solid samples** - Stirbars are removed from the sample. The contents of the vial are poured into a beaker, and the soil allowed to settle out. The soil is disposed of in the solid waste container.

16. REFERENCES

16.1. References

16.1.1. SW846, *Test Methods for Evaluating Solid Waste*, Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, Update III, December 1996

16.1.2. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260A, Update II, September 1994

16.1.3. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Purge-and-Trap for Aqueous Samples, Method 5030B, Rev 2, December 1996

16.1.4. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Purge-and-Trap for Aqueous Samples, Method 5030A, Rev 1, July 1992

16.1.5. SW846, *Test Methods for Evaluating Solid Waste*, Third Edition, Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035, Rev 0, December 1996

16.1.6 SW846, Test Methods for Evaluating Solid Waste Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035A, Draft Revision 1, July 2002

16.1.7 SW846, Test Methods for Evaluation Solid Waste, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Method 8260C, Revision 3, August 2006.

16.1.7 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.8 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.9 Corporate Quality Management Plan (CQMP), current version

16.1.10 Revision History

Historical File:		Revision 2.0: 12/15/97		Revision 0: 06/30/08 (NC-MS-019)
(formerly CORP-MS-0002NC)		Revision 2.1: 03/06/00		Revision 1: 01/07/09
		Revision 2.2: 11/28/00		
		Revision 2.3: 05/23/01		
		Revision 2.4: 09/27/04		
		Revision 2.5: 04/03/07		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection LimitsNC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6. Standards and Reagents, NC-QA-017

16.2.7. Laboratory Holding Blanks, NC-QA-020

16.2.8. Selection of Calibration Points, CA-T-P-002

16.2.9. Calibration Curves (General), CA-Q-S-005

16.2.10. Acceptable Manual Integration Practices, CA-Q-S-002

17. MISCELLANEOUS

17.1. Modifications from the reference method

17.1.1. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW846 in order to improve the reliability of qualitative identification.

17.2 The following are protocols that must be followed to achieve the lower reporting limits required when analyzing Michigan projects.

17.2.1 Modify Sections 8.5.4 and 8.6.8 (add 5 uL of 2500 ug/mL surrogate solution for a nominal 10g sample).

17.2.2 Modify Sections 8.5.5 and 8.6.9 (add 100 uL of 50 ug/mL spike solution for a nominal 10g sample).

17.2.3 Modify Sections 8.5.6 and 8.6.10 (add 100 uL of 50 ug/mL spike solution for a nominal 10g sample).

17.2.4 Michigan reporting limits for methanol preserved soils are achieved by injecting 100 uL of the methanol extract in a 5 mL purge. The instrument is calibrated using the recommended calibration range for water of 0.5 ug/L to 100 ug/L. Some analytes are prepared at higher concentrations.

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Table 1 - TestAmerica Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Dichlorodifluoromethane	75-71-8	5	1	5	250	250
Chloromethane	74-87-3	5	1	5	250	250
Bromomethane	74-83-9	5	1	5	250	250
Vinyl chloride	75-01-4	5	1	5	250	250
Chloroethane	75-00-3	5	1	5	250	250
Trichlorofluoromethane	75-69-4	5	1	5	250	250
Acrolein	107-02-8	100	20	100	5,000	5,000
Acetone	67-64-1	20	10	20	1,000	1,000
Trichlorotrifluoroethane	76-13-1	5	1	5	250	250
Iodomethane	74-88-4	5	1	5	250	250
Carbon disulfide	75-15-0	5	1	5	250	250
Methylene chloride	75-09-2	5	1	5	250	250
tert-Butyl alcohol	75-65-0	200	50	200	10,000	10,000
1,1-Dichloroethene	75-35-4	5	1	5	250	250
1,1-Dichloroethane	75-34-3	5	1	5	250	250
Trans-1,2-Dichloroethene	156-60-5	5.0	1.0	5.0	250	250
Acrylonitrile	107-13-1	100	20	100	5,000	5,000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	5	1	5	250	250
Hexane	110-54-3	5	1	5	250	250
cis-1,2-Dichloroethene	156-59-2	5	1	5	250	250
1,2-Dichloroethene (Total)	540-59-0	10	2	10	500	500
Tetrahydrofuran	109-99-9	20	5	20	1,000	1,000
Chloroform	67-66-3	5	1	5	250	250
1,2-Dichloroethane	107-06-2	5	1	5	250	250
Dibromomethane	74-95-3	5	1	5	250	250
2-Butanone	78-93-3	20	5	20	1,000	1,000
1,4-Dioxane	123-91-1	500	200	500	25,000	25,000
1,1,1-Trichloroethane	71-55-6	5	1	5	250	250
Carbon tetrachloride	56-23-5	5	1	5	250	250
Bromodichloromethane	75-27-4	5	1	5	250	250
1,2-Dichloropropane	78-87-5	5	1	5	250	250
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250	250
Trichloroethene	79-01-6	5	1	5	250	250
Dibromochloromethane	124-48-1	5	1	5	250	250
1,2-Dibromoethane	106-93-4	5	1	5	250	250
1,2,3-Trichloropropane	96-18-4	5	1	5	250	250

Table 1 - TestAmerica Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
1,1,2-Trichloroethane	79-00-5	5	1	5	250	250
Benzene	71-43-2	5	1	5	250	250
Ethylmethacrylate	97-63-2	5	1	5	250	250
Trans-1,3-Dichloropropene	10061-02-6	5	1	5	250	250
Bromoform	75-25-2	5	1	5	250	250
4-Methyl-2-pentanone	108-10-1	20	5	20	1000	1,000
2-Hexanone	591-78-6	20	5	20	1000	1,000
Tetrachloroethene	127-18-4	5	1	5	250	250
Toluene	108-88-3	5	1	5	250	250
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250	250
2-Chloroethyl vinyl ether	110-75-8	N/A[2]	N/A	50	1000	1,000
Vinyl acetate	108-05-4	10	2	10	500	500
Chlorobenzene	108-90-7	5	1	5	250	250
Ethylbenzene	100-41-4	5	1	5	250	250
Styrene	100-42-5	5	1	5	250	250
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	250	250
m and p Xylenes		10	2	10	500	500
o-xylene	95-47-6	5.0	1	5	250	250
Total xylenes	1330-20-7	10	2	10	500	500
1,3-Dichlorobenzene	541-73-1	5	1	5	250	250
1,4-Dichlorobenzene	106-46-7	5	1	5	250	250
1,2-Dichlorobenzene	95-50-1	5	1	5	250	250
2,2-Dichloropropane	590-20-7	5	1	5	250	250
Bromochloromethane	74-97-5	5	1	5	250	250
1,1-Dichloropropene	563-58-6	5	1	5	250	250
Bromodichloromethane	75-27-4	5	1	5	250	250
1,2-Dichloropropane	78-87-5	5	1	5	250	250
1,3-Dichloropropane	142-28-9	5	1	5	250	250
Isopropylbenzene	98-82-8	5	1	5	250	250
Bromobenzene	108-86-1	5	1	5	250	250
n-Propylbenzene	103-65-1	5	1	5	250	250
2-Chlorotoluene	95-49-8	5	1	5	250	250
4-Chlorotoluene	106-43-4	5	1	5	250	250
1,3,5-Trimethylbenzene	108-67-8	5	1	5	250	250
Tert-Butylbenzene	98-06-6	5	1	5	250	250
1,2,4-Trimethylbenzene	95-63-6	5	1	5	250	250
Sec-butylbenzene	135-98-8	5	1	5	250	250

Table 1 - TestAmerica Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
4-Isopropyltoluene	99-87-6	5	1	5	250	250
n-Butylbenzene	104-51-8	5	1	5	250	250
1,2,4-Trichlorobenzene	120-82-1	5	1	5	250	250
Napthalene	91-20-3	5	1	5	250	250
Hexachlorobutadiene	87-68-3	5	1	5	250	250
1,2,3-Trichlorobenzene	87-61-6	5	1	5	250	250
Acetonitrile	75-05-8	100	20	100	5000	500
Cyclohexane	110-82-7	10	1	10	500	500
Methyl Acetate	79-20-9	10	10	10	500	500
Methyl cyclohexane	108-87-2	10	1	10	500	500

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 2 - TestAmerica Primary Standard Calibration Levels, 5 mL purge Solid

Compound	Calibration Level ug/kg				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dichloroethane-d4 (Surrogate)	5	20	50	100	200
Toluene-d8 (Surrogate)	5	20	50	100	200
4-Bromofluorobenzene (Surrogate)	5	20	50	100	200
Dichlorodifluoromethane	5	20	50	100	200
Chloromethane	5	20	50	100	200
Bromomethane	5	20	50	100	200
Vinyl chloride	5	20	50	100	200
Chloroethane	5	20	50	100	200
Trichlorofluoromethane	5	20	50	100	200
Acrolein	50	200	500	1000	2000
Acetone	5	20	50	100	200
Trichlorotrifluoroethane	5	20	50	100	200
Iodomethane	5	20	50	100	200
Carbon disulfide	5	20	50	100	200
Methylene chloride	5	20	50	100	200
tert-Butyl alcohol	100	400	1,000	2,000	4,000
1,1-Dichloroethene	5	20	50	100	200

Table 2 - TestAmerica Primary Standard Calibration Levels, 5 mL purge Solid

Compound	Calibration Level ug/kg				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,1-Dichloroethane	5	20	50	100	200
trans-1,2-Dichloroethene	5	20	50	100	200
Acrylonitrile	10	40	100	200	400
Methyl *tert*-butyl ether (MTBE)	5	20	50	100	200
Hexane	5	20	50	100	200
cis-1,2-Dichloroethene	5	20	50	100	200
Tetrahydrofuran	5	20	50	100	200
Chloroform	5	20	50	100	200
1,2-Dichloroethane	5	20	50	100	200
Dibromomethane	5	20	50	100	200
2-Butanone	5	20	50	100	200
1,4-Dioxane	250	1000	2,500	5,000	10,000
1,1,1-Trichloroethane	5	20	50	100	200
Carbon tetrachloride	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
cis-1,3-Dichloropropene	5	20	50	100	200
Trichloroethene	5	20	50	100	200
Dibromochloromethane	5	20	50	100	200
1,2-Dibromoethane	5	20	50	100	200
1,2,3-Trichloropropane	5	20	50	100	200
Acetonitrile	50	200	500	1000	2000
1,1,2-Trichloroethane	5	20	50	100	200
Benzene	5	20	50	100	200
Ethylmethacrylate	5	20	50	100	200
trans-1,3-Dichloropropene	5	20	50	100	200
Bromoform	5	20	50	100	200
4-Methyl-2-pentanone	5	20	50	100	200
2-Hexanone	5	20	50	100	200
Tetrachloroethene	5	20	50	100	200
Toluene	5	20	50	100	200
1,1,2,2-Tetrachloroethane	5	20	50	100	200
2-Chloroethyl vinyl ether	10	40	100	200	400
Vinyl acetate	5	20	50	100	200
Chlorobenzene	5	20	50	100	200
Ethylbenzene	5	20	50	100	200
Styrene	5	20	50	100	200
t-1,4-Dichloro-2-butene	5	20	50	100	200
m and p Xylenes	10	40	100	200	400
o-xylene	5	20	50	100	200

Table 2 - TestAmerica Primary Standard Calibration Levels, 5 mL purge Solid

Compound	Calibration Level ug/kg				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,3-Dichlorobenzene	5	20	50	100	200
1,4-Dichlorobenzene	5	20	50	100	200
1,2-Dichlorobenzene	5	20	50	100	200
2,2-Dichloropropane	5	20	50	100	200
Bromochloromethane	5	20	50	100	200
1,1-Dichloropropene	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
1,3-Dichloropropane	5	20	50	100	200
Isopropylbenzene	5	20	50	100	200
Bromobenzene	5	20	50	100	200
n-Propylbenzene	5	20	50	100	200
2-Chlorotoluene	5	20	50	100	200
4-Chlorotoluene	5	20	50	100	200
1,3,5-Trimethylbenzene	5	20	50	100	200
tert-Butylbenzene	5	20	50	100	200
1,2,4-Trimethylbenzene	5	20	50	100	200
sec-butylbenzene	5	20	50	100	200
4-Isopropyltoluene	5	20	50	100	200
n-Butylbenzene	5	20	50	100	200
1,2,4-Trichlorobenzene	5	20	50	100	200
Napthalene	5	20	50	100	200
Hexachlorobutadiene	5	20	50	100	200
1,2,3-Trichlorobenzene	5	20	50	100	200

Table 2A - TestAmerica Primary Standard Calibration Levels, Low Level [1(Water)]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Dibromofluoromethane (Surrogate)	1	5	10	20	40
1,2-Dichloroethane-d4 (Surrogate)	1	5	10	20	40
Toluene-d8 (Surrogate)	1	5	10	20	40
Bromofluorobenzene (Surrogate)	1	5	10	20	40
Dichlorodifluoromethane	1	5	10	20	40
Chloromethane	1	5	10	20	40
Vinyl Chloride	1	5	10	20	40
Bromomethane	1	5	10	20	40
Chloroethane	1	5	10	20	40
Trichlorofluoromethane	1	5	10	20	40
Acrolein	10	50	100	200	400
Acetone	2	10	20	40	80
1,1-Dichloroethene	1	5	10	20	40
Trichlorotrifluoroethane	1	5	10	20	40
Iodomethane	1	5	10	20	40
Carbon Disulfide	1	5	10	20	40
Methylene Chloride	1	5	10	20	40
Acetonitrile	10	50	100	200	400
Acrylonitrile	2	10	20	40	80
Methyl tert-butyl ether	1	5	10	20	40
trans-1,2-Dichloroethene	1	5	10	20	40
Hexane	1	5	10	20	40
Vinyl acetate	1	5	10	20	40
1,1-Dichloroethane	1	5	10	20	40
tert-Butyl Alcohol	20	100	200	400	800
2-Butanone	2	10	20	40	80
cis-1,2-dichloroethene	1	5	10	20	40
2,2-Dichloropropane	1	5	10	20	40
Bromochloromethane	1	5	10	20	40
Chloroform	1	5	10	20	40
Tetrahydrofuran	1	5	10	20	40
1,1,1-Trichloroethane	1	5	10	20	40
1,1-Dichloropropene	1	5	10	20	40
Carbon Tetrachloride	1	5	10	20	40
1,2-Dichloroethane	1	5	10	20	40
Benzene	1	5	10	20	40
Trichloroethene	1	5	10	20	40
1,2-Dichloropropane	1	5	10	20	40

Table 2A - TestAmerica Primary Standard Calibration Levels, Low Level [1(Water)]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,4-Dioxane	50	250	500	1000	2000
Dibromomethane	1	5	10	20	40
Bromodichloromethane	1	5	10	20	40
2-Chloroethyl vinyl ether	2	10	20	40	80
cis-1,3-Dichloropropene	1	5	10	20	40
4-Methyl-2-pentanone	2	10	20	40	80
Toluene	1	5	10	20	40
trans-1,3-Dichloropropene	1	5	10	20	40
Ethyl Methacrylate	1	5	10	20	40
1,1,2-Trichloroethane	1	5	10	20	40
1,3-Dichloropropane	1	5	10	20	40
Tetrachloroethene	1	5	10	20	40
2-Hexanone	2	10	20	40	80
Dibromochloromethane	1	5	10	20	40
1,2-Dibromoethane	1	5	10	20	40
Chlorobenzene	1	5	10	20	40
1,1,1,2-Tetrachloroethane	1	5	10	20	40
Ethylbenzene	1	5	10	20	40
m + p-Xylene	2	10	20	40	80
Xylene-o	1	5	10	20	40
Styrene	1	5	10	20	40
Bromoform	1	5	10	20	40
Isopropylbenzene	1	5	10	20	40
1,1,2,2-Tetrachloroethane	1	5	10	20	40
1,4-Dichloro-2-butene	1	5	10	20	40
1,2,3-Trichloropropane	1	5	10	20	40
Bromobenzene	1	5	10	20	40
n-Propylbenzene	1	5	10	20	40
2-Chlorotoluene	1	5	10	20	40
1,3,5-Trimethylbenzene	1	5	10	20	40
4-Chlorotoluene	1	5	10	20	40
tert-Butylbenzene	1	5	10	20	40
1,2,4-Trimethylbenzene	1	5	10	20	40
sec-Butylbenzene	1	5	10	20	40
4-Isopropyltoluene	1	5	10	20	40
1,3-Dichlorobenzene	1	5	10	20	40
1,4-Dichlorobenzene	1	5	10	20	40
n-Butylbenzene	1	5	10	20	40
1,2-Dichlorobenzene	1	5	10	20	40
1,2-Dibromo-3-chloropropane	1	5	10	20	40

Table 2A - TestAmerica Primary Standard Calibration Levels, Low Level [1(Water)]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2,4-Trichlorobenzene	1	5	10	20	40
Hexachlorobutadiene	1	5	10	20	40
Naphthalene	1	5	10	20	40
1,2,3-Trichlorobenzene	1	5	10	20	40
Cyclohexane	1	5	10	20	40
Methyl Acetate	2	10	20	40	80
Methylcyclohexane	1	5	10	20	40
1,3,5-Trichlorobenzene	1	5	10	20	40

[1] 25 mL purge samples analyzed at 5 mL purge on more sensitive equipment.

Table 3 - TestAmerica Appendix IX Standard and Reporting Limits, 5 mL purge

Compound	CAS Number	Reporting Limits			
		5 mL Water µg/L	Low Level 5mL purge water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	10	2	10	500
Dichlorofluoromethane	75-43-4	10	2	10	500
Isopropyl ether	108-20-3	10	2	10	500
Chloroprene	126-99-8	5	2	5	250
n-Butanol	71-36-3	200	50	200	10,000
Propionitrile	107-12-0	20	4	20	1000
Methacrylonitrile	126-98-7	5	2	5	250
Isobutanol	78-83-1	200	50	200	10,000
Methyl methacrylate	80-62-6	5	2	5	250
1,1,1,2-Tetrachloroethane	630-20-6	5	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	10	2	10	500
Ethyl ether	60-29-7	10	2	10	500
Ethyl Acetate	141-78-6	20	4	20	1,000
2-Nitropropane	79-46-9	10	4	10	500
Cyclohexanone	108-94-1	50	20	50	2500
Isopropylbenzene	98-82-8	5	1	5	250
2-Methylnaphthalene (Michigan only)	91-57-6	NA	5	10	330

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Table 4

Recommended TestAmerica Appendix IX Standard Calibration Levels, µg/L

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Allyl Chloride	5	20	50	100	200
Dichlorofluoromethane	5	20	50	100	200
Isopropyl ether	5	20	50	100	200
Chloroprene	5	20	50	100	200
n-Butanol	100	400	1,000	2,000	4,000
Propionitrile	10	40	100	200	400
Methacrylonitrile	5	20	50	100	200
Isobutanol	100	400	1,000	2,000	4,000
Methyl methacrylate	5	20	50	100	200
1,1,1,2-Tetrachloroethane	5	20	50	100	200
1,2-Dibromo-3-chloropropane	10	40	100	200	400
Ethyl ether	5	20	50	100	200
Ethyl Acetate	10	40	100	200	400
2-Nitropropane	10	40	100	200	400
Cyclohexanone	50	200	500	1,000	2,000
2-Methylnaphthalene (Michigan only)	2	10	20	40	80
Ethyl tert-butyl ether	5	20	50	100	200
tert-Amyl methyl ether	5	20	50	100	200
1,2,3-Trimethylbenzene	5	20	50	100	200

Table 5 - Internal Standards

Compound	Standard Concentration µg/mL (may vary per matrix)	Quantitation ion
Fluorobenzene	50 – 250	96
Chlorobenzene-d5	50 – 250	117
1,4-Dichlorobenzene-d4	50 – 250	152

Notes:

1) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

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Table 6 - Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL (may vary per matrix)
1,2-Dichloroethane-d_4	50 – 250
Dibromofluoromethane (not required for Method 8260C)	50 – 250
Toluene-d_8	50 - 250
4-Bromofluorobenzene	50 – 250

Notes:

1) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

2) Recovery limits for surrogates are g enerated from historical data and are maintained by the QA Dept.

3) There is no corrective action for Dibromofluoromethane for Method 8260C.

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Table 7 - Matrix Spike / LCS Compounds

Compound	Standard Concentration µg /mL
1,1,1-Trichloroethane	50 – 250
1,1,2,2-Tetrachloroethane	50
1,1,2-Trichloro-1,2,2-trifluoroethane	50
1,1,2-Trichloroethane	50
1,1-Dichloroethane	50
1,1-Dichloroethene	50
1,1-Dichloropropene	50
1,2,3-Trichlorobenzene	50
1,2,3-Trichloropropane	50
1,2,4-Trichlorobenzene	50
1,2,4-Trimethylbenzene	50
1,2-Dibromo-3-chloropropane	50
1,2-Dibromoethane	50
1,2-Dichlorobenzene	50
1,2-Dichloroethane	50
1,2-Dichloroethene (total)	100
1,2-Dichloropropane	50
1,3,5-Trimethylbenzene	50
1,3-Dichlorobenzene	50
1,3-Dichloropropane	50
1,4-Dichlorobenzene	50
2,2-Dichloropropane	50
2-Butanone	50
2-Chloroethyl Vinyl Ether	100 – 500
2-Chlorotoluene	50
2-Hexanone	50
4-Chlorotoluene	50
4-Methyl-2-pentanone	50
Acetone	50
Acetonitrile	500 – 2500
Acrolein	500
Acrylonitrile	100 – 500
Benzene	50
Bromobenzene	50
Bromochloromethane	50
Bromodichloromethane	50
Bromoform	50
Bromomethane	50
Carbon disulfide	50
Carbon tetrachloride	50
Chlorobenzene	50
Chloroethane	50

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Table 7 - Matrix Spike / LCS Compounds

Compound	Standard Concentration µg /mL
Chloroform	50
Chloromethane	50
cis-1,2-Dichloroethene	50
cis-1,3-Dichloropropene	50
Cyclohexane	50
Dibromochloromethane	50
Dibromomethane	50
Dichlorodifluoromethane	50
Ethylbenzene	50
Hexachlorobutadiene	50
Iodomethane	50
Isopropylbenzene	50
Isopropylether	50
Methyl acetate	50
Methyl tert-butyl ether (MTBE)	50
Methylcyclohexane	50
Methylene chloride	50
Naphthalene	50
n-Butylbenzene	50
n-Hexane (Ohio VAP only)	50
n-Propylbenzene	50
p-Isopropyltoluene	50
sec-Butylbenzene	50
Styrene	50
tert-Butylbenzene	50
Tetrachloroethene	50
Toluene	50
trans-1,2-Dichloroethene	50
trans-1,2-Dichloroethene	50
trans-1,3-Dichloropropene	50
Trichloroethene	50
Trichlorofluoromethane	50
Vinyl Acetate	50
Vinyl chloride	50
Xylenes (total)	150 – 750

Notes: 1) 5 µL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Recovery and precision limits for LCS and MS/MSD are generated from historical data and are maintained by QA Dept.

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Table 8 - BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 9 - SPCC Compounds and Minimum Response Factors for Method 8260B

Compound	Methods 8260B and 8260A Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 10 - Method 8260C. Recommended Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification		
Volatile Compound	**Minimum Response Factor**	**Typical Response Factor**
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408

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Table 10 - Method 8260C: Recommended Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification (cont'd)

Volatile Compound	Minimum Response Factor	Typical Response Factor
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

Table 11 - CCC Compounds for Method 8260B

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	30	20
1,1-Dichloroethene	30	20
Chloroform	30	20
1,2-Dichloropropane	30	20
Toluene	30	20
Ethylbenzene	30	20
n-Hexane (Ohio VAP only)	30	20

Table 12 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Acetone	43	58	
Methylene chloride	84	49	51, 86

Table 12 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133

Table 12 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
Allyl Chloride	76	41	78
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	
Cyclohexane	56	69	84
Methyl Acetate	43	74	
Methyl cyclohexane	83	55	98

* The primary ion must be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

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Title: INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSES

[Methods: SW846 Methods 6010B, 6010C, and EPA Method 200.7]

Approvals (Signature/Date):

Technology Specialist	02/14/11 Date	Health & Safety Coordinator	02/15/11 Date
Quality Assurance Manager	02/16/11 Date	Laboratory Director	02/09/11 Date
Technical Director	02/04/11 Date		

This SOP was previously identified as SOP NC-MT-012, Rev 1, dated 01/07/09

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1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Methods 6010B, 6010C, and EPA Method 200.7. Tables I and IA of Appendix A lists the elements appropriate for analysis by Methods, 6010B, 6010C, and 200.7 plus the associated reporting limit. Additional elements may be analyzed under Methods, 6010B, 6010C, and 200.7 provided that the method performance criteria presented in Section 13.0 are met.

1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity, and optimum concentration ranges of the metals will vary with the matrices and instrumentation used.

1.3. Methods 6010B and 6010C are applicable to the determination of dissolved, suspended, total recoverable, and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, biological, and TCLP, EP, and other leachates/extracts. All matrices require digestion prior to analysis. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.

1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis. Silver concentrations must be below 0.1 mg/L in aqueous samples.

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of multi elements in solution using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by radio frequency inductively-coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences must also be recognized, and appropriate actions taken. Alternatively, multivariate calibration methods may be chosen for

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which point selection for background correction is superfluous since whole spectral regions are processed.

2.1. Refer to NC-IP-010, Acid Digestion of Soils by SW846 Method 3050B, and NC-IP-011, Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods, for details on sample preparation methods.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version for additional definitions. Refer to Appendix B for a cross reference of method definitions.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization, and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects) at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid

concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard, and/or use of a high solids nebulizer can reduce the effect. Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4-ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3.1. The plasma emits strong UV light and is harmful to vision. **NOTE**: **AVOID looking directly at the plasma**.

5.3.2. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers must not go near the instrument while in operation.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Metals digestates can be processed outside of a fume hood. Solvent and waste containers must be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator

6.3. Argon gas supply, welding grade or equivalent

6.4. Coolflow or appropriate water cooling device

6.5. Peristaltic Pump

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes – ranging from 5 µL → 10 ml

6.7. Class A volumetric flasks – range from 50 ml → 2000 ml

6.8. Autosampler tubes

7. REAGENTS AND STANDARDS

7.1 Intermediate standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Expiration dates can be extended provided that the

acceptance criteria described in laboratory-specific SOPs are met. Additional information can be found in SOP NC-QA-017. Standard or spiking concentrations, as well as vendors, are subject to change.

7.2 Working calibration, calibration verification solutions, and internal standard solutions must be prepared in a matrix of 5% hydrochloric and 5% nitric acids. Refer to Tables II, III, IV and V (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction, and spiking solutions. Refer to the laboratory Standard Logbook or Reagent Logbook for details on standard or reagent preparation.

7.3 Concentrated nitric acid (HN_{O3}), trace metal grade or better.

7.4 Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.5 Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Soil samples do not require preservation, but must be stored at 4°C ± 2° until the time of preparation.

8.4. Metals samples that are preserved at the laboratory must be held for 24 hours before digestion.

Note: If the samples are preserved the same day of collection, the 24-hour waiting period is not required

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. Prior to analysis of any analyte using Methods 200.7, 6010B, or 6010C, the following requirements must be met.

9.1.2. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used for each instrument. The IDL must be determined annually for client-specific projects. For DoD work, refer to SOP NC-QA-016. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined. The IDL will be determined by multiplying by 3,

the standard deviation obtained from the analysis of a blank solution, with seven consecutive measurements. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples).

9.1.3. Instrument Detection Limits (IDLs), Method 6010C – IDLs are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit. It may be helpful to compare the calculated IDLs to the established lower limit of quantitation; however, it should be understood that the lower limit of quantitation needs to be verified using the criteria in Section 10.7.

9.1.4. Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client samples. Refer to TestAmerica North Canton SOP NC-QA-021 and CA-Q-S-006 for details on MDL analysis and criteria.

9.1.5. Linear Range Verification (LR) - The linear range must be verified every six months for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample. The standards used to verify the linear range limit must be analyzed during a routine analytical run, and must read within 10% of the expected value.

For the **initial** determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from a minimum of three to five different concentration standards across the estimated range. One standard must be near the upper limit of the estimated range. The concentration measured at the LDR must be no more than 10% less than the expected level extrapolated from lower standards. If the instrument is adjusted in any way that may affect the LRs, new dynamic ranges must be determined. The LR data must be documented and kept on file.

9.1.6. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference, or a computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the ICP instrument manual for specific procedures to be used in setting background correction points.

9.1.7. Inter-element Corrections (IECs) - ICP inter-element correction factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be

redetermined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC, then the possibility of contamination must be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., ICP-MS). Published wavelength tables (e.g., MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than ± the RL as defined in Tables I or IA. For elements with a reporting limit of 10 ug/L or less, the signal must be ± two times the RL. To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the actual concentration of the "interfering element."

Note: Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response. Additional spectral interference is present from easily ionizable elements such as potassium and sodium in axial viewing instruments.

9.1.8. Rinse Time Determination - Rinse times must be determined upon initial set-up of an ICP instrument. To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see Section 9.1.4) must be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes, it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be re-analyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch of up to 20 samples. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at, or above, 10 % of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of ten times higher than the

blank contamination level). For Ohio VAP projects, all analytes must be less than the reporting limit with the following exceptions: (a) insufficient sample for re-digestion, (b) expired holding times, or (c) the elements detected in the method blank are non-detect for the associated samples.

- If the analyte is a common laboratory contaminant (copper, iron, lead, or zinc), the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. Such action must be addressed in the project narrative.

- Repreparation and re-analysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action must be addressed in the project narrative.

- If the above criteria are not met and re-analysis is not possible, then the sample data must be qualified. This anomaly must be addressed in the project narrative.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table II (Appendix A). The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits, the system is out of control and corrective action must occur. Unless in-house control limits are established, a control limit of 80 - 120% recovery must be applied for Method 6010B and 6010C. For Method 200.7, control limits of 85-115% must be applied.

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The LCSD recovery is evaluated using the same control limits as the LCS. The RPD of the LCS and LCSD must be compared to in-house limits.

- In the instance where the LCS recovery is greater than the upper control limit and the sample results are < RL, the data may be reported with qualifiers. Such action must be addressed in the report narrative.

- Corrective action will be repreparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable. For Ohio VAP projects, the LCS/LCSD must meet acceptance criteria. The laboratory may re-analyze an aliquot of the LCS/LCSD to verify the outlier; however, if the LCS/LCSD exhibits the same anomaly upon re-analysis, the sample batch must be redigested or re-extracted and re-analyzed. The exceptions are as follows: (a) insufficient sample for re-extraction/re-digestion, (b)

expired holding times, or (c) the LCS is biased high and the samples are non-detect for those analytes.

9.4 Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of, or in addition to, MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables II and V (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. For Methods 6010B and 6010C, control limits of 75-125% (70-130% for Method 200.7) recovery and 20% RPD or historical acceptance criteria must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and re-analysis of the sample and MS/MSD. MS/MSD results, which fall outside the control limits, must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds four times the spike level for that analyte, the recovery data is reported as NC, MSB (i.e., not calculated). Two other narrative notes for metals analyses: Matrix spike/spike duplicate spike recovery/recoveries was/were outside the acceptance limits of some analytes. The acceptable LCS analysis data indicated that the analytical system was operating within control and this condition is most likely due to matrix interference. See the Matrix Spike Report for the affected analytes which will be flagged with N. Matrix spike/spike duplicate relative percent difference (RPD) exceeded the acceptance limits for some analytes. The imprecision may be attributed to sample heterogeneity. See the Matrix Spike Report for the affected analytes, which will be flagged with *.

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For Method 6010C samples – If the MS/MSD recoveries are unacceptable, the same sample from which the MS/MSD aliquots were prepared should also be spiked with a post digestion spike. Otherwise, another sample from the same preparation batch should be used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and must be recovered to within 80% to 120% of the known value. If this spike fails, then the dilution test must be run on this sample. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed.

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9.6 Dilution test – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5X dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample after correction for dilution must agree within 10% of the original sample determination when the original sample concentration is greater than 50 times the MDL. If the results are not within 10%, the possibility of chemical or physical interference exists and the data is flagged.

9.7 Control Limits

9.7.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.7.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMS (QC Browser program).

9.8 Method Detection Limits (MDLs) and MDL Checks

9.8.1 MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.8.2 MDLs are easily accessible via LIMS.

9.9 Nonconformance and Corrective Action

9.9.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager. Deviations are not allowed for Ohio VAP projects.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the instructions in Appendix F.

10.3 Initial Calibration - Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures. Flush the system with the calibration blank between each standard or as the manufacturer recommends. The calibration curve must consist of a minimum of a blank and a standard. Refer to Appendix F for detailed setup and operation protocols. Refer to Instruction Manuals in laboratory. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument standardization date and time must be included in the raw data

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10.4 Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after the initial calibration. For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution with relative standard deviation <3% from replicate (minimum of two) exposures. For Methods 6010B and 6010C, the ICV must fall within ±10% of the true value for that solution. For Method 6010B, the relative standard deviation must be <5% from replicate (minimum of two) exposures. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The calibration blank is prepared with reagent water to the same concentrations of the acids found in the standards. The calibration blank will also be used for all initial (ICB) and continuing calibration blank (CCB) determinations. The ICB result must fall within ± the RL from zero. If either the ICV or ICB fail to meet criteria, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified (see Sections 11.6 through 11.8 for required run sequence).

10.5 Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the sample run. The CCV is to be a mid-range standard made from a dilution of the calibration standard. The CCV for all methods must fall within 10% of the true value for that solution. For Methods 6010B and 200.7, the relative standard deviation must be <5% from replicate (minimum of two) exposures. For Method 6010C, there is no criteria for RSD from replicate exposures. A CCB is analyzed immediately following each CCV (see Sections 11.6 through 11.8 for required run sequence). The calibration blank is prepared with reagent water to the same concentrations of the acids found in the standards. The calibration blank will also be used for all initial (ICB) and continuing calibration blank (CCB) determinations. The CCB result must fall within ± RL from zero. If the blank is less than 1/10 the concentration of the action level of interest and no sample is within 10% of the action limit, |re-analysis and recalibration are not required before continuation of the run. This is not acceptable for Ohio VAP samples. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV or CCB fails, all affected samples must be re-analyzed with valid CCV/CCB pairs (refer to Section 11.7 for an illustration of the appropriate rerun sequence). Exceptions: If CCB > RL, samples < RL can be reported with an NCM. If CCV is outside of criteria on the high side, samples < RL can be reported with an NCM.

10.6 Interference Check Analysis (ICSA/ICSAB) - The validity of the inter-element correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table IV (Appendix A) for the details of ICSA and ICSAB composition. Custom multi-element ICS solutions must be used. All analytes must be spiked into the ICSAB solution; therefore, if a non-routine analyte is required, then it must be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. If the ICP will display overcorrection as a negative number, then the non-routine elements can be controlled from the ICSA. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium, and magnesium must always be included in all ICP runs.

10.6.1 The ICSA and ICSAB solutions must be run at the beginning of the run (see Sections 11.6 or 11.7 for required run sequence).

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10.6.2 The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the samples rerun.

10.6.3 ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within ± 2 times the RL from zero. ICSA results for the non-interfering elements with RLs > 10 µg/L must fall within ± 1 times the RL from zero. If the ICSA results for the non-interfering elements do not fall within ± two times RL (RL ≤10) or ± 1xRL (RL>10) from zero, the field sample data must be evaluated as follows.

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.
- If the affected element was not required, then the sample data can be accepted.
- If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than ± two times the RL from zero, then the field sample data can be accepted.
- If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± two times the RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than ten times the analyte signal in the ICSA.
- If the data does not meet the above conditions, then the IECs must be re-evaluated and corrected if necessary and the affected samples re-analyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually, the calculations must be clearly documented on the raw data.

10.7 CRI (CRA or LLICV/LLCCV (6010C) - To verify linearity near the RL for ICP analysis, a CRI standard is run at the beginning of each sample analysis run. Additionally, some projects may require CRI analysis at the end of the run (see Sections 11.6 or 11.7 for required run sequence). Evaluate associated samples based upon advisory limits of ± 50% of true value. For Method 6010C, it must be analyzed at the beginning and end of the analytical run. The control limit for this method is 70-130%.

Note: The custom CRI mix contains most analytes at a level near the standard lab reporting limit.

11. PROCEDURE

11.1. A minimum of <u>two</u> <u>exposures</u> for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.2. Prior to calibration and between each sample/standard, the system is rinsed with the calibration blank solution.

11.3. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV,CCV), blanks (ICB,CCB,PB), interference checks (ICSA,ICSAB), and field samples (linear range) to improve the data review process.

11.4. To facilitate the early identification of QC failures and samples requiring rerun, it is strongly recommended that sample data be reviewed periodically throughout the run.

11.5. The following procedural guidelines must be followed when using an internal standard:

11.5.1 Typically used internal standard is: yttrium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.5.2 The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards must be added to blanks, samples, and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.5.3 The concentration of the internal standard must be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.5.4 The internal standard raw intensity counts must be printed on the raw data.

11.5.5 The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte). The instrument automatically adjusts sample results based on comparison of the internal standard intensity in the sample to the internal standard intensity at calibration.

11.5.5.1 If the internal standard counts fall within ±30% of the counts observed in the ICB, then the data is acceptable.

11.5.5.2 If the internal standard counts in the field samples are more than ± 30% higher than the expected level, evaluate the run to determine if a dilution is needed due to matrix interference.

11.6 The following analytical sequence must be used for Methods 6010B, 6010C, and 200.7:

Instrument Calibration
ICV
ICB

CRI/LLICV (6010C)
ICSA
ICSAB
CCV
CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CRI (The CRI counts as a sample analysis.) /LLICV (6010C)
CCV
CCB

Refer to Quality Control Section 9.0 for Methods 6010B, 6010C, and 200.7 quality control criteria.

11.7 The following run sequence provides an illustration of a mid-run CCV or CCB failure and the appropriate corrective action run sequence as described in Section 10.5.

Original Run: Instrument Calibration
ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
10 samples **
CCV * * Failure occurs at CCV/CCB
CCB * **Samples requiring rerun for affected analytes
10 samples **
CCV
CCB
10 samples
CCV
CCB

11.8 The instrument may be reprofiled between CCV/CCB pairs to correct for environment-induced drift.

11.9 Guidelines are provided in the Appendix C, D, and E on procedures to minimize contamination of samples and standards, preventive maintenance, and troubleshooting.

11.10 All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range. If an inter-element correction exists for an analyte, which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is an interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

11.11 Any variation in procedure must be completely documented using instrument run logs, maintenance logs, report narratives, a Nonconformance Memo, or an anomaly report; and is approved by a Supervisor/Group Leader and QA Manager.

11.12 Nonconformance documentation must be filed in the project file.

11.14 Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.15 Analytical Documentation

11.15.1 Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.15.2 All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. The supervisor or designee reviews logbooks.

11.15.3 Documentation, such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs, is available for each data file.

11.15.4 Sample results and associated QC are entered into LIMs after final technical review.

12 DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%\ R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg\ /\ L = \frac{C \times V1 \times D}{V2}$$

Where:
C = Concentration (mg/L) from instrument readout (mean of two exposures)
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples is calculated as follows:

$$mg / Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout (mean of two exposures)
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading × 5)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Trivalent Chromium

12.10.1 Trivalent chromium (CR^{+3}) is the result obtained by subtracting the hexavalent chromium (CR^{+6}) results for a sample from the total chromium result from that sample. The total chromium result is determined using the procedures in this SOP. The hexavalent chromium result is determined using the procedures in TestAmerica North Canton SOP NC-WC-024.

12.10.2 Reporting Limits

12.10.1 The TestAmerica North Canton water reporting limit for trivalent chromium is 0.02 mg/l.

12.10.2 The TestAmerica North Canton solid reporting limit for trivalent chromium is 2.0 mg/kg, wet weight.

12.10.3 Calculations: Cr^{+3} = Cr, total – Cr^{+6}

12.10.3 Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Tables I and IA in Appendix A for the list of analytes that may be analyzed using this SOP.

13.3. Training Qualification

13.3.1 The Group/Team Leader or the Supervisor has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the corporate environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by this Method

15.2.1. The following waste streams are produced when this method is carried out:

15.2.2. Acid waste consisting of sample and rinse solution. Any sample waste generated must be collected and disposed of in the acid waste drum located in the Metals Lab.

15.2.3. Standards must be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

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16. REFERENCES

16.1. References

16.1.1. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits

16.1.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B

16.1.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Final Update IV, Method 6010C, Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 3, February 2007

16.1.4. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7

16.1.5. Inductively Coupled Plasma – Atomic Emission Spectrometric Method for Trace Element Analysis of water and wastes Method 200.7, 40 CFR – Chapter I – Part 136 – Appendix C. Electronic version dated September 30, 2002

16.1.6. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.7. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.8. Corporate Quality Management Plan (CQMP), current version

16.1.9. Revision History

Historical File:		Revision 2.0: 10/27/97		Revision 0: 01/08/04 (NC-MT-012)
(formerly CORP-MT-0001NC)		Revision 2.1: 04/19/99		Revision 1: 01/07/09
		Revision 3.1: 10/04/00		
		Revision 3.2: 01/19/01		
		Revision 3.3: 12/05/01		
		Revision 3.4: 01/08/04		

16.2. Associated SOPs and Policies, current version

16.2.1. TestAmerica North Canton QC Program, QA-003

16.2.2. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.3. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.4. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.5. Hexavalent Chromium (Colorimetric), NC-WC-024

16.2.6. Acid Digestion of Soils, SW846 Method 3050B, NC-IP-010

16.2.7. Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods, NC-IP-011

16.2.8. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications/interpretations from Methods 6010B and 200.7

17.1.1.1. TestAmerica North Canton Laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in-house as recommended by the subject methods.

17.1.1.2. Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution must fall within a specific concentration range around the calibration blank. In determining IECs because of lack of definition clarification for "concentration range around the calibration blank," TestAmerica North Canton has adopted the procedure in EPA CLP ILMO4.0.

17.1.1.3. Whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical interference exists. Because TestAmerica North Canton laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, TestAmerica North Canton may select to perform a dilution test on one sample in each prep batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At TestAmerica North Canton, matrix interference is determined by evaluating data for the LCS and MS/MSD. TestAmerica North Canton REQUIRES documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

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17.1.2. Modifications from Method 200.7

17.1.2.1. Method 200.7 defines the IDL as the concentration equivalent to a signal due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the same wavelength. TestAmerica North Canton labs utilize the IDL definition as defined in Section 9.1 of this SOP.

17.1.2.2. The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

17.1.2.3. Method Section 9.3.4 specifies that "Analysis of the ICV (ICSA/AB) solution immediately following calibration must verify that the instrument is within ± 5% of calibration with a relative standard deviation <3% from replicate integrations ≥ 4". TestAmerica North Canton uses a minimum of two exposures.

17.1.2.4. The 40 CFR version of Method 200.7 requires the instrument check standard to agree within ± 5% of expected values and less than, or equal to, 3% RSD. Also, the 40 CFR version requires the interference check sample to be analyzed at the beginning, end, and at periodic intervals throughout the sample run. At TestAmerica North Canton, the instrument check standard equals the CCV, which must agree within ± 10% of expected values and 5% RSD, and the ICSA standards are analyzed only at the beginning of a sample run. TestAmerica's procedures are in line with the Rev. 4.4, May 1994 version of Method 200.7.

17.1.2.5. Section 7.12 of Method 200.7 indicates that the QCS (ICV) must be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

17.1.2.6. The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 Section 10.4 states that results must fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The control limits listed in this SOP are those applicable to the EPA designed solution.

17.1.2.7. Method 200.7 Section 9.3.4 states the CCB must be less than the IDL, but more than the lower 3-sigma control limit of the calibration blank. The

intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica North Canton has adopted an absolute control limit of ± RL from zero for calibration blank criteria. SOP Section 10.5 provides the detailed corrective action criteria that must be followed.

17.1.3. Modifications from Method 6010B

17.1.3.1. Chapter 1 of SW-846 states that the method blank must not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client. This is not acceptable for Ohio VAP projects.

17.1.3.2. Method 6010B Section 8.6.1.3 states that the results of the calibration blank are to agree within three times the IDL. If not, repeat the analysis two or more times, and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and re-analyze the previous ten samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica North Canton has adopted an absolute control limit of ± RL from zero for calibration blank criteria. See SOP Section 10.5 for a detailed description of the required corrective action procedures.

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APPENDIX A - TABLES

TABLE I: Methods 200.7, 6010B, and 6010C Target Analyte List

Element	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	200	20
Antimony	Sb	7440-36-0	60	6
Arsenic	As	7440-38-2	300	30
Barium	Ba	7440-39-3	200	20
Beryllium	Be	7440-41-7	5.0	0.5
Boron	B	7440-42-8	200	20
Cadmium	Cd	7440-43-9	5.0	0.5
Calcium	Ca	7440-70-2	5000	500
Chromium	Cr	7440-47-3	10	1
Cobalt	Co	7440-48-4	50	5
Copper	Cu	7440-50-8	25	2.5
Iron	Fe	7439-89-6	100	10
Lead	Pb	7439-92-1	100	10
Magnesium	Mg	7439-95-4	5000	500
Manganese	Mn	7439-96-5	15	1.5
Molybdenum	Mo	7439-98-7	40 (100 for 200.7)	4
Nickel	Ni	7440-02-0	40	4
Potassium	K	7440-09-7	5000	500
Selenium	Se	7782-49-2	250	25
Silver	Ag	7440-22-4	10	1
Sodium	Na	7440-23-5	5000	500
Thallium	Tl	7440-28-0	2000	200
Vanadium	V	7440-62-2	50	5
Zinc	Zn	7440-66-6	20 (50 for 200.7)	2
Tin	Sn	7440-31-5	100	10
Titanium	Ti	7440-32-6	50	5

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TABLE IA: Methods 200.7, 6010B, and 6010C Trace ICP Target Analyte List

Element	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Arsenic	As	7440-38-2	10	1.0
Lead	Pb	7439-92-1	3.0	0.3
Selenium	Se	7782-49-2	5.0	0.5
Thallium	Tl	7440-28-0	10	1.0
Antimony	Sb	7440-36-0	10	1.0
Cadmium	Cd	7440-43-9	2.0	0.2
Silver	Ag	7440-22-4	5.0	0.5
Chromium	Cr	7440-47-3	5.0	0.5
Cobalt	Co	7440-48-4	7.0	5.0
Titanium	Ti	7440-32-6	50	5.0
Vanadium	V	7440-62-2	7.0	5.0
Molybdenum	Mo	7439-98-7	10	1.0

TABLE II: Matrix Spike and Aqueous Laboratory Control Sample Levels

Element	Concentration (ug/L)
Aluminum	2000
Antimony	500
Arsenic	2000
Barium	2000
Beryllium	50
Cadmium	50
Calcium	50000
Chromium	200
Cobalt	500
Copper	250
Iron	1000
Lead	500
Magnesium	50000
Manganese	500
Molybdenum	1000
Nickel	500
Potassium	50000
Selenium	2000
Silver	50
Sodium	50000
Thallium	2000
Vanadium	500
Zinc	500
Boron	1000
Tin	2000
Titanium	1000

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TABLE III: Trace ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	12500	25000
Antimony	1000	10	250	500
Arsenic	1000	10	250	500
Barium	4000	10	1000	2000
Beryllium	4000	5	1000	2000
Cadmium	1000	2	250	500
Calcium	100000	5000	25000	50000
Chromium	4000	5	1000	2000
Cobalt	4000	50	1000	2000
Copper	4000	25	1000	2000
Iron	50000	100	12500	25000
Lead	1000	3	250	500
Magnesium	100000	5000	25000	50000
Manganese	4000	15	1000	2000
Molybdenum	4000	40	1000	2000
Nickel	4000	40	1000	2000
Potassium	100000	5000	25000	50000
Selenium	1000	5	250	500
Silver	2000	5	500	1000
Sodium	100000	5000	25000	50000
Thallium	2000	10	500	1000
Vanadium	4000	50	1000	2000
Zinc	4000	20	1000	2000
Boron	10000	200	1000	5000
Tin	10000	100	1000	5000
Titanium	10000	50	1000	5000

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TABLE IV: Interference Check Sample Concentrations

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	1000
Vanadium	-	500
Zinc	-	1000
Tin	-	1000
Boron		1000
Titanium		1000

TABLE V: TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

Element	Reporting Level (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

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APPENDIX B - CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW 6010B, 6010C, AND TESTAMERICA NORTH CANTON SOP

EPA 200.7	SW 6010B / 6010C	TestAmerica North Canton SOP
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	N/A	N/A
Laboratory fortified blank (LFB)	N/A	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

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APPENDIX C - TROUBLESHOOTING GUIDE

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber
Instrument Drift	Replace torch (Crack) Clean or replace nebulizer (blockage) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Reprofile
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage (back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

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APPENDIX D - CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the Metals Lab. All work areas must be kept scrupulously clean.

Powdered Gloves must not be used in the Metals Lab since the powder contains silica and zinc as well as other metallic analytes. Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipette tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

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APPENDIX E - PREVENTATIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs, indicate the date, time and instrument number. Then identify the problem and corrective action in the Maintenance Log.

The following procedures are required to ensure that that the instrument is fully operational:

Change sample pump tubing and pump windings

As Needed:
Check rinse solution and fill if needed
Check waste containers and empty if needed
Check sample capillary tubing is clean and in good condition
Check droplet size to verify nebulizer is not clogged.
Check sample flow for cross flow nebulizer
Check pressure for vacuum systems
Clean plasma torch assembly to remove accumulated deposits
Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance
Replace peristaltic pump tubing, sample capillary tubing and autosampler sipper probe
Apply silicon spray on autosampler tracks
Check water level in cool flow
Change oil for vacuum systems
Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

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APPENDIX F - ICP Operating Instructions
ICP Analysis (TJA 61E) Example

1. SETUP

a. Plasma Control Panel (enter)
b. (F1) - Startup
c. (F9) - Continue
d. (F2) - Levels
    1. Change auxiliary gas to low – use space bar to toggle
    2. Change nebulizer gas flow to 0.5 L/min.
    3. Change pump rate to 130
    4. Escape
    5. Allow instrument to warm up approximately 30 minutes.

2. DEVELOPMENT

a. Methods (enter)
b. Enter method name
c. (F3)-Method Info.
d. Change file name
e. (F9) - Done
f. (F9) - Done/Keep

3. OPERATION

a. Analysis (enter)
b. (F5)-Profile
    1. (F3) - Automatic
    2. (F1) - Run
    3. If peak position is greater than ± 0.05 units from the center peak position, you must adjust the profile. If it is within ± 0.05 units, press (F9) - Done.
    4. To adjust select (F1) - CalcSS and enter current vernier position. (enter)
    5. Adjust to new vernier position (F9) - Done
    6. Rerun profile until peak position is ± 0.05 units.
    7. (F9) - Done
c. Autosampler (F9)
    1. Enter method name (enter)
    2. Enter autosampler table name (enter)
    3. (F1) - Run

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Title: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS AND SOLID SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY

[Method: MCAWW Method 245.1, SW846 Method 7470A, SW846 7471A, and 7471B]

Approvals (Signature/Date):

Technology Specialist	09/22/10 Date	Health & Safety Coordinator	09/09/10 Date
Quality Assurance Manager	09/22/10 Date	Laboratory Director	09/12/10 Date
Technical Director	09/09/10 Date		

This SOP is a new SOP combining SOP NC-MT-011, Rev 1, dated 01/07/09 and SOP NC-MT-013 Rev 0, dated 01/07/09

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW846 Method 7470A, MCAWW Method 245.1 Method 7471B, and Method 7471A.

1.2. The associated LIMs method codes are BL (Method 245.1), J& (Method 7471B), O8 (Method 7470A), and O9 (Method 7471A).

1.3. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants and potassium permanganate has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity, and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation, and volume of sample used.

1.4. Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, TCLP, and other leachates/extracts. Certain solid and sludge type wastes may also be analyzed; however, Method 7471A is usually the method of choice. All matrices require sample preparation prior to analysis.

1.5. Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters, and domestic and industrial wastes. All matrices require sample preparation prior to analysis.

1.6. Methods 7471A and 7471B are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, wastes, wipes, biological material, and sludge-type materials. All matrices require sample preparation prior to analysis.

1.7. The TestAmerica North Canton reporting limit for mercury in aqueous matrices is 0.0002 mg/L except for TCLP or SPLP leachates for which the reporting limit is 0.002 mg/L. The TestAmerica North Canton reporting limit for mercury in solid matrices is 0.1 mg/kg.

1.8. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric (aqueous samples), hydrochloric, and nitric acids. Organic mercury compounds are oxidized with potassium permanganate, potassium persulfate (aqueous samples), and the mercury reduced

to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Chemical and physical interferences may be encountered when analyzing samples using this method.

4.3. Potassium permanganate, which is used to break down organic mercury compounds, also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.4. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.5. Chlorides can cause a positive interference. Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (maximum 25 mL); because during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride

4.6. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances

present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.7. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs, the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.8. The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. Refer to Appendix B for Contamination Control Guidelines.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 g/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.

Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydroxyl-amine Hydro-chloride	Corrosive Poison	None	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Corrosive to the eyes. Irritant and possible sensitizer. May cause burns to the skin.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.7. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.8. Cylinders of compressed gas must be handled with caution in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory, and the gas led to the instrument through approved lines.

5.9. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature-controlled water bath (capable of maintaining temperature of 90- 95 °C).

6.2. Atomic Absorption Spectrophotometer equipped with:

6.2.1. Absorption cell with quartz end windows perpendicular to the longitudinal axis. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.2.2. Mercury-specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL)

6.2.3. Peristaltic pump which can deliver 1 L/min air

6.2.4. Flowmeter capable of measuring an airflow of 1 L/min

6.2.5. Recorder or printer

6.2.6. Drying device to prevent condensation in cell.

Note: Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.

6.3. Plastic bottles – 250 mL

6.4. Nitrogen or argon gas supply, welding grade or equivalent

6.5. Calibrated automatic pipettes

6.6. Class A volumetric flasks

6.7. Top-loading balance, capable of reading up to two decimal places

6.8. Thermometer (capable of accurate readings at 95 °C)

6.9. Disposable cups or tubes

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (10 ppm) mercury standards are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017. Refer to the Standards Logbook for details on standard preparation.

7.3. Working mercury standard (0.1 ppm): Take 1 mL of the stock mercury standard (Section 7.2) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot. Refer to the Standards Logbook for details on standard preparation.

7.4. The calibration standards must be prepared fresh daily from the working standard (Section 7.3) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into sample preparation bottles and proceeding as specified in Section 11.1. The LCS solution is prepared by transferring 5.0 mL of working standard (Section 7.3) into sample preparation bottles and proceeding as specified in Section 11.1. Refer to the Standards Logbook for details on standard preparation.

Note: Alternate approaches to standard preparation may be taken, and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I (Appendix A) are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.5. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.6. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.7. Nitric acid (HNO_3), concentrated, trace metal grade or better

7.8. Hydrochloric acid (HCl), concentrated, trace metal grade or better

7.9 Sulfuric acid (H_2SO_4), concentrated, traces metal grade or better.

7.10 Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.11 Stannous chloride solution: Add 50g ± 0.5g of stannous chloride and 25 mL of concentrated HCl, and bring to a final volume of 500 mL with DI water.

Note: Stannous sulfate may be used in place of stannous chloride. Prepare the stannous sulfate solution according to the recommendations provided by the instrument manufacturer.

7.12 Sodium chloride-hydroxylamine hydrochloride solution: Add 240g ± 0.5g of sodium chloride and 240g ± 0.5g of hydroxylamine hydrochloride to every 2000 mL of reagent water.

7.13 Potassium permanganate, 5% solution (w/v): Dissolve 100g of potassium permanganate for every 2000 mL of reagent water.

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7.14 Potassium persulfate, 5% solution (w/v): Dissolve 100 g of potassium persulfate for every 2000 mL of reagent water.

8 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample holding time for mercury is 28 days from time of sample collection to the time of sample analysis.

8.2 Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3 Soil samples and biological material do not require preservation, but must be stored at 4° C ± 2° C (and/or freezing for tissues) until the time of analysis.

9 QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria, and corrective action.

9.1 Initial Demonstration of Capability

9.2 Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well-characterized, laboratory-generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.2.1 Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.3 Preparation Batch - A group of up to 20 samples, excluding QC Samples (LCS, Method Blank, MS, MSD), that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.4 Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis.

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The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at, or above, the reporting or at, or above, 10% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of ten times higher than the blank contamination level).

Note: For Ohio VAP projects, the result must be below the reporting limit or samples must be redigested unless the samples are non-detect.

- Repreparation and re-analysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

- If the above criteria are not met and re-analysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative.**

9.5 Laboratory Control Sample (LCS) - One LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. If the LCS is outside established control limits, the system is out of control and corrective action must occur. See Section 12 for the LCS calculation.

- In the instance where the LCS recovery is greater than the upper control limit and the sample results are less than RL, the data may be reported. Such action must be addressed in the project narrative. LCS recovery must be within 80% - 120%, or in-house control limits for 7470A. For Method 245.1, the LCS must be 85% - 115%.

- In the event an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The LCSD recovery is evaluated using the same control limits as the LCS.

- Corrective action must be repreparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable. For Ohio VAP projects the corrective action must be repreparation and reanalysis of the batch.

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9.6 Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.7 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of, or in addition to, MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix I). See Section 12 for the MS/MSD and RPD calculation.

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 70 - 130 % recovery and 20% RPD must be applied to the MS/MSD for Method 7471A. A control limit of 75 - 125 % recovery for Method 7470A, 70 – 130% for Method 245.1, and 20% RPD must be applied to the MS/MSD, until in-house limits are established. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action must include repreparation and re-analysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds four times the spike level for that analyte, the recovery data are reported as “amount” MSB. The Exception Code is changed to NC. The following two footnotes will appear on the report page—“NC: The recovery and/or RPD were not calculated”, and “MSB: The recovery and RPD were not calculated, because the sample amount was greater than four times the spike amount.”

9.8 Control Limits

9.8.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.8.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMS (QC Browser program).

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9.9 Method Detection Limits (MDLs) and MDL Checks

9.9.1 MDLs and MDL Checks are estab lished by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.9.2 MDLs are easily accessible via LIMS (QC Browser program)

9.10 Nonconformance and Corrective Action

9.10.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager. Procedural deviations are not allowed for Ohio VAP Projects.

10 CALIBRATION AND STANDARDIZATION

10.1 Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2 Due to the differences in preparation protocols, separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3 Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility-specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

10.5 Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at, or below, the reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7 and Table I for additional information on preparing calibration standards and calibration levels.

10.6 The calibration curve must have a correlation coefficient of ≥ 0.995, or the instrument must be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient. NOTE: If any digested calibration standard does not meet SW846 criteria, all associated Ohio VAP samples must be redigested.

10.7 Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level

accuracy and system cleanliness. The ICB result must fall within ± the reporting limit (RL) from zero. See Section 12 for the ICV calculation. If either the ICV or ICB fail to meet criteria, the analysis must be terminated, the problem corrected, and the instrument recalibrated (see Section 11.2.6 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument-related,

the corrective action must include repreparation of the ICV, ICB, CRA, CCV, and CCB with the calibration curve. For Ohio VAP, the sample batch must be redigested.

10.8 Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard at a concentration other than that of the ICV. For Method 245.1, the CCV must be 5% immediately following the calibration. All other CCVs for Method 245.1 must be 90-110%. The CCV result for Methods 7470A, 7471A, 7471B must fall within 20% of the true value for that solution. See Section 12 for the CCV calculation. A CCB is analyzed immediately following each CCV (see Section11.2.6 for required run sequence). The CCB result must fall within ± RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. If the CCV/CCB is biased high and the sample results associated with the CCV/CCB are below the requested reporting limit, then the results can be reported. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If any digested calibration standard does not meet SW846 criteria, all associated Ohio VAP samples must be redigested.

10.9 Detection Limit Standard (CRA) -To verify linearity at the reporting limit, a CRA standard is run at the beginning of each sample analysis run after the ICV/ICB. The CRA standard mercury concentration is 0.2 ug/L. It is recommended that the recovery be ± 50% of the true value, or the standard is either rerun or the problem corrected and the instrument re-calibrated. The CRA is only required when requested.

10.10 For DoD work, refer to SOP NC-QA-016 for specific details.

11 PROCEDURE

11.1 Standard and Sample Preparation Solids

11.1.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples.

11.1.2 Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard (Section 7.3) into a series of sample digestion bottles. The ICB/CCB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. For the ICV,

transfer a 2.5 ml aliquot of the working standard. The ICV working standard must be made from a source other than that used for the calibration standards. For the CCV, transfer a 5 mL aliquot of the working standard into a sample digestion bottle.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

11.1.3 Add reagent water to each standard bottle to make a total volume of 10 mL. Continue preparation as described under Section 11.2 below.

11.1.4 Transfer 0.6g of a well-mixed sample into a clean sample digestion bottle. Continue preparation as described under Section 11.2.

11.2 Water Bath Protocol – Solid Samples

11.2.1 To each LCS **standard** bottle, add 5 mL of reagent water , 5 mL of aqua regia, and 5 mL of the working mercury standard (0.1 ppm) (see Section 7.3).

11.2.2 To each **sample** and Method Blank bottle, add 10 mL of reagent water and 5 mL of aqua regia.

11.2.3 Heat for two minutes in a water bath at 90 - 95 ° C.

11.2.3.1 Add 40 mL of distilled water.

11.2.3.2 Add 15 mL of potassium permanganate solution. Cover containers.

11.2.3.3 Heat for 30 minutes in the water bath at 90 - 95 °C.

11.2.3.4 Cool

11.2.3.4 Add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate.

11.2.3.5 To each **standard** and sample bottles, add 50 mL of reagent water.

11.2.3.6 Continue as described under Section 11.5.

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11.3 Standard and Sample Preparation Waters

11.3.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples. Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard (Section 7.3) into a series of 100 ml class A volumetrics, then dilute to volume. For the ICV, use a 2.5 ml aliquot of the working standard. The ICV working standard must be made from a source other than that used for the calibration standards. For the CCV, transfer a 5 mL aliquot of the working standard into a Class A volumetric, then dilute to volume. The Method Blank consists of 100 mL of reagent water.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

11.3.2 Transfer 100 mL of well-mixed sample or standard to a clean sample digestion bottle. Continue preparation as described under Section 11.4.

Note: Reduced sample volumes can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the same sample to reagent ratio. All samples and standards must be processed similarly.

Note: Spiking is done before the addition of acids or reagents.

11.4 Water Bath Protocol – Water Samples

11.4.2 Add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3.

11.4.3 Add 15 mL of potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If after the addition of up to 25-mL additional permanganate the color does not persist, sample dilution prior to re-analysis may be required.

Note: The sample dilution resultant from the addition of more than the original aliquot of permanganate solution must be compensated for in the final calculation.

11.4.4 Add 8 mL of potassium persulfate solution, cover, and heat for two hours in a water bath at 90 - 95 °C.

11.4.5 Cool samples.

11.4.6 Add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate.

11.5 Sample Analysis

11.5.1 Automated determination. Refer to Appendix C for instrument setup and operation.

11.5.2 Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. concentration of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. The calibration acceptance criteria is listed in Section 10.6. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.5.3 All measurements must fall within the defined calibration range to be valid. Dilute and re-analyze all samples for analytes that exceed the highest calibration standard.

11.5.4 The following analytical sequence is consistent with Methods 7470A, 245.1, and 7471A.

Instrument Calibration
ICV
ICB
CRA if required
Maximum 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.5.5 To facilitate the early identification of QC failures and samples requiring rerun, it is strongly recommended that sample data be reviewed periodically throughout the run.

11.5.6 Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance, and troubleshooting.

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11.6 Analytical Documentation

11.6.2 Record all analytical information in the analytical logbook/logsheet, which may be in electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.6.3 All standards and reagents are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.6.4 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.6.5 Sample results and associated QC are entered into LIMs after final technical review.

11.6.6 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file.

11.6.7 Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. Procedural deviations are not allowed for Ohio VAP projects.

12. DATA ANALYSIS AND CALCULATIONS

12.1 ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2 CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3 Matrix spike recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4 The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

12.5 The final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg, dry\ weight = (C \times V \times D)/(W \times S)$$

Where:

C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor must be omitted from the above equation.

12.6 The final concentration for an aqueous sample is calculated as follows:

$$mg/L = C \times D$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor

12.7 The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8 Appropriate factors must be applied to sample values if dilutions are performed.

12.9 Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002.

13. METHOD PERFORMANCE

13.1 Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.1.

13.2 Training Qualification

13.2.1 The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14 POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15 WASTE MANAGEMENT

15.1 All waste must be disposed of in accordance with Federal, State, and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method

and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2 Waste Streams Produced by this Method

15.2.1 The following waste streams are generated by this method.

15.2.1.1 Acid Waste. This waste disposed of in the designated container labeled "Acid Waste".

15.2.1.2 Acid waste-aqueous waste generated by the analysis. Samples are disposed of in the acid waste drum located in the Metals lab. The contents of the drum are neutralized and released to the POTW.

16. REFERENCES

16.1 References

16.1.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury)

16.1.2 "Methods for the Chemical Analysis of Water and Wastes", Rev. 3.0 (1994)

16.1.3 Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury)

16.1.4 Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Revision 2, January 1998, Method 7471B (Mercury)

16.1.5 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.6 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.7 Corporate Quality Management Plan (CQMP), current version

16.1.8 Revision History

Historical File:		Revision 1.1: 04/17/97		(NC-MT-011) Revision 0: 12/12/07
(formerly CORP-MT-0007NC)		Revision 2.2: 02/06/01		(NC-MT-011 Rev 1 & NC-MT-013 Rev 0): 01/07/09
		Revision 2.3: 05/15/01		
		Revision 2.4: 10/28/02		
		Revision 2.5: 11/22/04		

16.2 Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6. Standards and Reagents, NC-QA-017

16.2.7. Calibration Curves (General), CA-Q-S-005

16.2.8 Section of Calibration Points, CA-T-P-002

16.2.9 Subsampling, NC-IP-001

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1 Modifications/Interpretations from Reference Method

17.1.1 Modifications from Method 7471A

17.1.1.1 Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.1.2 Chapter 1 of SW-846 states that the method blank must not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.1.2 Modifications from both Methods 7470A and 245.1

17.1.2.1 The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.2.2 This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."

17.1.3 Modifications from Method 7470A

17.1.3.1 Chapter 1 of SW-846 states that the method blank must not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit if the samples associated with the method blank are equal to or above the reporting limit.

17.1.4 Modifications from Method 245.1

17.1.4.1 Method 245.1 states that standards are not heated. TestAmerica North Canton prepares heated standards for this method.

17.1.4.2 Stannous Chloride is prepared in hydrochloric acid, instead of sulfuric acid, per instrument manufacturer recommendations.

17.1.4.3 Section 9.3.4 of the method states that the CCB must be less than the MDL. The laboratory uses the criteria that the CCB result must fall within ± RL from zero.

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APPENDIX A - TABLES

TABLE I . MERCURY REPORTING LIMITS, CALIBRATION STANDARD, QC STANDARD AND SPIKING LEVELS

Soil RL (mg/kg)	0.1
Standard Aqueous RL	0.0002
TCLP RL	0.002
Std 0 (mg/L)	0
Std 1/CRA (mg/L)	0.0002
Std 2 (mg/L)	0.0005
Std 3 (mg/L)	0.001
Std 4 (mg/L)	0.005
Std 5 (mg/L)	0.010
ICV (mg/L)	0.0025
CCV/LCS/LCSD (mg/L)	0.005
MS (mg/L)	0.001
TCLP MS	0.005

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TABLE II. SUMMARY OF QUALITY CONTROL REQUIREMENTS

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
ICAL	Daily (every 24 hours)	Curve consists of 5 standards and a blank. Correlation Coefficient of 0.995 or higher and passing ICV/ICB pair.	Recalibrate. If any digested calibration standard does not meet SW846 criteria, all associated Ohio VAP samples must be redigested.
ICV	Beginning of every analytical run	90 - 110 % recovery	Terminate analysis, correct the problem, recalibrate, or reprep with calibration curve (see Section 10). Redigestion of samples is required for OVAP.
ICB	Beginning of every analytical run, immediately following the ICV	The result must be within ± RL from zero	Terminate analysis, correct the problem, recalibrate, or reprep with calibration curve (see Section 10). Redigestion of the samples is also required for OVAP.
CRA	Beginning of every analytical run following the ICB and prior to sample analyses at or near the end of a run required only when requested	Recommended recovery of 50%-150%	Rerun to verify; or correct problem and recalibrate or reprep with the calibration curve (See Sec. 10). CRA only required when requested.
CCV	Every 10 samples and at the end of the run	80 - 120 % recovery for solid samples. Recovery for Method 245.1 = 95-105% following a calibration. All other CCVs for 245.1 are 90-110%. Recovery for Method 7470A, 7471A and 7471B = 80-120% following a calibration. If the CCV is biased high and the samples are < RL the results are acceptable.	Terminate analysis, correct the problem, recalibrate, and rerun all samples not bracketed by acceptable CCV or reprep with calibration curve. If CCV is biased high and samples are ND, results can be reported (see Section 10). If any digested standard does not meet SW846 criteria, all associated Ohio VAP samples must be redigested

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TABLE II. SUMMARY OF QUALITY CONTROL REQUIREMENTS

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
CCB	Immediately following each CCV	The result must be within ± RL from zero	Terminate analysis, correct the problem, recalibrate, and rerun all samples not bracketed by acceptable CCB or reprep with calibration curve. Redigestion of the samples is also required for OVAP. If CCB is biased high and samples are ND, results can be reported (see Section 10).
Method Blank	One per sample preparation batch of up to 20 samples	The result must be less than or equal to the RL Sample results greater than 20x the blank concentration are acceptable Samples for which the contaminant is < RL do not require redigestion (see Section 9.4)	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.
Laboratory Control Sample/Laboratory Control Sample Duplicate(LCS/LCSD)	One per sample preparation batch of up to 20 samples	Aqueous LCS/LCSD must be within 80 - 120% recovery or in-house control limits For Method 245.1, the LCS must be 85 – 115%.	Terminate analysis, correct the problem, redigest, and re-analyze all samples associated with the LCS (see Section 9.5).
Matrix Spike	One per sample preparation batch of up to 20 samples	75 - 125 % recovery or in-house control limits for 7470A. For Method 245.1, recovery limits are 70% - 130%. If the MS/MSD is out for an analyte, it must be in control in the LCS	In the absence of client-specific requirements, flag the data. No flag required if the sample level is > 4 times the spike added (see Section 10).
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits for 7470A. For Method 245.1, recovery limits are 70% - 130%. RPD ≤ 20% (see MS)	See Corrective Action for Matrix Spike

APPENDIX B - CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas should be kept scrupulously clean.

Powdered Gloves should not be used in the metals laboratory since the powder contains zinc, as well as other metallic analytes. Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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APPENDIX C – INSTRUMENT SETUP

Hg Analysis (Leeman PS200II)
System Initialization and Warmup

To Set Up Instrument for Analysis

1. F1 Menu
2. **A**utosampler
    A. **R**ack Entry
    B. **E**dit (ex. Rack 1), Enter
    C. Cup **I**D - Enter (clears sample #'s)
    E. Press Insert Key and move cursor with arrows to cup ID and begin typing labels.
3. Press F2 Macro key and type in – Hg
    A. Enter folder name - ex., HG0306, Enter. If folder does not exist, type Y - Enter.
    B. Type in: "Rack 1", "Rack 2" etc., Enter.
    C. Type in: FROM CUP TO CUP
        Ex. = 1 30

Do the same for Position 2 if needed. If not needed, you must press "Enter" three times to begin analysis.

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Hg Analysis (HYDRA AA)

Instrument Instruction

H4 Analysis

There are 3 separate screens to use--WinHgRunner, WinHgDatabase, and Rack Editor

(Turn on Hg lamp prior to analysis)

1. To Set your Protocol and Dataset

   WinHgDatabase – Select a previous protocol, then Save Protocol. Type in file name (ex.: HG40101), then select the (RN) key. This will take you to the WinHgRunner screen.

   WinHgRunner – File, New, type in dataset name (ex.: 0101A). Type in batch name (ex.: Water or Solid). Go back to WinHgDatabase to locate the new protocol.

2. Typing Labels

   Rack Editor - File, New (pick 44 rack). Type in labels under sample ID, and Save As (ex.: 0101A).

3. Activate Gas and Pump

   WinHgRunner – under Control tab, turn on Gas and pump, and pour calibration standards.

4. To Calibrate Curve

   WinHgRunner – under Standard tab, select S1 S2 S3 S4 S5 S6 Rep1. To begin analyzing, select Stnd Auto tab.

5. To Check Calibration Curve

   WinHgDatabase – under Cal Curve tab linear range >0.995, then accept curve.

6. Verification Standards

   WinHgRunner – under the Standard tab, select C1 (ICV) C2 (ICB) C3 (CRA). To begin analysis, select Ck Std Auto tab.

7. Checking Verification Standards:

   WinHgRunner – Select the Report Tab to review results.

8. To Begin Analyzing Sample with CCV and CCB:

   WinHgRunner – under Standard tab, select C4 (CCB) and C5 (CCV).

   WinHgRunner – under Sample tab, type in rack name, start cup, end cup, cups per rack (44). To start analysis, select the Run Auto tab.

9. View Results

   WinHgRunner – Select the Report tab.

PRINTING REPORTS

1. To Print Cal Curve

   WinHgDatabase – under Cal Curve tab**,** select Print Cal to print curve.

2. To Print Report

   WinHgDatabase – under Report tab under Format, turn on Report. Then select Generate to print.

3. To Transfer Run

   WinHgDatabase – under Report tab under Format, turn on PRN file and type in file (ex. H:/40101A). Then select Generate to transfer run.

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Title: ACID DIGESTION FOR SOLID SAMPLES

[Method: SW846 Method 3050B]

Approvals (Signature/Date):

Technology Specialist	08/09/10 Date	Health & Safety Coordinator	07/28/10 Date
Quality Assurance Manager	08/09/10 Date	Laboratory Director	08/08/10 Date
Technical Director	08/09/10 Date		

This SOP was previously identified as SOP No. NC-IP-010, Rev 1, dated 01/07/09

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of soil samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and Inductively Coupled Plasma-Mass Spectrometry (ICPMS) as specified in SW846 Method 3050B.

1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP or ICPMS for the elements listed in Table I (Appendix A). Other elements and matrices may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This method is not a total digestion, but will dissolve almost all metals that could become "environmentally available". By design, metals bound in silicate structures are not dissolved by this procedure, as they are not usually mobile in the environment. This SOP can be applied to metals in solids, sludges, wastes, sediments, biological samples, and wipes.

1.4. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. A representative 1 gram (wet weight) portion of sample is digested in nitric acid and hydrogen peroxide. The digestate is refluxed with hydrochloric acid for ICP and ICPMS analysis. The digestates are then filtered and diluted to 100 mL.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), latest version.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination, and take appropriate measures to minimize or avoid them. All glassware is cleaned per SOP NC-QA-014.

4.2. The entire work area, including the bench top and fume hood, must be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

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4.3. Boron from the glassware may leach into the sample solution during, and following, sample processing. For critical low-level determinations of boron, only quartz and/or plastic labware are recommended.

4.4. Visual interferences or anomalies, such as foaming, emulsions, precipitates, etc., must be documented.

4.5. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric media.

4.6. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.

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Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.5 The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples must be done in a fume hood. The analyst must also be aware of the potential for a vigorous reaction.

5.6 Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.7 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8 Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.9 Acid/peroxide spills must be neutralized immediately, flushed with water and cleaned up using appropriate spill kits.

5.10 Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire polished as an alternative to disposal.

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6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, digestion block, steam bath, or other heating source capable of maintaining a temperature of 91-99°C

6.2. Calibrated thermometer that covers a temperature range of 0-200°C

6.3. Vapor recovery device (Watch glasses, ribbed or other device)

6.4. Whatman No. 41 filter paper or equivalent

6.5. Funnels or equivalent filtration apparatus

6.6. Analytical balance capable of accurately weighing to the nearest 0.01 grams

6.7. Repeaters or suitable reagent dispensers

6.8. Calibrated automatic pipettes with corresponding pipette tips: 100uL, 500uL, 1mL-5mL

6.9. Class A volumetric flasks

6.10. Plastic digestate storage bottles, such as Corning Snap Seals™ (may be used if their accuracy is documented and is better than 2%)

6.11. Boiling Stones: Ultra Pure PTFE or equivalent

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017.

7.3. Working ICP LCS/MS spike solution. Prepare the ICP LCS/MS working spike solutions from custom stock standards to the final concentration listed in Table II. The working spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS solution must be made fresh every three months. Refer to the Standards

Logbook for details on standard preparation.

7.4. ICPMS LCS/MS spike solution. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are custom made so the final concentrations after spiking equals the concentrations listed in Table III.

7.5. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom solution, the individual facility must purchase a solution from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.6. Nitric acid (HNO_3), concentrated, trace metal grade or better

7.7. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water

Note: When preparing diluted acids, always add acid to water. If the water is added to the acid a violent reaction may occur.

7.8. Hydrochloric acid (HCl), concentrated, trace metal grade or better

7.9. 30% Hydrogen peroxide (H_2O_2), Ultrapure grade

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of sample collection to the date of analysis.

8.2. Soil samples do not require preservation.

9. QUALITY CONTROL

Table IV (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria, and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using Method 3050B the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The result of the MDL determination must be below the TestAmerica North Canton reporting limit. Criteria for DoD work is noted in SOP NC-QA-016.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples.

The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which must contain all the analytes of interest. The results of the initial demonstration study must be acceptable before analysis of samples may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.1.2.2. Calculations and acceptance criteria for QC check samples are given in the determinative SOP NC-MT-012.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks are contained within the individual analytical method SOPs. If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be redigested.

9.4.1. The MB is prepared by weighing a 1g aliquot of boiling chips. The MB is then processed as described in Section 11.9.

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch. Table II provide the details regarding the stock, working standards, and final spike concentrations for ICP and ICPMS. Refer to Section 7 for instructions on preparation of the aqueous LCS.

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9.5.1. The ICP LCS is prepared by spiking a 1g aliquot of boiling chips with 2 mL of the working LCS/MS spike solution (Section 7.4). The ICPMS LCS is prepared by spiking a 1g aliquot of boiling chips with 1 mL of the LCS/MS solution (Section 7.4). The LCS is then processed as described in Section 11.9.

9.6. Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.7. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. Corrective action when MS results fail to meet control limits does not include repreparation of samples unless the results indicate that a spiking error may have occurred. Table II provides the details regarding the stock, working standards and final matrix spike concentrations for ICP and ICPMS. Refer to Section 7for instructions on preparation of the working matrix spike solutions.

9.7.1. The ICP matrix spike is prepared by spiking a 1g aliquot of sample with 2 mL of the working LCS/MS spike solution (Section 7). The ICPMS matrix spike is prepared by spiking a 1g aliquot of sample with 1 mL of the LCS/MS solution (Section 7.4). The matrix spike is then processed as described in Section 11.9.

10. CALIBRATION AND STANDARDIZATION

10.1. Hotblock temperature must be verified daily for each unit used, and must be recorded in a hotblock temperature log.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. Deviations are not allowed for Ohio VAP projects.

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11.3. The heating procedures are carried out in a properly functioning hood.

11.4. All samples are to be checked out of Sample Control with an electronic Chain-of-Custody.

11.5. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample. An automatic label printing program is used to reduce transcription errors (QuantIMS option).

11.6. Samples are typically logged in as either waters or soils. Wastes, such as organic liquids or sludges and tissues (animal/vegetable), are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation.

11.7. If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and Reporting Group.

11.8. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards. Refer to Appendix B for details.

11.9. Preparation of Soils, Sediments, and Sludges for Analysis by ICP and ICPMS

11.9.1. Mix sample thoroughly by stirring with a clean plastic or wooden spoon or spatula.

11.9.2. For each digestion procedure required (i.e., ICP or ICPMS), weigh a 1g portion of solid and record the exact weight to the nearest 0.01g. A 2g sample size may also be used if needed to meet the reporting limits.

Note: Wipe samples are not weighed. The entire wipe is used.

11.9.3. Measure additional aliquots of the designated samples for the MS and MSD analyses.

11.9.4. Add 10 mL of 1:1 HNO_3 and mix the sample.

11.9.5. Heat sample to $95^{o} \pm 4°$ C and reflux for 10 minutes without boiling, using a vapor recovery device.

Note: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of analyte and the sample must be reprepared.

11.9.6 Add 5 mL of concentrated HNO_3.

11.9.7 Reflux at $95^{o} \pm 4°$ C for 30 minutes. (Add reagent water, as needed, to ensure that the volume of solution is not reduced to less than 5 mL.)

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11.9.8 Add approximately 2 mL of reagent water and 1 mL of 30 % H_2O_2. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

11.9.9 Continue adding 30% H_2O_2 in 1 mL aliquots until effervescence is minimal or sample appearance is unchanged. Make sure effervescence subsides before each addition of H_2O_2.

Note: Do not add more than a total of 10 mL of 30 % H_2O_2.

11.9.10 Continue heating at $95^o \pm 4°$ C until the volume is reduced by cooking two hours or to approximately 5-10 mL.

11.9.11 Add 10 mL of concentrated HCL and reflux for an additional 15 minutes without boiling.

11.9.12 Allow the sample to cool.

11.9.13 Filter sample through Whatman 41 filter paper or equivalent, that has been rinsed with deionized water, into a measuring bottle (for example, Corning Snap Seals™). These may be used if their accuracy is documented and is better than $\pm$ 2%. Rinse sample container and filter paper with reagent water to ensure complete sample transfer.

11.9.14 Dilute sample to 100 mL with reagent water into a 120 mL graduated Snap Seal. The sample is now ready for analysis.

11.10. Analytical Documentation

11.10.1 Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.10.2 All standards and reagents are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. Acceptance criteria are given in the determinative SOPs.

13.2. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the analysis of field samples under this SOP may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

13.3. Training Qualification

The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recyling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1. The following waste streams are produced when this method is carried out.

15.2.1.1. Acidic waste containing nitric acid generated by the extraction. This waste is disposed of in a designated container labeled "Acid Waste".

15.2.1.2. Contaminated disposable materials utilized for the analysis. This waste is disposed of in a designated container labeled "Solid Waste".

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996, Method 3050B

16.1.2. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5. Revision History

Historical File:		Revision 2.1: 02/11/00		Revision 0: 07/18/08 (NC-IP-010)
(formerly CORP-IP0002NC)		Revision 2.2: 09/25/01		Revision 1: 01/07/09
		Revision 2.3: 01/18/02		
		Revision 2.4: 02/19/03		
		Revision 2.5: 12/02/04		
		Revision 2.6: 07/29/07		

16.2. Associated SOPs and Policies, current version

16.2.1. Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Methods 6010B and 200.7, NC-MT-012

16.2.2. Inductively Coupled Plasma-Mass Spectrometry, EPA Methods 6020 and 200.8, NC-MT-002

16.2.3. TestAmerica North Canton Quality Control Program, QA-003

16.2.4. Glassware Washing, NC-QA-014

16.2.5. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.6. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.7. Supplemental Practices for DOD Project Work, NC-QA-016

16.2.8. Standards and Reagents, NC-QA-017

16.2.9. Subsampling, NC-IP-001

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Method Deviations

17.1.1. The laboratory uses the same preparation procedure for Method 6010B and 6020. Hydrochloric acid can be used for Method 6020 due to the collision cell technology on newer instruments. Due to the potential chloride interferences, and possible inability to analyze for arsenic and tin, the laboratory must follow the instrument manufacturer guidelines pertaining to the use of HCL and ICP/MS analyses.

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APPENDIX A: TABLES

TABLE I

Method 3050A Approved Analyte List

Element	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

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TABLE II

ICP Soil Matrix Spike and LCS Levels

Element	Working LCS/MS Standard (mg/L)	Soil MS/LCS Level * (mg/kg)
Aluminum	100	200
Antimony	25	50
Arsenic	100	200
Barium	100	200
Beryllium	2.5	5
Cadmium	2.5	5
Calcium	2500	5000
Chromium	10	20
Cobalt	25	50
Copper	12.5	25
Iron	50	100
Lead	25	50
Magnesium	2500	5000
Manganese	25	50
Molybdenum	50	100
Nickel	25	50
Potassium	2500	5000
Selenium	100	200
Silver	2.5	5
Sodium	2500	5000
Thallium	100	200
Vanadium	25	50
Zinc	25	50
Boron	50	100
Tin	100	200
Titanium	50	100

* Final soil spike concentration based on the addition of 2.0 mL working spike (Section 7.3) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

TABLE III

ICPMS Soil Matrix Spike and LCS Levels

Element	Working LCS/MS Standard (mg/kg)	Soil MS/LCS Level * (mg/kg)
Aluminum	1000	1000
Antimony	10	10
Arsenic	10	10
Barium	10	10
Beryllium	10	10
Cadmium	10	10
Calcium	1000	1000
Chromium	10	10
Cobalt	10	10
Copper	10	10
Iron	1000	1000
Lead	10	10
Magnesium	1000	1000
Manganese	10	10
Molybdenum	10	10
Nickel	10	10
Potassium	1000	1000
Selenium	10	10
Silver	10	10
Sodium	1000	1000
Strontium	10	10
Thallium	10	10
Vanadium	10	10
Zinc	10	10
Boron	10	10
Tin	10	10
Titanium	10	10

* Final soil spike concentration based on the addition of 1.0 mL working spike (Section 7.4) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

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TABLE IV

Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples	Refer to determinative SOPs: NC-MT-002 NC-MT-012	Redigest and re-analyze samples
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples	Refer to determinative SOPs: NC-MT-002 NC-MT-012	Redigest and re-analyze all samples associated with the LCS
Matrix Spike	One per sample preparation batch of up to 20 samples	Refer to determinative SOPs: NC-MT-002 NC-MT-012	Reprep not required unless preparation error suspected
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: NC-MT-002 NC-MT-012	See Corrective Action for Matrix Spike

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APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the Metals Lab. All work areas must be kept scrupulously clean.

Powdered gloves must not be used in the Metals Lab since the powder contains zinc, as well as other metallic analytes.

Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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Title: ACID DIGESTION FOR AQUEOUS SAMPLES

[Methods: SW846 3005A, 3010A, and MCAWW 200 Series]

Approvals (Signature/Date):

Technology Specialist	05/06/11 Date	Health & Safety Coordinator	05/09/11 Date
Quality Assurance Manager	05/13/11 Date	Technical Director	05/06/11 Date
Laboratory Director	05/12/11 Date		

This SOP was previously identified as SOP No. NC-IP-011, Rev 1, dated 01/28/10

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of aqueous samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma-Mass Spectrometry (ICPMS) using the MCAWW 200 series methods (NPDES) and SW846 Methods 3005A, and 3010A.

1.2. The applicability of each of these preparation protocols to specific analytes is detailed in Tables I and II (Appendix A). Additional elements may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This SOP provides procedures applicable to the preparation of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, certain aqueous sludges, and leachates/extracts.

1.4. SW846 Method 3005A / MCAWW Method 200.8 are used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP and ICPMS.

1.5. MCAWW Method 200.7 is used to prepare surface water, domestic and industrial waste samples for total, total recoverable, and dissolved metals determination by ICP.

1.6. SW846 Method 3010A is used to prepare aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for total metals analysis by ICP.

2. SUMMARY OF METHOD

2.1. Method 3005A / Method 200.7 / Method 200.8 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP and ICPMS

2.1.1 A representative aliquot of sample is heated with nitric and hydrochloric acids and reduced to a low volume. The digestate is filtered (if necessary) and diluted to volume.

2.2. Method 3010A / Method 200.7 / - Preparation for Total Metals Analysis by ICP

2.2.1 A representative aliquot of sample is refluxed with nitric acid. After the digestate has been reduced to a low volume, it is refluxed with 1:1 hydrochloric acid, filtered (if necessary), and diluted to volume.

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3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. All glassware is cleaned per SOP NC-QA-014.

4.2. The entire work area, including the bench top and fume hood, must be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix B for additional contamination control guidelines.

4.3. Boron from the glassware will migrate into the sample solution during and following sample processing. For critical low level determinations of boron, it is recommended quartz or plastic labware be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not soluble with acids.

4.5. Visual interferences or anomalies (such as dilution due to oily matrix) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.7. Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample.

4.8. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

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5.2. Samples that contain high concentrations of carbonates, or organic material or samples that are at elevated pH, can react violently when acids are added.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.5. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples must be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

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5.6. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.9. Acid spills must be neutralized immediately, flushed with water, and cleaned up using appropriate spill kits.

5.10. Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire-polished as an alternative to disposal.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, digestion block, or other adjustable heating source capable of maintaining a temperature of 90-95°C

6.2. Calibrated thermometer that covers a temperature range of 0-200°C

6.3. Griffin beakers of assorted sizes or equivalent. Equivalent containers may be disposable digestion cups for digestion blocks which are certified by the manufacturer and calibrated by the laboratory.

6.4. Watch glasses, ribbed or equivalent

6.5. Whatman No. 41 filter paper or equivalent

6.6. Funnels or equivalent filtration apparatus

6.7. Graduated cylinder or equivalent capable of measuring 50 mL within 3% accuracy

6.8. Analytical balance capable of accurately weighing to the nearest 0.01 grams

6.9. Repeaters or suitable reagent dispensers

6.10. Calibrated automatic pipettes with corresponding pipette tips

6.11. Class A volumetric flasks

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6.12. pH indicator strips (pH range 0 - 6)

6.13. Plastic digestate storage bottles, such as Corning Snap Seals (may be used if their accuracy is documented and is better than 2%)

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks (MB) as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017.

7.3. Working ICP laboratory control sample (LCS)/matrix spike (MS) spike solution: Prepare the ICP laboratory control sample (LCS)/matrix spike (MS) working spike solution from custom stock standards to the final concentration listed in Table III. The working spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP laboratory control sample (LCS) solution must be made fresh every six months.

7.4. The ICPMS laboratory control sample (LCS)/matrix spike (MS) spike solution is provided directly by the vendor. No further standard preparation is necessary.

7.5. The TCLP matrix spike (MS) working spike solution is provided directly by the vendor, no further standard preparation is necessary. Refer to Table V for final digestate spike concentrations.

7.6. The laboratory control sample (LCS) and matrix spike (MS) samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom solution, the individual facility must purchase a solution from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.7. Aqueous laboratory control samples (LCS) and matrix spike samples are prepared as described in Sections 9.5 and 9.6. Refer to Tables III and IV (Appendix A) for details regarding the stock, working standard and final digestate spike concentrations for ICP and ICPMS laboratory control sample (LCS) and matrix spike preparations.

7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better

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7.9. Hydrochloric acid (HCl), concentrated, trace metal grade or better

7.10. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids, always add acid to water. If the water is added to the acid, a violent reaction may occur.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and must be stored in either plastic or glass. If boron is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Metals samples that are preserved at the laboratory must be held for 24 hours before digestion. For metals samples that require preservation, the time of acid addition must be noted by the Sample Receiving Dept.

Note: If the samples are preserved the same day of collection, the 24-hour waiting period is not required

8.4. For dissolved metals analysis, the samples must be filtered through a 0.45 um filter prior to preservation. Filtration must be done in the field. In the event that samples are not field filtered, filtration occurs in the laboratory prior to preparation.

9. QUALITY CONTROL

9.1. Preparation Batch: A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank (MB), a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the matrix spike/matrix spike duplicate (MS/MSD). If clients specify specific samples for matrix spike/matrix spike duplicate (MS/MSD), the batch may contain multiple matrix spike/matrix spike duplicate (MS/MSD) pairs.

9.2. Sample Count: Laboratory generated QC samples (method blanks (MB), laboratory control sample (LCS), matrix spike/matrix spike duplicate (MS/MSD) are not included in the sample count for determining the size of a preparation batch.

9.3. Method Blank (MB): One method blank (MB) must be processed with each preparation batch. The method blank (MB) consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank (MB) is used to identify any system and process interferences or

contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

9.3.1 Aqueous method blanks (MB) are prepared by taking 50 mL of reagent water through the appropriate procedure as described in Section 11.

9.3.2 TCLP method blanks (MB) are prepared by taking 50 mL of leachate fluid through the appropriate procedure as described in Section 11.

9.4. Laboratory Control Sample (LCS): One aqueous laboratory control sample (LCS) must be processed with each preparation batch. The laboratory control sample (LCS) must contain all analytes of interest and must be carried through the entire analytical procedure. The laboratory control sample (LCS) is used to monitor the accuracy of the analytical process. Ongoing monitoring of the laboratory control sample (LCS) results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of laboratory control sample (LCS) results are contained within the individual analytical method SOPs. Refer to Sections 7.3 and 7.4 for instructions on preparation of the aqueous laboratory control sample (LCS) spike solution.

9.4.1 The aqueous laboratory control sample (LCS) is prepared by spiking a 50 mL aliquot of reagent water with 1.0 mL for ICP and 0.5 mL for ICPMS of the working laboratory control sample (LCS) matrix spike (MS) spike solution

(Sections 7.3 or 7.4). The laboratory control sample (LCS) is then processed through the appropriate procedure as described in Section 11.

Note: TCLP laboratory control sample (LCS) is prepared by taking 50 mL of leachate fluid through the appropriate procedure as described in Section 11.

9.5. Additional information on QC samples can be found in QA Policy QA-003.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD): One matrix spike/matrix spike duplicate MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the matrix spike(MS)) prepared and analyzed along with the sample and matrix spike (MS). Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to matrix spike/matrix spike duplicates (MS/MSDs). The matrix spike/matrix spike duplicate (MS/MSD) results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for matrix spike/matrix spike duplicate (MS/MSD) analysis.

9.6.1 The aqueous matrix spike (MS) sample is prepared by spiking a 50 mL aliquot of a sample with 1.0 mL for ICP and 0.5 mL for ICPMS of the working laboratory control sample (LCS)/ matrix spike/matrix spike duplicate (MS/MSD) spike solution (Sections

7.3 or 7.4). The matrix spike (MS) sample is then processed as described in Section 11.

9.6.2 The TCLP matrix spike/matrix spike duplicate (MS/MSD) sample is prepared by spiking a 50 mL aliquot of a leachate with 0.5 mL of the working TCLP spike solution (Section 7.5). The matrix spike/matrix spike duplicate (MS/MSD) sample is then processed as described in Section 11.

Note: The TCLP matrix spike standard must be added prior to preservation of the leachate.

Note: If analytes outside of the RCRA list are requested, l mL of additional spiking solution(s) are added.

9.7. Control Limits

9.7.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.7.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMS.

9.8 Method Detection Limits (MDLs) and MDL Checks

9.8.1 MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.8.2 MDLs are easily accessible via LIMS.

9.9 Nonconformance and Corrective Action

9.9.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager. Procedural deviations are not allowed for Ohio VAP projects.

10. CALIBRATION AND STANDARDIZATION

10.1 The hotplate/hotblock temperature must be verified daily for each hotplate used, and must be recorded on a hotplate/hotblock temperature log.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or

other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file. Procedural deviations are not allowed for Ohio VAP projects.

11.2. All digestion procedures must be carried out in a properly functioning hood.

11.3. All samples are to be checked out of Sample Control with an electronic chain of custody.

11.4. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample.

11.5. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment, etc.), contact the lab supervisor or project manager for further instructions. In some cases, it may be more appropriate to process these samples as solids.

11.6. If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and Reporting group.

11.7. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.8. For DoD work, refer to SOP NC-QA-016 for specific details.

11.9. The following procedure must be followed for all aqueous sample preparations.

11.9.1 Mix sample by shaking the container.

11.9.2 Measure 50 mL of the sample into a calibrated digestion tube. (Beakers may be used for oil matrices.)

Note: For samples with particulate matter, the aliquot may be taken through a repeated series of shake and pour steps.

11.9.3 Measure two extra aliquots of sample selected for the matrix spike/matrix spike duplicate (MS/MSD) analysis. Spike each aliquot with the appropriate spiking solutions (Sections 7.3 to 7.5 and 9.6).

11.9.4 Measure 50 mL of reagent water into a calibrated digestion tube for the method blank (MB).

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11.9.5 Measure 50 mL of reagent water into a calibrated digestion tube for the laboratory control sample (LCS) and add the appropriate spiking solutions (Sections 7.3 to 7.5 and 9.6).

11.10. Method 3005A / Method 200.7 / Method 200.8 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP / ICPMS

11.10.1 To the sample container, add 1 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl.

11.10.2 Cover with ribbed watch glass.

11.10.3 Heat at 90-95°C until volume is reduced to between 15 and 20 mL. For DoD samples, the final volume is reduced to between 5 and 10 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

11.10.4 Cool the beaker in a fume hood.

11.10.5 Filter sample, if insoluble materials are present, through Whatman 41 filter paper into a plastic storage container, such as a Corning Snap Seal™

Note: If any samples in a preparation batch are filtered, the method blank (MB) and laboratory control sample (LCS) associated with that batch must also be filtered.

11.10.6 Rinse container and filter paper with reagent water to ensure complete sample transfer.

11.10.7 Adjust the final volume to 50 mL with reagent water in the Snap Seal™ container if the digestate was filtered or in the hot block digestion tube if filtering was not necessary. The sample is now ready for analysis.

11.11. Method 3010A / Method 200.7 - Preparation for Total Metals Analysis by ICP Spectroscopy

11.11.1 To the sample container, add 3.0 mL of concentrated HNO_3.

11.11.2 Cover with ribbed watch glass.

11.11.3 Place container on hotblock 90-95°C, and evaporate to a volume of 15-20 mL, while ensuring that no portion of the bottom of the beaker is allowed to go dry. For DoD projects, the sample is evaporated to 5-10 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

11.11.4 Add 5 mL of 1:1 HCl.

11.11.5 Cover and reflux for an additional 15 minutes to dissolve precipitate or residue. Cool in a fume hood.

11.11.6 Filter sample, if insoluble materials are present, through Whatman 41 filter paper into a plastic storage container, such as a Corning Snap Seal™.

Note: If any samples in the QC batch are filtered, the method blank (MB) and laboratory control sample (LCS) associated with that batch must also be filtered.

11.11.7 Rinse container and filter paper with reagent water to ensure complete sample transfer.

11.11.8 Adjust final volume to 50 mL with reagent water in the Snap Seal™ container if the digestate was filtered, or in the hot block digestion tube if filtering was not necessary. The sample is now ready for analysis.

11.12. Analytical Documentation

11.12.1 Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, method blanks (MB), laboratory control samples (LCSs), matrix spike/matrix spike duplicates (MS/MSDs), and any corrective actions or modifications to the method.

11.12.2 All standards and reagents are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.12.3 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.12.4 Sample results and associated QC are entered into LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC Check samples

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs.

13.2. Training Qualification

13.2.1 The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2 Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1 The following waste streams are produced when this method is carried out.

15.2.1.1 Acidic waste containing nitric acid generated by the extraction. This waste is disposed of in the designated container labeled "Acid Waste".

15.2.1.2 Contaminated disposable materials utilized for the analysis. This waste is disposed of in a designated container identified as "Solid Waste".

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, Revision 1, July 1992. Methods 3005A and 3010A

16.1.2 Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983

16.1.3 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.4 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.5 Corporate Quality Management Plan (CQMP), current version

16.1.6 Revision History

Historical File:		Revision 1.2: 03/20/00		Revision 0: 01/07/09 (NC-IP-011)
(formerly CORP-IP-0003NC)		Revision 1.3: 09/25/01		Revision 1: 01/28/10 (NC-IP-011)
		Revision 1.4: 02/19/03		
		Revision 1.5: 12/07/04		
		Revision 1.6: 02/07/07		

16.2. Associated SOPs and Policies, current version

16.2.1 TestAmerica QC Program, QA-003

16.2.2 Glassware Washing, NC-QA-014

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

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16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5 Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6 Standards and Reagents, NC-QA-017

16.2.7 Subsampling, NC-IP-001

16.2.8 Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses, SW846 Methods 6010B, 6010C, and 200.7, NC-MT-012

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference methods

17.1.1 Modifications applicable to SW-846 reference methods

17.1.1.1 The referenced methods as well as Table 3-1 of SW-846 refer to the use of a 100 mL aliquot for digestion. This SOP requires the use of a 50 mL sample size to reduce waste generation. The use of reduced sample volumes are supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document stated "flexibility to alter digestion volumes is addressed and "allowed" by Table 3-1 and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples. EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology." Additionally, in written correspondence from the Office of Solid Waste, Oliver Fordham stated " As a "representative sample" can be assured, scaling causes no loss of precision and accuracy in the analysis."

17.1.2 Modifications Specific to Method 3010A

17.1.2.1 Section 11.11.3 of this SOP requires the sample be reduced to a volume of 15 - 20 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL, but also states that no portion of the bottom of the beaker should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the beaker so as to prevent the loss of critical analytes through volatilization.

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17.1.3 Modifications Specific to MCAWW Methods

17.1.3.1 It was determined by technical review that several of the MCAWW methods were equivalent to the SW-846 methods and therefore were combined under the scope of this SOP as described in Section 11.0. The nature of the differences were deemed insignificant in regards to the amount of acid added and the evaporative volume based on the flexibility allowed by the methods (i.e., add additional acid as required) and the subjective wording of the methods (i.e., evaporate to near dryness vs. an exact volume).

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APPENDIX A - TABLES

TABLE I. Approved Preparation Method Analytes - SW846

ELEMENT	Symbol	CAS Number	3005A	3010A
Aluminum	Al	7429-90-5	X	X
Antimony	Sb	7440-36-0	X	
Arsenic	As	7440-38-2	X	X
Barium	Ba	7440-39-3	X	X
Beryllium	Be	7440-41-7	X	X
Cadmium	Cd	7440-43-9	X	X
Calcium	Ca	7440-70-2	X	X
Chromium	Cr	7440-47-3	X	X
Cobalt	Co	7440-48-4	X	X
Copper	Cu	7440-50-8	X	X
Iron	Fe	7439-89-6	X	X
Lead	Pb	7439-92-1	X	X
Magnesium	Mg	7439-95-4	X	X
Manganese	Mn	7439-96-5	X	X
Molybdenum	Mo	7439-98-7	X	X
Nickel	Ni	7440-02-0	X	X
Potassium	K	7440-09-7	X	X
Selenium	Se	7782-49-2	X	X
Silver	Ag	7440-22-4	X	X
Sodium	Na	7440-23-5	X	X
Thallium	Tl	7440-28-0	X	X
Vanadium	V	7440-62-2	X	X
Zinc	Zn	7440-66-6	X	X

X - Designates that the preparation method is approved for an element.

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE II. Approved Preparation Method Analytes – NPDES

ELEMENT	Symbol	CAS Number	200.7 (9.4)	200.7 (9.3)
Aluminum	Al	7429-90-5	X	X
Antimony	Sb	7440-36-0	X	X
Arsenic	As	7440-38-2	X	X
Boron	B	7440-42-8	X	X
Barium	Ba	7440-39-3	X	X
Beryllium	Be	7440-41-7	X	X
Cadmium	Cd	7440-43-9	X	X
Calcium	Ca	7440-70-2	X	X
Chromium	Cr	7440-47-3	X	X
Cobalt	Co	7440-48-4	X	X
Copper	Cu	7440-50-8	X	X
Iron	Fe	7439-89-6	X	X
Lead	Pb	7439-92-1	X	X
Magnesium	Mg	7439-95-4	X	X
Manganese	Mn	7439-96-5	X	X
Molybdenum	Mo	7439-98-7	X	X
Nickel	Ni	7440-02-0	X	X
Potassium	K	7440-09-7	X	X
Selenium	Se	7782-49-2	X	X
Silicon	Si	7631-86-9	X	X
Silver	Ag	7440-22-4	X	X
Sodium	Na	7440-23-5	X	X
Thallium	Tl	7440-28-0	X	X
Vanadium	V	7440-62-2	X	X
Zinc	Zn	7440-66-6	X	X

X - Designates that the preparation method is approved for an element

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

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TABLE III. ICP Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	Working Laboratory Control Sample (LCS)/Matrix Spike (MS)Standard (mg/L)	Aqueous Laboratory Control Sample (LCS)/ Matrix Spike (MS) Level * (ug/l)
Aluminum	100	2000
Antimony	25	500
Arsenic	100	2000
Barium	100	2000
Beryllium	2.5	50
Cadmium	2.5	50
Calcium	2500	50000
Chromium	10	200
Cobalt	25	500
Copper	12.5	250
Iron	50	1000
Lead	50	500
Magnesium	2500	50000
Manganese	25	500
Molybdenum	50	1000
Nickel	25	500
Potassium	2500	50000
Selenium	100	2000
Silver	2.5	50
Sodium	2500	50000
Thallium	100	2000
Vanadium	25	500
Zinc	25	500
Boron	50	1000
Tin	100	2000
Titanium	50	1000

* Levels shown indicate the spike concentration in the final digestate of the aqueous laboratory control sample (LCS) or matrix spike (MS) based on the addition of 1.0 mL working spike (7.3) to 50 mL of sample.

TABLE IV. ICPMS Aqueous Laboratory Control Sample (LCS) and Matrix Spike/Matrix Spike Duplicate (MS/MSD) Levels

ELEMENT	Working Laboratory Control Sample (LCS)/Matrix Spike (MS) Standard (mg/L)	Aqueous Laboratory Control Sample (LCS)/Matrix Spike (MS) Level* (ug/L)
Aluminum	1000	10000
Antimony	10	100
Arsenic	100	1000
Barium	100	1000
Beryllium	100	1000
Cadmium	100	1000
Calcium	1000	10000
Chromium	100	1000
Cobalt	100	1000
Copper	100	1000
Iron	1000	10000
Lead	100	1000
Magnesium	1000	10000
Manganese	100	1000
Molybdenum	10	100
Nickel	100	1000
Potassium	1000	10000
Selenium	100	1000
Silver	10	100
Sodium	1000	10000
Strontium	100	1000
Thallium	100	1000
Vanadium	100	1000
Zinc	100	1000
Boron	10	100
Tin	10	100
Titanium	10	100

* Levels shown indicate the spike concentration in the final digestate of the aqueous laboratory control sample (LCS) ormatrix spike (MS) based on the addition of 0.5 mL working spike (Section 7.4) to 50 mL of sample.

Note: Spiking levels may be adjusted as long as the concentrations are documented.

TABLE V. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels for ICP

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)*
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

* Levels shown indicate the spike concentration in the final digestate of the aqueous laboratory control sample (LCS) or matrix spike (MS) based on the addition of 0.5 mL working spike (Section 7.5) to 50 mL of sample.

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APPENDIX B

CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains zinc, as well as other metallic analytes. Only vinyl or nitrile gloves must be used in the metals laboratory.

Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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Title: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHODS 8000B, 8021B, 8081A, 8081B, 8082, 8082A, 8151A, 615, 8015B, and 8015C

Approvals (Signature/Date):

Technology Specialist	04/30/11 Date	Health & Safety Coordinator	04/21/11 Date
Quality Assurance Manager	05/01/11 Date	Laboratory Director	04/27/11 Date
Technical Director	04/20/11 Date		

This SOP was previously identified as SOP NC-GC-038, Rev 1, Dated 01/15/09

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1. SCOPE AND APPLICATION

1.1. This SOP describes procedures for analysis of organic analytes by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). The main body of this SOP is based on SW846 Method 8000B. Individual analytes and methods are described in the appendices. See the list of appendices noted in the Table of Contents to determine the appropriate section of the SOP. Reporting limits are listed in each appendix.

2. SUMMARY OF METHOD

2.1. In general, semivolatile analytes in aqueous samples are prepared for analysis using continuous or separatory funnel liquid / liquid extraction (SOP NC-OP-037 and NC-OP-038). Solid samples are prepared using sonication or soxhlet (SOP NC-OP-039 and NC-OP-040). Volatile analytes are prepared for analysis using purge and trap methodology (Appendix A).

2.2. After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4. INTERFERENCES

4.1. Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors such as the Flame Ionization Detector (FID). See Appendices A through E for interferences specific to individual tests and suggested corrective actions. All glassware is cleaned per SOP NC-QA-014.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that prevents splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Refer to the TestAmerica North Canton Corporate Environmental Health and Safety Manual for a complete description of personal protection equipment. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded; other gloves must be cleaned immediately. Latex, Nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review

the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. It is recommended that analysts break up work tasks to avoid repetitive motion tasks, such as opening a large number of vials or containers in one time period.

5.5. Exposure to chemicals must be maintained as low as reasonably achievable. All samples with stickers that read “Caution/Use Hood!” must be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. Opened containers of neat standards must be handled in a fume hood.

5.7. Sample extracts and standards, which are in a flammable solvent, must be stored in an explosion-proof refrigerator.

5.8. When using hydrogen gas as a carrier, all precautions listed in the CSM must be observed.

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5.9. Standard preparation and dilution must be performed inside an operating fume hood.

5.10. The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.11. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.12. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. An analytical system complete with a gas chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

7. REAGENTS AND STANDARDS

7.1. Stock Standards

7.1.1. Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standards for Method 8021B are stored at -10 to -20°C. Other stock standard solutions are stored as recommended by the manufacturer. All stock standards must be protected from light. Stock standard solutions must be brought to room temperature before using.

7.1.2. Semivolatile stock standard solutions must be replaced after one year. Stock standards of gases must be replaced at least every week, unless the acceptability of the standard is demonstrated (Less than 20% drift from the initial calibration is an acceptable demonstration). Other volatile stock standards must be replaced every six months or sooner if comparison with check standards prepared from an independent source indicates a problem.

7.1.3. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If vendor-supplied standard has an earlier expiration date then that date is used. Refer to SOP NC-QA-017, Standards and Reagents, for additional information. The standard preparation information is detailed in the Standard Logbook.

7.2. Calibration Standards

7.2.1. Volatile Calibration Standards

7.2.1.1. The procedure for preparation of volatile standards is given in Appendix A.

7.2.2. Semivolatile Calibration Standards

7.2.2.1. Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at ≤ 6°C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.3. Gases for carrier and make-up: Hydrogen, Helium, Nitrogen, Zero Air.

7.4. Quality control (QC) Standards

7.4.1. QC standards (matrix spiking and Laboratory Control Sample [LCS] standards) are prepared and stored in the same way as calibration standards. They must be made from a stock independent from the calibration standards.

8. SAMPLE PRESERVATION AND STORAGE

8.1. The holding time for semivolatile extracts is 40 days from extraction to analysis. Samples must be refrigerated at $\leq 6^{o}$C. Volatile sample storage conditions and holding times are given in Appendix A.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2. Batch Definition

9.2.1. Batches are defined at the sample preparation stage. Batches must be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TestAmerica North Canton QC Program document (Policy QA-003) for further details of the batch definition. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC.

9.2.2. Quality Control Batch

9.2.2.1. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Laboratory generated QC samples (Method Blank, Laboratory Control Sample (LCS), matrix spike / spike duplicate (MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the matrix spike / spike duplicate (MS/MSD) may be replaced with a matrix spike and sample duplicate (MS/SD).

9.3. Control Limits

9.3.1. In-house historical control limits may be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined periodically. The recovery limits are mean recovery $\pm$ 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Project or program specific control limits may be used in place of in-house limits. Refer to Policy QA-003 for more details.

9.3.2. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries must be reported unless the dilution is more than 5X.

9.3.3. All surrogate, Laboratory Control Sample (LCS), and Matrix Spike (MS) recoveries (except for dilutions) must be entered into LIMS (when available) or other database so that accurate historical

control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes must be reported for all dilutions.

9.3.4. Refer to the QC Program document (Policy QA-003) for further details of control limits.

9.4. Surrogates

9.4.1. All methods must use surrogates to the extent possible. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. Surrogate recoveries must be met in the method blank (MB) and Laboratory Check Samples (LCS). If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure instrument performance is acceptable.
- Recalculate the data and/or re-analyze the extract if either of the above checks reveals a problem.
- The decision to re-analyze or flag the data must be made in consultation with the client. It is only necessary to reprepare / re-analyze a sample once to demonstrate poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

  Note: For DoD QSM, all surrogates must meet criteria. For Ohio VAP Projects, all surrogates must meet criteria unless the samples are ND and the surrogates are out high. Reanalysis or repreparation of the samples is required if these criteria are not met.

9.4.2. If dual column analysis is used, the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in-control result is reported.

9.4.3. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate (MS/MSD), then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the matrix spike (MS) and matrix spike duplicate (MSD) are in control, then repreparation or flagging of the data is required. Repreparation includes the parent sample and matrix spike / spike duplicate (MS/MSD).

9.4.4. Refer to TestAmerica North Canton QC Program document (Policy QA-003) for further details of the corrective actions.

9.5. Method Blanks

9.5.1. For each batch of samples, analyze a method blank. The method blank consists of reagent water for aqueous semivolatile samples and sodium sulfate for semivolatile soils tests (Refer to SOPs NC-OP-037, NC-OP-038, NC-OP-039, and NC-OP-040 for details). For low-level volatile soils, the method blank consists of reagent water and Ottawa sand. For medium-level volatile solids, the method blank consists of methanol and Ottawa sand. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the method blank and affected samples will normally be required. Consultation with the client must take place. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

9.5.2. The method blank must not contain any analyte of interest at, or above, the reporting limit (except common laboratory contaminants, see below) or at, or above, 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.5.3. If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone, phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

9.5.4. Re-extraction and re-analysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.5.5. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action must be taken in consultation with the client.

Note: For Ohio VAP projects, the result must be below the reporting limit or samples must be re-extracted unless the samples are non-detect.

9.5.6. Refer to TestAmerica North Canton QC Program document (Policy QA-003) for further details of the corrective actions.

9.5.7. Refer to SOP NC-QA-016 for further details concerning DoD Project Work.

9.6. Laboratory Control Samples (LCS)

9.6.1. For each batch of samples, analyze a Laboratory Control Sample (LCS). The Laboratory Control Sample (LCS) contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The Laboratory Control Sample (LCS) may also contain the full set of analytes with a subset of control analytes. If any control analyte is outside the laboratory established historical control limits, corrective action must occur. All non-controlling compounds must attain a recovery of 10% or greater if the compound is on the client's list. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

9.6.2. If the batch is not re-extracted and re-analyzed, the reasons for accepting the batch must be clearly presented in the project record and the report.

9.6.3. If re-extraction and re-analysis of the batch is not possible due to limited sample volume or other constraints, the Laboratory Control Sample (LCS) is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6.4. The Laboratory Control Sample (LCS) must have acceptable surrogate recoveries. If surrogate recoveries are low, re-extraction of the Laboratory Control Sample (LCS) and affected samples will normally be required. Consultation with the client should take place. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

9.6.5. Refer to TestAmerica North Canton QC Program document (Policy QA-003) for further details of the corrective action.

9.6.6. If dual column analysis is used, the choice of which result to report is made in the same way as for samples (Section 12.1.2), unless one column is out of control, in which case the in control result is reported.

9.6.7. Laboratory Control Sample (LCS) compound lists are included in the appendices.

9.7. Matrix Spikes/Spike Duplicates (MS/MSD)

9.7.1. For each QC batch, analyze a matrix spike and matrix spike duplicate (MS/MSD). Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory-specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur unless samples for this compound are ND. The initial corrective action must be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the Laboratory Control Sample (LCS) is within limits, then the laboratory operation is in control and analysis may proceed.
- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate (MS/MSD) and the Laboratory Control Sample (LCS), the laboratory is out of control and corrective action must be taken. Corrective action must normally include repreparation and re-analysis of the batch.
- If a matrix spike / matrix spike duplicate (MS/MSD) is not possible due to limited sample, then a Laboratory Control Sample (LCS) duplicate may be analyzed if required by client or regulatory programs. The recovery for each spike of the pair must be within established control limits. If the RPD is out of control, but both accuracy recoveries are within acceptance criteria, prepare an NCM and qualify report.
- The matrix spike / matrix spike duplicate (MS/MSD) must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.7.2. If dual column analysis is used, the choice of which result to report is made in the same way as for samples (Section 12.1.2), unless one column is out of control, in which case the in control result is reported.

9.8. Control Limits

9.8.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.8.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs.

10. CALIBRATION AND STANDARDIZATION

10.1. Internal or external calibration may be used. In either event, prepare standards containing each analyte of interest at a minimum of five concentration levels. The low-level standard must be at, or below, the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.

10.2. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, changing PID lamps or replacing the ECD detector. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance.

10.3. With the exception of Section 10.4 below, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve, AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least five points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points. Third order calibrations require at least seven points.

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10.4. A level may be removed from the calibration if the reason can be clearly documented (for example, a broken vial or no purge run). A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be re-analyzed and the re-analysis used for the calibration. All initial calibration points in a single calibration curve must be analyzed without any changes to instrument conditions, and all points in a single calibration curve must be analyzed within 24 hours.

10.5. External Standard Calibration

10.5.1. Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach, introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

$$\textit{Calibration Factor or Response Factor} \quad (CF) \text{ or } (RF) = \frac{\textit{Area or Height of Peak}}{\textit{Mass Injected (ng)}}$$

Some data systems may use the inverse of this formula. This is acceptable so long as the same formula is used for standards and samples. It is also possible to use the concentration of the standard rather than the mass injected. (This would require changes in the equations used to calculate the sample concentrations). Use of peak area or height must be consistent. However, if matrix interferences would make quantitation using peak area inaccurate for a particular sample, then peak height may be used as a substitute.

10.6. Internal Standard Calibration

10.6.1. The internal standard approach assumes that variations in instrument sensitivity, amount injected etc. can be corrected by determining the ratio of the response of the analyte to the response of an internal standard that has been added to the extract. To use this approach, select one or more internal standard(s) that are similar in analytical behavior to the compounds of interest. Recommended internal standards are given in the appendices. The analyst must demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. If matrix interference is observed, reanalysis of the sample is required. If matrix interference is confirmed, report the second analysis and note in the Case Narrative. . For Ohio VAP projects, the laboratory must re-analyze any sample where the internal standard fails, and there is no evidence of matrix interference. If there is no matrix interference, the sample must be reanalyzed at the original dilution. If the internal standard is within criteria, report the second analysis. If the internal standard is still outside of criteria, the sample must be analyzed at a second dilution. If the internal standard still does not meet criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required.

10.6.2. Introduce each calibration standard into the GC using the technique that will be used for samples. Response factors (RF) for each compound are calculated as follows.

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Response for the analyte to be measured
A_{is} = Response for the internal standard
C_{is} = Concentration of internal standard
C_s = Concentration of the analyte to be determined in the standard

10.7. Calibration Curve Fits

10.7.1. Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the average % RSD of the response factors or calibration factors of all the analytes in the calibration standard taken together is ≤ 20%. The average % RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes. NOTE: This is not allowed for Ohio VAP projects or Update IV Methods.

10.7.2. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits must only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector. Linear or quadratic curve fits may be used if the compounds have historically exhibited a non-linear response and cannot be used to extend the calibration range for compounds that normally exhibit a linear response, but within a narrower calibration range.

10.7.3. Average Response Factor

The average response factor may be used if the average percent relative standard deviation (% RSD) of all the response factors taken together is ≤ 20%.

The equation for average response factor is:

$$\textit{Average response factor} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.7.4. Linear Regression

The linear fit uses the following functions:

10.7.4.1. External Standard

$$y = ax + b$$

or

$$x = \frac{(y - b)}{a}$$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.7.4.2. Internal Standard

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a}$$

Where: C_s = Concentration in the sample
A_s = Area of target peak in the sample
A_{is} = Area of internal standard in the sample
C_{is} = Concentration of the internal standard

10.7.5. Quadratic Curve

The quadratic curve uses the following functions:

10.7.5.1. External standard

$$y = ax + cx^2 + b$$

Where c is the curvature

10.7.5.2. Internal Standard

$$y = a\left(\frac{A_s \times C_{is}}{A_{is}}\right) + c\left(\frac{A_s \times C_{is}}{A_{is}}\right)^2 + b$$

10.8. Evaluation of Calibration Curves

10.8.1. The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.8.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level. The percent relative standard error is calculated as follows.

$$\%\ RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N - P)}}$$

Where:
N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
C_I = True concentration for level i
PC_i = Predicted concentration for level i

Note: When average response factors are used, % RSE is equivalent to % RSD.

10.9. The following requirements must be met for any calibration to be used.

- Response must increase with increasing concentration.
- If a curve is used, the calculated intercept of the curve at zero response must be less than ± the reporting limit for the analyte.
- The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.
- Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

  Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (*r*) and Coefficient of Determination (r^2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on *r*. As a result, a curve may have a very good correlation coefficient (>0.995) while also having > 100% error at the low point.

10.10. Weighting of Data Points

10.10.1. In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and must be used if the data system has this capability.

10.11. Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five-point initial calibration. This action must be with client approval. If the analyte is detected in any of the samples, a five-point initial calibration must be generated, and the sample(s) re-analyzed for quantitation.

10.12. Calibration Verification

10.12.1. 12-hour Calibration

10.12.1.1. The working calibration curve or RF must be verified by the analysis of a mid point calibration standard at the beginning, after every 12 hours, and at the end of the analysis sequence.

10.12.2. Daily Calibration Verification

10.12.2.1. It may be appropriate to analyze a mid-point standard more frequently than every 12 hours. If these calibration verification standards are analyzed, requirements are the same as the 12-hour calibration with the exception that retention times are not updated.

10.12.2.2. Any individual compounds with % D < 15% meet the calibration criteria. The calibration verification is also acceptable if the average of the % D for all the analytes is < 15%, or as noted in individual test sections. This average is calculated by summing the entire absolute % D results in the calibration (including surrogates) and dividing by the number of analytes. Only ND or results below the RL are reported. Any analyte that is reportable as found must have a % difference of < 15% in the calibration verification or 12-hour calibration on the column used for quantitation. Refer to Section 12.1.2 for which result to report. Update IV does not allow use of grand mean. CCV must pass by ± 20%.

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10.12.3. An ICV is analyzed immediately after an initial calibration. The acceptance criteria is ± 20%. If this is not met, a new initial calibration curve is analyzed.

10.12.4. It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.12.5. Samples quantitated by external standard methods must be bracketed by calibration verification standards that meet the criteria listed above. The bracketing standards on the column used for calibration must meet the same criteria as the opening standards. Bracketing is not necessary for internal standard methods.

10.12.6. If the analyst notes that a CCV has failed and can document the reason for failure (e.g., no purge, broken vial, carryover from the previous sample etc.), then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria, then the preceding samples have been successfully bracketed. If adjustments to the instrument are performed before the repeat CCV, then the proceeding samples have not been successfully bracketed; but analysis may continue.

10.12.7. In general, it is not advisable to analyze repeat CCVs on unattended runs. If repeat CCVs are analyzed, then the first must serve as the bracketing standard for the preceding samples and the last must serve as the CCV for the following samples.

10.12.8. If highly contaminated samples are expected, it is acceptable to analyze solvent blanks or primers at any point in the run.

10.12.9. Percent Difference Calculation

10.12.9.1. Percent difference for internal and external methods is calculated as follows:

Internal Standard

$$\%D = \frac{RF_c - \overline{RF}}{\overline{RF}} \times 100$$

External standard

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where: RF_c and CF_c are the response and calibration factors from the continuing calibration

$\overline{RF}$ and $\overline{CF}$ are the average response & calibration factors from the initial calibration

10.12.10. Percent Drift Calculation

10.12.10.1. Percent drift is used for comparing the continuing calibration to a linear or quadratic curve. The criteria for percent drift are the same as for percent difference

$$\%\ Drift = \frac{Calculated\ Conc. - Theoretical\ Conc.}{Theoretical\ Conc.} \times 100\%$$

10.12.10.2. Corrective Actions for Continuing Calibration

10.12.10.3. If the overall average percent drift of all analytes is greater than ± 15%, corrective action must be taken. This may include clipping the column, changing the liner, or

other minor instrument adjustments, followed by re-analyzing the standard. If the overall average percent drift still varies by more than ± 15%, a new calibration curve must be prepared.

10.12.10.4. Corrective Action for Samples

10.12.10.4.1. For internal standard methods, any samples injected after a standard not meeting the calibration criteria must be re-injected.

10.12.10.4.2. For external standard methods, any samples injected after the last good continuing calibration standard must be re-injected.

10.12.10.4.3. If the average percent drift for all the analytes in the calibration is over 15%; but all of the analytes requested for a particular sample have percent drift ≤ 15%, then the analysis is acceptable for that sample.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file. Procedural deviations are not allowed for Ohio VAP Projects.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. Extraction

11.3.1. Extraction procedures are referenced in the SOPs NC-OP-037, NC-OP-038, NC-OP-039, and NC-OP-040, current revision.

11.4. Cleanup

11.4.1. Cleanup procedures are referenced in the SOP NC-OP-025, current revision.

11.5. Gas Chromatography

11.5.1. Chromatographic conditions for individual methods are presented in the appendices.

11.6. Sample Introduction

11.6.1. In general, volatiles analytes are introduced using Purge and Trap as described in Appendix A.

11.6.2. Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.7. Analytical Sequence

11.7.1. An analytical sequence starts with an initial calibration or a calibration verification. Refer to the individual method appendices (Appendices A, B, C, D, and E) for method-specific details of calibration verifications and analytical sequences.

11.7.2. The calibration verification includes analysis of standards containing all single response analytes and updating the retention time windows.

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11.7.3. If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a calibration verification.

11.8. Retention Time Windows

11.8.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three-day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). For multi-response analytes (e.g., Aroclors), use the retention time of major peaks. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

11.8.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid-point of the initial calibration and each 12-hour calibration. The widths of the windows must remain the same until new windows are generated following the installation of a new column.

11.8.3. If the retention time window as calculated above is less than ± 0.05 minutes, use a retention time window appropriate for the analysis and run time. This allows for slight variations in retention times caused by sample matrix.

11.8.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.8.5. Retention time studies are filed in the laboratory.

11.8.6. Corrective Action for Retention Times

11.8.6.1. The retention times of all compounds in the 12-hour calibration or calibration verification standard must be within the retention time window. If this condition is not met, all samples analyzed after the last compliant standard must be re-analyzed, unless the following conditions are met for any compound that elutes outside the retention time window.

11.8.6.2. The retention time of that compound in the standard must be within a retention time range equal to twice the original window.

11.8.6.3. No peak that would be reportable must be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

11.9. Daily Retention Time Windows

11.9.1. The center of the retention time windows determined in Section 11.8 is adjusted to the retention time of each analyte as determined in the 12-hour calibration standards or continuing calibration verification standards. (See Methods 8081A and 8082 Appendices B and C for exceptions for multi-response components.) The retention time windows must be updated at the beginning of each analytical sequence and with each 12-hour calibration or continuing calibration verification.

11.10. Procedural Variations

11.10.1. Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC Manager. The Nonconformance Memo must be filed in the

project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. Procedural deviations are not allowed for Ohio VAP Projects.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1. Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required. Normally confirmation is required on a second column; but if the detector is sufficiently specific or if the sample matrix is well enough defined, single column analysis may be adequate. In some cases, GC/MS confirmation may be required. Client-specific requirements may also define the need for second column confirmation and/or GC/MS confirmation. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column at a concentration greater than the reporting limit (MDL if J flag confirmation required). Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. Many programs require chromatograms before and after manual integration. Additional information on manual integration can be found in SOP CA-Q-S-002.

12.1.2. Dual column quantitation

For confirmed results, two approaches are available to the analyst:

a) the primary column approach, or
b) the better result approach

Both are acceptable to avoid the reporting of erroneous or unconfirmed data.

12.1.2.1. Primary column approach

12.1.2.2. The result from the primary column is normally reported. The result from the secondary column is reported if any of the following three bulleted possibilities are true.

- There is obvious chromatographic interference on the primary column
- The result on the primary column is 40% greater than the result on the secondary column
- Continuing or bracketing standard fails on the primary column but is acceptable on the secondary column. (If the primary column result is > 40% higher than the secondary and the primary column calibration fails, then the sample must be evaluated for re-analysis.)

12.1.2.3. Better result approach

The higher of the two results is normally reported. The higher result is considered better because the higher result is generally higher because of chromatographic interference. For Ohio VAP projects, the higher result must be reported unless the laboratory can demonstrate that a matrix interference caused the result to be elevated. For Ohio VAP projects both columns must meet calibration criteria. The lower result is reported if any of the following two bulleted possibilities are true.

- There is obvious chromatographic interference on the column with the higher result
- The continuing or bracketing calibration on the column with the higher result fails. (If the higher result is > 40% higher and the calibration on the column with the lower result fails, then the sample must be evaluated for re-analysis.)

12.1.3. If the Relative Percent Difference (RPD) between the response on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. RPD is calculated using the following formula:

$$RPD = \frac{|R - R_2|}{\frac{1}{2}(R_1 + R_2)}$$

Where: R=Result

12.1.4. Multi-response Analytes

12.1.4.1. For multi-response analytes, the analyst must use the retention time window, but must rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

12.1.5. The experience of the analyst must weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2. Calibration Range

12.2.1. If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and re-analyzed. Dilutions must target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

12.3. Dilutions

12.3.1. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be re-analyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.3.1.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and only minor matrix peaks are detected, then the sample must be re-analyzed at a more concentrated dilution. Analyst judgement is required to determine the most concentrated dilution that will not result in instrument contamination.

12.3.2. Reporting Dilutions

12.3.2.1. The most concentrated dilution with no target compounds above the calibration range must be reported. Other dilutions may be reported at client request if the lower dilutions will not cause detector saturation, column overload, or carryover. Analyst judgement and client site history will be factors in the reporting of dual dilutions.

12.4. Interferences

12.4.1. If peak detection is prevented by interferences, further cleanup must be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

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12.5. Internal Standard Criteria

12.5.1. If internal standard calibration is used, then the internal standard response in a continuing calibration standard must be within 50 to 150% of the response in the mid level of the initial calibration.

12.5.2. The internal standard response in samples must be within 50-150% of the response of previous continuing calibration standard.

12.6. Calculations

12.6.1. Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

12.6.2. External Standard Calculations

12.6.2.1. Aqueous samples

$$Concentration\ (mg/L) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:
A_x = Response for the analyte in the sample
V_i = Volume of extract injected, µL
D_f = Dilution factor
V_t = Volume of total extract, µL
V_s = Volume of sample extracted or purged, mL
CF = Calibration factor, area or height/ng

12.6.2.2. Non-aqueous Samples

$$Concentration\ (mg/kg) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W)}$$

Where:
W = Weight of sample extracted or purged, g

12.6.2.3. Internal Standard Calculations

12.6.2.4. Aqueous Samples

$$Concentration\ (mg/L) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times V_s)}$$

Where:
C_{is} = Amount of internal standard added, ng
A_{is} = Response of the internal standard
RF = Response factor for analyte

12.6.2.5. Non-aqueous Samples

$$Concentration\ (mg\ /\ kg) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times W \times D)}$$

12.6.3. Surrogate Recovery

12.6.3.1. Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation.

$$\%\ \text{Recovery} = \frac{Concentration\ (or\ amount)\ found}{Concentration\ (or\ amount)\ spiked} \times 100$$

12.6.4. Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.1.1. Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOPs NC-QA-021 and CA-Q-S-006.

13.2. Initial Demonstration

13.2.1. Each laboratory must make a one-time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests, it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample must be equivalent to a mid-level calibration.

13.2.1.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in each appendix.

13.2.1.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

13.3.1. The Group/Team Leader has the responsibility to ensure an analyst who has been properly trained in its use and has the required experience performs this procedure.

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14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

15.2.1. The following waste streams are produced when this method is carried out.

15.2.1.1. Vials containing sample extracts. These vials are placed in the vial waste located in the GC/MS laboratory.

15.2.1.2. **Tubes containing sample extracts for TPH, pesticides, PCBs, and herbicides.** These capped tubes are placed in the PCB/flammable waste located the GC prep laboratory.

15.2.1.3. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCB's must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB and PCB vial waste. PCB containing samples are located through a LIMS query and disposed of as PCB containing.

15.2.1.4. **Extracted solid samples contaminated with methylene chloride/acetone or acetone/hexane.** These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.1.5. **Discarded samples.** These samples are collected in the solid debris drum.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, and Section 8000B

16.2 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.3 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.4 Corporate Quality Management Plan (CQMP), current version

16.5 Revision History

Historical File:		Revision 2.1: 08/12/96		Revision 0: 01/21/08 (NC-GC-038)
(formerly CORP-GC-0001NC)		Revision 3.0: 12/01/97		Revision 1: 01/15/09 (NC-GC-038)
		Revision 5.3: 11/18/99		
		Revision 5.4: 11/10/00		
		Revision 5.5: 03/16/01		
		Revision 5.6: 05/25/01		
		Revision 5.7: 10/01/03		
		Revision 5.8: 02/06/06		

16.2. Associated SOPs and Policies, current version

16.5.1 QA Policy, QA-003

16.5.2 Glassware Washing, NC-QA-014

16.5.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.5.4 Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.5.5 Standards and Reagents, NC-QA-017

16.5.6 Cleanup Procedures for Organic Extractable Samples, NC-OP-025

16.5.7 Supplemental Practices for DoD Project Work, NC-QA-016

16.5.8 Acceptable Manual Integration Practices, CA-Q-S-002

16.5.9 Calibration Curves (General), CA-Q-S-005

16.5.10 Section of Calibration Points, CA-T-P-002

16.5.11 Continuous Liquid / Liquid Extraction of Organic Compounds from Waters Based on Methods SW846 3520C and 600 Series and Waste Dilution Based on Method 3580A, NC-OP-037

16.5.12 Separatory Funnel Extraction of Organic Compounds from Waters Based on Methods SW846 3510C and 600 Series and Waste Dilution Based on Method, NC-OP-038

16.5.13 Sonication Extraction of Organic Compounds from Soils Based on Method SW846 3550C and Waste Dilution Based on Method 3580A, NC-OP-039

16.5.14 Soxhlet (Traditional) Extraction of Organic Compounds from Soils Based on Method SW846 3540C and Waste Dilution Based on Method 3580A, NC-OP-040

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Chapter 1 of SW-846 states the method blank must not contain any analyte of interest at, or above, the Method Detection Limit. This SOP states the Method Blank must not contain any analyte of interest at, or above, the reporting limit. Common lab contaminants are allowed to be up to five times the reporting limit in the method blank following consultation with the client.

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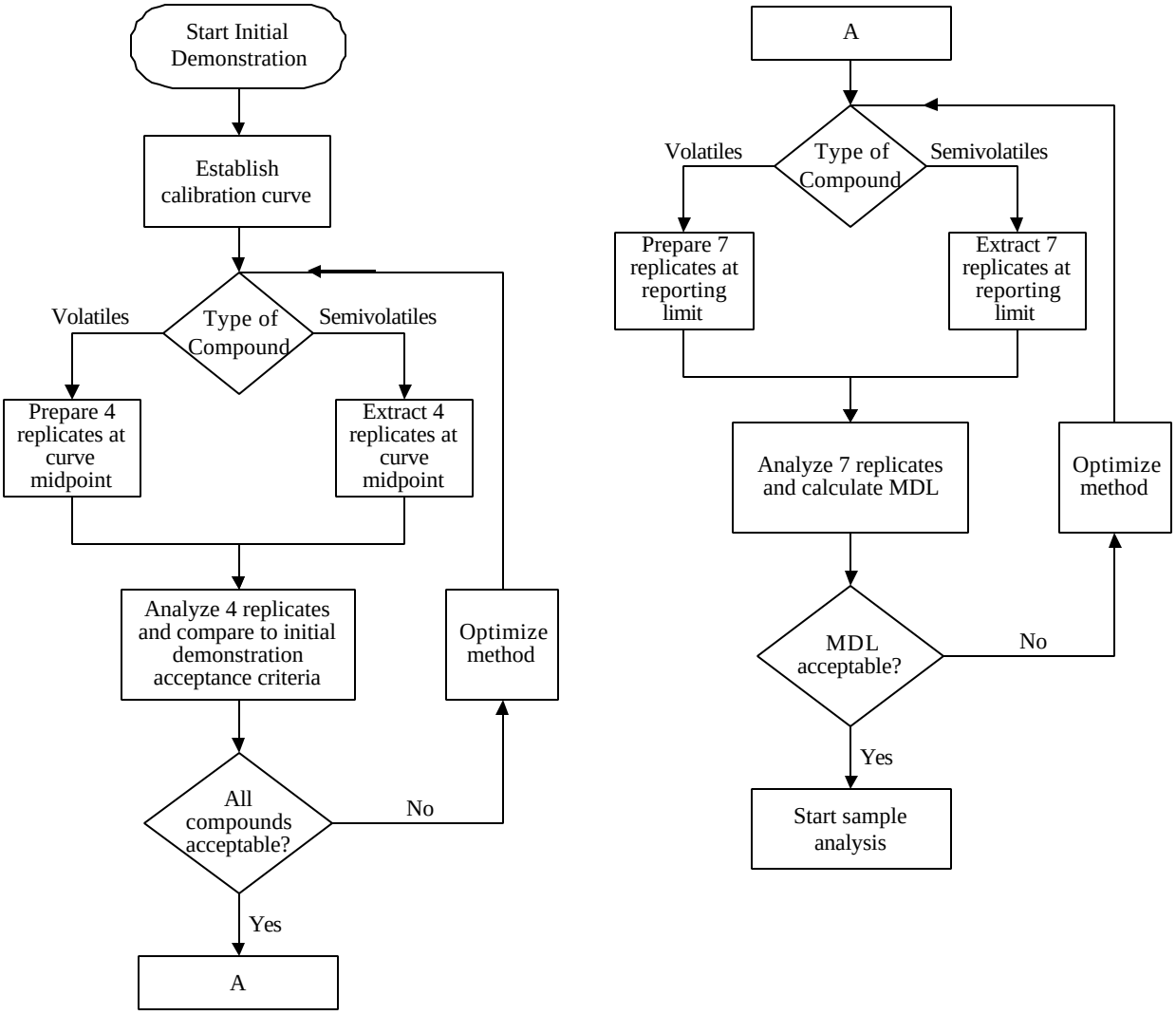
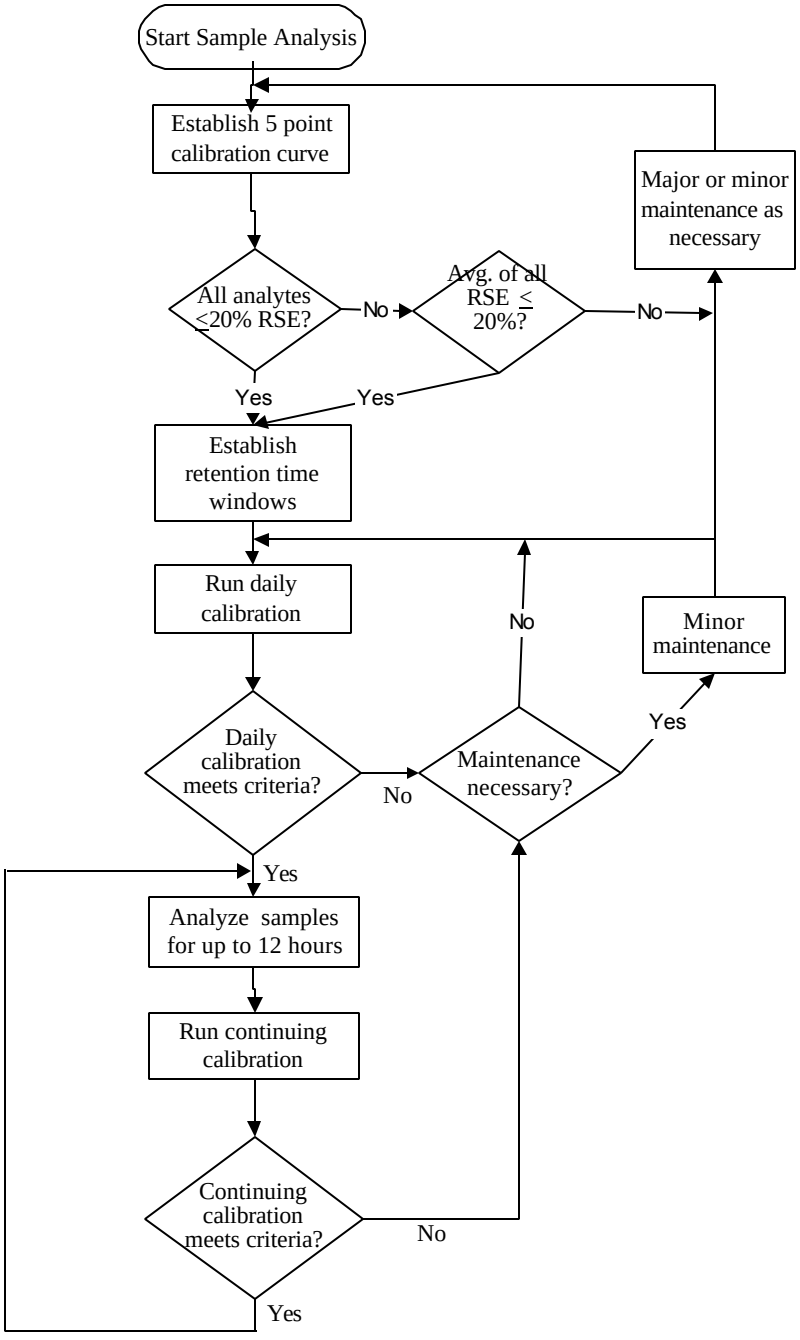
17.2. Flow Diagrams

17.2.1. Initial demonstration and MDL



17.2.2 Sample Analysis [1]



[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes sample preparation and extraction for the analysis of volatile organics by a Purge and Trap procedure following Method 8021B. All requirements of the 8000B section of this SOP must be met except when superseded by this Appendix. Refer to Table A1 for the individual analytes normally determined by these procedures. Routine reporting limits are listed in Table A1.

1.2. Compounds within the scope of this method have boiling points below 200°C and are insoluble or slightly soluble in water.

1.3. Water samples and soils samples with low levels of contamination may be analyzed directly by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in soil may be determined by the medium level methanol extraction procedure.

1.4. This method also describes the preparation of water-miscible liquids, non-water-miscible liquids, solids, wastes, and soils/sediments for analysis by the Purge and Trap procedure.

2. SUMMARY OF METHOD

2.1. An inert gas is bubbled through the sample at ambient temperature or at 40°C (40°C required for low-level soils), and the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and back-flushed with inert gas to desorb the components onto a gas chromatographic column. Analytes are detected using a photo-ionization detector, an electrolytic conductivity detector, or a combination of both.

2.2. For soil samples, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water. It is then analyzed by Purge and Trap GC following the normal water method.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4. INTERFERENCES

4.1. Refer to the main body of this SOP for general information on chromatographic interferences.

4.2. Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device must be avoided.

4.3. Samples can be contaminated by diffusion of volatile organics through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

4.4. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it must be followed by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the

system are subject to contamination. Therefore, frequent bake-out and purging of the system may be required.

4.5. A holding blank is kept in the sample refrigerator. This is analyzed and replaced every seven days. If the holding blank does not meet the method blank criteria, the source of contamination must be found and corrected. Evaluation of all samples analyzed in the seven-day period prior to the analysis of the contaminated holding blank is required. Refer to SOP NC-QA-020 for additional information on holding blanks.

5. SAFETY

5.1. Refer to the main body of this SOP for general safety requirements.

5.2. Benzene has been tentatively classified as known or suspected human or mammalian carcinogens.

5.3. GC VOA instruments use an ultraviolet (UV) light source, which must be shielded from view.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes – 5 µL, 10µL, 25µL, 100µL, 250µL, 500µL, and 1000µL. These must be equipped with a 20 gauge (0.006" ID) needle. These must be used to measure and dispense methanolic solutions and aqueous samples.

6.2. Purge and Trap Apparatus -- A device capable of extracting volatile compounds, trapping on a sorbent trap, and introducing onto a gas chromatograph.

6.3. Purge and Trap Autosampler -- In order to maintain high sample throughput, an autosampler is highly recommended.

6.4. Trap -- The trap used is dependent on the class of compound to be analyzed. Refer to Table A2 for suggested traps for specific tests.

6.5. Purge Vessels -- These are dependent on the purge and trap unit/autosampler used. Both disposable culture tubes (needle sparge units) and specially designed vessels with fritted bottoms may be used. Follow the manufacturer's suggestions for configuration.

6.6. Columns -- Refer to Table A2 for details of columns

6.7. Volumetric flasks, Class A -- 5 mL to 250 mL

6.8. pH paper: Range 0-14

6.9. Balance capable of weighing to 0.01g for samples

6.10. Chlorine test strips

6.11. Hach chlorine test pillows

6.12. Mechanical pipettes: 5 mL, 10 mL and 20 mL

7. REAGENTS AND SUPPLIES

7.1. Refer to the main body of this SOP for general requirements for reagents and supplies.

7.2. Organic-Free Water

7.2.1. Organic-free water is defined as water in which an interferent is not observed at the reporting limit of the compounds of interest. The laboratory method for generating organic free water is continuously sparging water with helium or nitrogen.

7.3. Methanol -- Purge and Trap Grade

7.4 Standards

7.4.1 Refer to Table A3 for details of surrogate, matrix spiking, and internal standards. Calibration standard levels are not specified, since they may depend on the sensitivity and linear range of specific detectors. However, the low-level standard must be equivalent to the reporting limits specified in Table A1.

7.4.2 Volatile standards are prepared by injecting a measured volume of the stock standard into a volumetric flask containing the appropriate volume of methanol. See the Standards Logbook for details on sample preparation.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection to analysis.

8.2. Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid. Unpreserved samples must be analyzed within seven days from sample collection.

8.3. Solid samples are field preserved with methanol for medium-level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore™ sample. (The 5g or 25g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium-level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes), then it will be necessary to use two additional 5g EnCore™ samplers or to use field preservation.

8.5. Sample collection for medium-level analysis using EnCore™ samplers.

8.5.1. Ship one 5g (or 25g) EnCore™ sampler per field sample position.

8.5.2. An additional bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 1 µL of a 50 µg/mL solution for a nominal 5g sample, 50 µL for a medium level prep sample, 50 µL for a waste prep, and 2.5 mL for a 25g soil sample.)

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples (MS/MSD). (Add 10 µL of a 10 µg/mL solution for a nominal 5g sample, 500 µL for a medium level prep sample, 500 µL for a waste prep, and 2.5 mL for a 25g soil sample.) The addition of spike introduces a slight error (0.4%), which can be neglected, into the calculations.

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8.5.6. Prepare a Laboratory Control Sample (LCS) for each batch by adding the correct amount of matrix spiking solution to clean methanol. (Add 10 µL of a 10 µg/mL solution for a nominal 5g sample, 500 µL for a medium level prep sample, 500 µL for a waste prep, and 2.5 mL for a 25g soil sample.)

8.5.7. Method blanks are prepared using 5 g of Ottawa sand, 5mL of reagent water, and a stir bar.

8.5.8. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.5.9. Allow to settle and store in a clean Teflon®-capped vial at 4 ± 2°C until analysis.

8.6. Sample collection for medium level analysis using field methanol preservation

8.6.1. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.2. Add the correct amount of surrogate spiking mixture. (Add 250 µL of 50 µg/mL solution for a nominal 25 g sample, 50 µL for a nominal 5g sample.)

8.6.3. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal sample.) The addition of spike introduces a slight error (0.4%), which can be neglected, into the calculations.

8.6.4. Prepare a Laboratory Control Sample (LCS) for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500 µL of 10 ug/mL spike to 5 mL methanol or 20 µL spike to 5 mL methanol).

8.6.5. For medium level Method Blanks, prepare 5 mL of methanol, 50uL of surrogate and 5 g of Ottawa sand in a 20 mL vial.

8.6.6. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6.7. Allow to settle and store in a clean Teflon®-capped vial at 4 +/-2°C until analysis.

8.7. Aqueous samples are stored in glass containers with Teflon®-lined septa at 4°C ± 2°C with minimum headspace. For volatile water samples, the method blank consists of reagent water

8.8. Medium-level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon®-lined caps and stored at 4°C ± 2°C. The extracts are stored with minimum headspace.

8.9. The maximum holding time is 14 days from sampling until the sample is analyzed. Maximum holding time for the EnCore™ sampler (before the sample is added to methanol) is 48 hours.

8.10. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise, it is replaced every 14 days.

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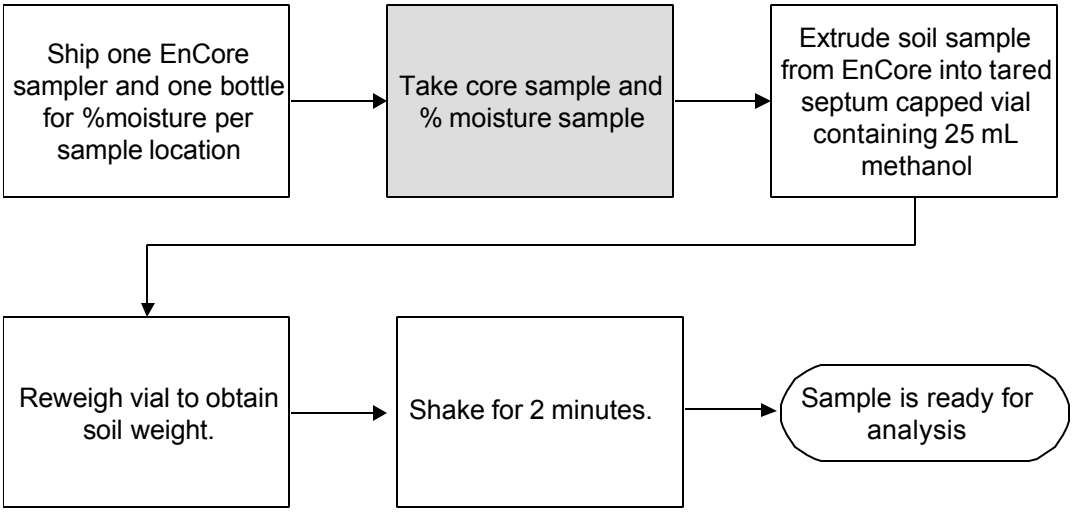
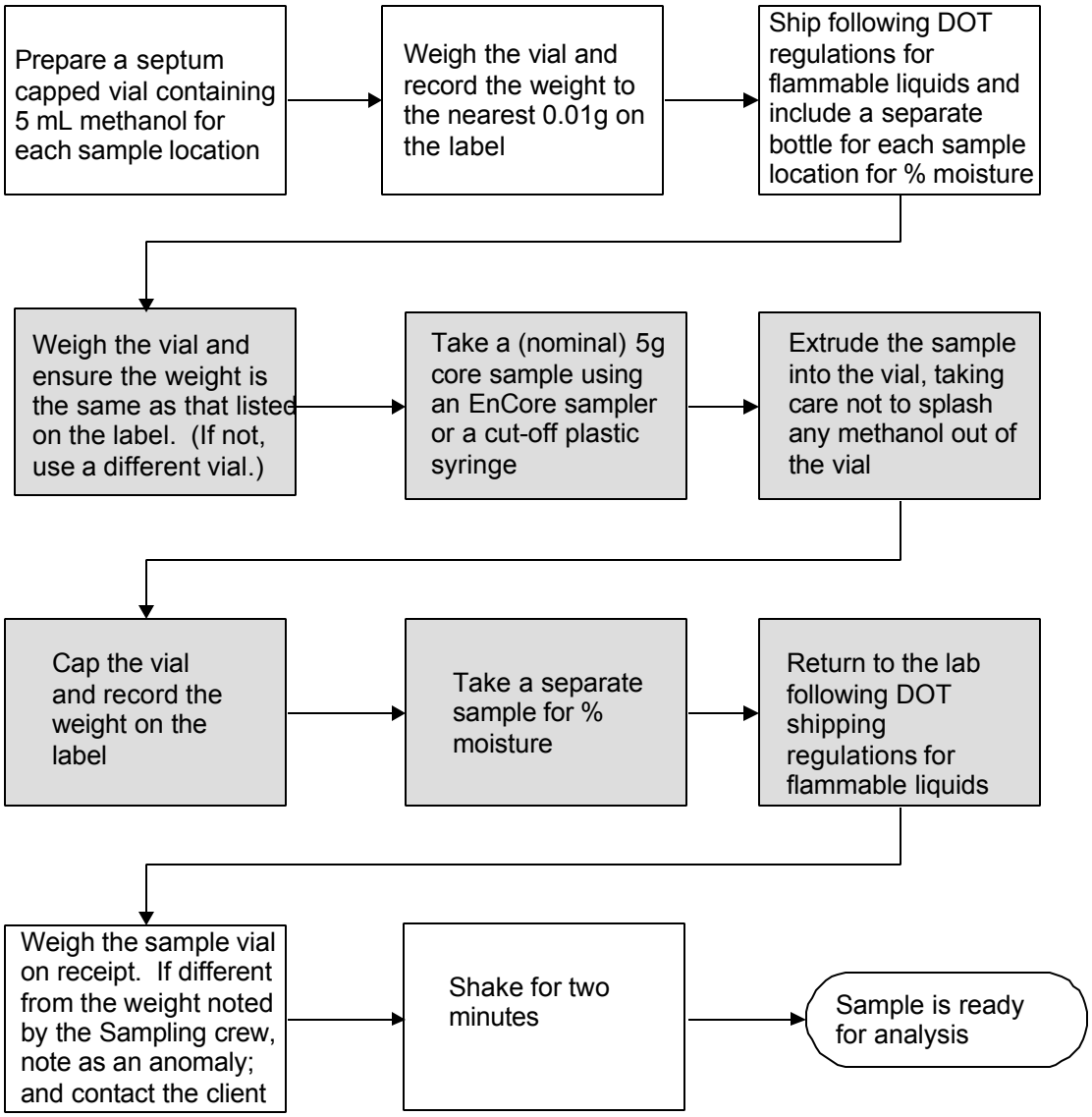
EnCore procedure when low level is not required (field steps in gray)



Field methanol extraction procedure (field steps in gray)



9. QUALITY CONTROL

9.1. Refer to the main body of this SOP for general quality control procedures, including batch definition, requirements for method blanks, Laboratory Control Sample (LCS), matrix spikes/ spike duplicate (MS/MSD), surrogates, and control limits.

9.2. Refer to Table A4 for the components and levels of the Laboratory Control Sample (LCS) and matrix spikes / spike duplicates (MS/MSD) mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the main body of this SOP for general calibration procedures.

10.2. Gas Chromatograph Operating Conditions

10.2.1. Various column configurations are possible. If dual column confirmation is necessary, the sample may be split using a Y splitter at the injector end to direct the sample to two columns and two detectors.

10.2.2. Refer to Tables A2, A3, and A4 for GC operating conditions. Additional operating instruction may be found in instrument manuals located in the laboratory.

10.3. Initial Calibration

10.3.1. Refer to Section 10 of the Method 8000B section of this SOP for details of initial calibration criteria.

10.3.2. Low-level soil samples must be purged at 40°C; therefore the calibration curve must also be purged at 40°C.

10.3.3. The low-level calibration must be at the reporting limit or below. The remaining standards encompass the working range of the detector. See Table A5 for the I-cal level and amounts.

10.3.4. Calibrate the instrument using the same volume that will be used during sample analysis.

10.4. Initial Calibration Verification (ICV)

10.4.1. An initial calibration verification (ICV) standard is analyzed immediately following the initial calibration. Acceptance criteria is ± 20%. If this is not met, a new initial calibration curve is analyzed.

10.5. Daily Calibration Verification

10.5.1. A mid-level calibration standard is used for the calibration verification. Acceptance criteria is ± 15%.

10.5.2. A calibration verification run is performed after every ten samples or 12 hours for this method.

10.5.3. Bracketing of samples with calibration verification runs is only necessary for external standard analysis. Analytes are detected using a photo-ionization detector are quantitated using the internal standard method.

11. PROCEDURE

11.1. Refer to the main body of this SOP for general procedural requirements.

11.2. Analytical Sequence

11.2.1. The analytical sequence starts with an initial calibration of at least five points or a 12-hour calibration that meets % difference criteria from an existing initial calibration.

11.3. Confirmation

11.3.1. The PID detector is sufficiently selective that second column confirmation is not always necessary. Requirements for second column confirmation must be decided in consultation with the client.

11.4. Aqueous and Soil Sample Analysis (Autosampler Purge and Trap units that sample directly from the VOA vial)

11.4.1. Check the pH of the sample in the VOA vial prior to analysis. Samples are also checked for residual chlorine at this time.

11.4.2. Units, which sample from the VOA vial, should be equipped with a module, which automatically adds surrogate and internal standard solution, as needed, to the sample prior to purging the sample.

11.4.3. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise, the internal and surrogate standards must be added to the vial.
Note: Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to be analyzed as soils.

11.4.4. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.5. Low-Level Solids Analysis using discrete autosamplers Bulk Solids

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.5.1. This procedure cannot be used for Ohio VAP samples. Refer to Section 1.4 for the appropriate procedure.

11.5.2. Do not discard any supernatant liquids. Mix the contents of the container

11.5.3. *Weigh out 5g (or other appropriate aliquot) of sample into a disposable culture tube or40mL vial. Record the weight to the nearest 0.1g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 0.5g. If the sample is contaminated with analytes such that a purge amount less than 0.5g is appropriate, use the medium-level method described in Section 8.6, Appendix A.*

11.5.4. *Place in autosampler.*

11.5.5. *Before placing on the autosample, add 5 mL of organic free water to each vial. Add surrogate/internal standard (and matrix spike solutions if required.) (See Table A3.) Add directly to the sample from Section 11.8.1.*

11.5.6. *The above steps must be performed rapidly and without interruption to avoid loss of volatile organics*

11.6. Methanol Extract Soils

11.6.1. Take an appropriate aliquot from the sample prep container. Add to 5mL of organic free water in a 40 mL VOA vial. Turn off the automatic surrogate function on the autosampler. Place the sample in the autosampler and analyze as for aqueous samples. If less than 1µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume not less than 1µL will be added to the water in the VOA vial.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the main body of this SOP. Refer to Tables A5 and A6 for details of the calibration curves.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced in the Method 8000B section of this SOP.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of the main body of this SOP

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the main body of this SOP

15.2. Waste streams produced by the method.

15.2.1. The following waste streams are produced when this method is carried out.

15.2.1.1. **Acidic material from the auto-sampler.** Waste stream must be collected and neutralized before discharge to a sewer system if the pH is less than 4.

15.2.1.2. **Methanol waste standards**. Methanol waste is discarded as a flammable liquid.

15.2.1.3. **All samples including purged and extracted soils and waters:** Samples are collected in boxes and removed from the lab to storage. The waste coordinator handles crushing the vials and proper disposal.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035 and 8021B.

16.2. SW846, *Test Methods for Evaluating Solid Waste*, Third Edition, Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035, Rev 0, December 1996.

16.3. Laboratory Holding Blanks, NC-QA-020

17. MISCELLANEOUS

17.1. Tables

TABLE A1

Standard Analyte List for Method 8021B				
Compound	**CAS No.**	**Reporting Limit, µg/L or µg/kg**		
		Aqueous	**Low Soil**	**Medium Soil**
1,2,4-Trimethylbenzene	95-63-6	1.0	1.0	50
1,3,5-Trimethylbenzene	108-67-8	1.0	1.0	50
Benzene	71-43-2	1.0	1.0	50
Ethylbenzene	100-41-4	1.0	1.0	50
Methyl tert-butyl ether (MTBE)	1634-04-4	1.0		
Toluene	108-88-3	1.0	1.0	50
Xylenes (total)	1330-20-7	1.0	1.0	50

TABLE A2

Recommended Conditions for Method Combined Aromatic and Halogenated Volatiles	
Parameter	**Recommended Conditions**
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	RTX - Volatiles 120m x 0.53mm ID df = 2.0um
Carrier gas	Hydrogen
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

TABLE A3

Surrogate and Internal Standard Concentrations			
Components	**Working Solution µg/mL**	**Spike amount µL**	**Final concentration µg/L (µg/kg)**
Fluorobenzene (IS)	50	1	10
a,a,a-Trifluorotoluene (SS)	50	1	10

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TABLE A4

Concentrations for Laboratory Control Sample (LCS) and MS/MSD Compounds			
Components	**Working Solution mg/mL**	**Spike A mL**	**Final Concentration mg/L (mg/kg)**
Benzene	10	10	20
Ethylbenzene	10		20
m&p Xylenes	20		40
o-Xylene	10		20
Xylenes, Total	30		60
MTBE	10		20
1,2,4-Trimethylbenzene	10		20
1,3,5-Trimethylbezene	10		20
Toluene	10		20

TABLE A5

Initial Calibration for Water Anlaysis		
Cal Level	**On Column Amount (ng)**	**Final Concentration (ug/L)**
1	5	1
2	10	2
3	50	10
4	100	20
5	200	40
6	400	80
7	800	160

TABLE A6

Soil or Water Heated Initial Calibration Curve		
Cal Level	**On Column Amount (ng)**	**Final Concentration (ug/kg)**
1	5	1
2	10	2
3	50	10
4	100	20
5	200	40
6	400	80
7	800	160

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1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8081A and 8081B is applied to the analysis of organochlorine pesticides by GC/ECD. This appendix applicable to extracts derived from any matrix which are prepared according to the appropriate sample extraction SOPs.

1.2. Table B1 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

2. SUMMARY OF METHOD

2.1. This method presents conditions for the analysis of prepared extracts of organochlorine pesticides. The pesticides are injected onto the column and separated and detected by electron capture detection. Quantitation is by external standard methods.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4. INTERFERENCES

4.1. Refer to the main body of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.

4.3. Sulfur will interfere and can be removed using procedures described in SOP NC-OP-025, Cleanup SOP.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Using hexane / acetone as the extraction solvent (rather than hexane / methylene chloride) will reduce the amount of interferences extracted.

5. SAFETY

5.1. Refer to main body of this SOP for general safety requirements.

5.2. The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, and the BHCs. Primary standards of these toxic compounds must be prepared in a hood.

5.3. All ^{63}Ni sources must be leak-tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.4. All ^{63}Ni sources must be inventoried every six months. If a detector is missing, the Director, EH&S must be immediately notified, and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

6.1. A ^{63}Ni electron capture detector is required.

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6.2. Refer to Table B2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.4 Autosampler vials, inserts, and caps

7. REAGENTS AND STANDARDS

7.1. Refer to the main body of this SOP for general requirements for reagents and supplies.

7.2. Refer to Table B3 for details of calibration standards. See the Standards Logbook for details on sample preparation.

7.3. Surrogate Standards

7.3.1. Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to Tables B5 and B6 for details of surrogate standards.

7.4. Column Degradation Evaluation Mix

7.4.1. A mid-level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This solution also contains the surrogates. Refer to Table B4 for details of the column degradation evaluation mix.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The holding time for pesticide extracts is 40 days from extraction to analysis. Samples must be refrigerated at $\leq 6^{o}C$.

9. QUALITY CONTROL

9.1. Refer to the main body of this SOP (Section 9) for general quality control procedures, including batch definition, requirements for method blanks, Laboratory Control Sample (LCS), matrix spike / spike duplicate (MS/MSD), surrogates, and control limits.

9.2. Refer to Table B5 for the components and levels of the Laboratory Control Sample (LCS) and matrix spikes / spike duplicates (MS/MSD) mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the main body of this SOP for general calibration requirements.

10.2. Refer to Table B2 for recommended details of GC operating conditions. The conditions listed must result in resolution of all analytes listed in Table B1 on both columns.

10.3. Column Degradation Evaluation

10.3.1. Before any calibration runs, either initial or 12 hour, the column evaluation mix must be injected before each initial or daily calibration. The degradation of DDT and endrin must be calculated (see Equations 9 and 10) and each shown to be less than 15% before calibration can proceed. This is only necessary if the target compound list includes DDT, Endrin, or any of their degradation products.

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10.3.2. If the breakdown of DDT and/or endrin exceeds the limits given above, corrective action must be taken. This action may include:

10.3.3. Replacement of the injection port liner or the glass wool.

10.3.4. Cutting off a portion of the injection end of a capillary column.

10.3.5. Replacing the GC column.

10.4. Initial Calibration

10.4.1. Refer to Section 10 of the Method 8000B section of this SOP for details of calibration procedures. The low-level calibration standard must be at, or below, the reporting limit.

10.4.2. Refer to Table B7 for the initial calibration analytical sequence.

10.4.3. The response for each single-peak analyte must be calculated by the procedures described in the main body of this SOP.

10.4.4. The surrogate calibration curve is calculated from the AB mix. If there are resolution problems, then the A and B mixes may be analyzed separately.

10.4.5. For multi-component pesticides:

10.4.5.1. A calibration with a minimum of five points is used for multi-component pesticides (typically toxaphene and technical chlordane). Two options are possible; the same quantitation option must be used for standards and samples. Refer to Section 12.3 for guidance on which option to use.

10.4.5.2. A full calibration for any of the multi-component analytes is analyzed.

10.5. Daily 12-hour Calibration Verification

10.5.1. The 12-hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration. A mid-level calibration standard is used for the 12-hour calibration. For Method 8081B, the CCV acceptance criteria is ± 20%

10.5.2. At a minimum, the 12-hour calibration includes analysis of the breakdown mix followed by mid-level standards of the AB mix. At a minimum, multi-component analytes are analyzed at the beginning of a sequence.

10.5.3. The retention time windows for any analytes included in the 12-hour calibration are updated.

10.6. Initial Calibration Verification (ICV)

10.6.1. An initial calibration verification (ICV) standard, from a second source, is analyzed immediately following the initial calibration. Acceptance criteria is ± 20%.If this is not met, a new initial calibration curve is analyzed.

10.7. Daily Continuing Calibration

10.7.1. A mid-level AB calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spike / spike duplicate (ms/msd), Laboratory Control Sample (LCS), and method blanks. If 12 hours elapse, analyze the 12-hour standard sequence instead. The continuing calibration standard need not include multi-component analytes. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

11. PROCEDURE

11.1. Refer to the main body of this SOP for general procedural requirements.

11.2. Suggested gas chromatographic conditions are given in Table B2.

11.3. Allow extracts to warm to ambient temperature before injection.

11.4. The suggested analytical sequence is given in Table B7.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the main body of this SOP for identification and quantitation of single component analytes.

12.2. Identification of Multi-Component Analytes

12.2.1. Retention time windows are also used for identification of multi-component analytes, but the "fingerprint" produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3. Quantitation of Multi-Component Analytes

12.3.1. Use 3-10 major peaks (or total area for quantitation) as described in Section 10.4.4, initial calibration of multi-component analytes.

12.3.2. If there are no interfering peaks within the envelope of the multi-component analyte, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.3.2.1. Multiple peak option

12.3.3. This option is particularly valuable if toxaphene is identified but interferences make quantitation based on total area difficult. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per multi-peak pesticide, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e., peaks which are significantly larger than would be expected from the rest of the pattern.)

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12.3.4. Chlordane may be quantitated either using the multiple peak option (Section 12.3.1, Appendix B), total area option (Section 12.3. 2., Appendix B), or by quantitation of the major components, α-chlordane, γ-chlordane and heptachlor.

12.4. Second column confirmation multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence. For Ohio VAP projects, both columns must meet criteria.

12.5. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

12.6. Calculation of Column Degradation/% Breakdown (%B)

Equation 9

$$DDT\ \%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

Where:

A_{DDD}, A_{DDE}, and A_{DDT} = the response of the peaks for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT in the column degradation evaluation mix.

Equation 10

$$Endrin\ \%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100$$

Where:

A_{EK}, A_{EA}, and A_E = the response of endrin ketone, endrin aldehyde, and endrin in the column degradation evaluation mix.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced in the main body of this SOP.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of the main body of this SOP.

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the main body of this SOP.

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8081A

16.2. SW846, Revision 2, February 2007, 8081B

17. MISCELLANEOUS

17.1. Modifications from Reference Method - None

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17.2. Tables

TABLE B1

Standard Analyte List and Reporting Limits for Methods 8081A and 8081B			
Compound	**Reporting Limit, µg/L or µg/kg**		
	Water	**Soil**	**Waste**
Aldrin	0.05	1.7	50
α-BHC	0.05	1.7	50
β-BHC	0.05	1.7	50
δ-BHC	0.05	1.7	50
γ-BHC (Lindane)	0.05	1.7	50
α-Chlordane	0.05	1.7	50
γ-Chlordane	0.05	1.7	50
Chlordane (technical)	0.5	17	500
4,4'-DDD	0.05	1.7	50
4,4'-DDE	0.05	1.7	50
4,4'-DDT	0.05	1.7	50
Dieldrin	0.05	1.7	50
Endosulfan I	0.05	1.7	50
Endosulfan II	0.05	1.7	50
Endosulfan Sulfate	0.05	1.7	50
Endrin	0.05	1.7	50
Endrin Aldehyde	0.05	1.7	50
Heptachlor	0.05	1.7	50
Heptachlor Epoxide	0.05	1.7	50
Methoxychlor	0.1	3.3	100
Toxaphene	2.0	67	2000
APPENDIX IX ADD-ONs			
Diallate	1.0	33	1000
Isodrin	0.1	3.3	100
Chlorobenzilate	0.25	16.7	500
Kepone[1]	1.0	33	1000
Hexachlorobenzene	0.05	33	1000

[1] Kepone is sometimes requested for analysis by method 8081A. However, kepone may produce peaks with broad tails that elute later than the standard by up to a minute (presumably due to hemi-acetal formation). As a result kepone analysis by 8081A is unreliable and not recommended. Analysis by method 8270C is a possible alternative.

Note: alpha chlordane, gamma chlordane, and endrin ketone are not required for some projects. The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.		Final Vol.
Ground water	1000 mL	5 mL	
Low-level soil	30g		10 mL
High-level soil / waste	1g		10 mL

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TABLE B2

Recommended Conditions for Methods 8081A and 8081B

Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	120°C for 1 min, 8.5°C/min to 285°C, , 6 min hold
Column 1	Rtx-CLPesticides 30m x 0.53mm id, 0.5µm
Column 2	Rtx CLPesticideII 30m,0.53mm id, 0.5um
Injection	1µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

TABLE B3

Calibration Levels ng/mL for Methods 8081A and 8081B

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
Individual Mix AB[1]						
Aldrin	5	10	25	50	100	200
g-BHC (Lindane)	5	10	25	50	100	200
Heptachlor	5	10	25	50	100	200
Methoxychlor	5	10	25	50	100	200
Dieldrin	5	10	25	50	100	200
Endosulfan I	5	10	25	50	100	200
Endosulfan II	5	10	25	50	100	200
4,4'-DDT	5	10	25	50	100	200
Endrin Aldehyde	5	10	25	50	100	200
Endrin Ketone	5	10	25	50	100	200
β-BHC	5	10	25	50	100	200
δ-BHC	5	10	25	50	100	200
α-BHC	5	10	25	50	100	200
4,4'-DDD	5	10	25	50	100	200
4,4'-DDE	5	10	25	50	100	200
Endosulfan Sulfate	5	10	25	50	100	200
Endrin	5	10	25	50	100	200
α-Chlordane[3]	5	10	25	50	100	200
γ-Chlordane[3]	5	10	25	50	100	200
Multi-component Standards						
Chlordane (Technical)	20	50	100	200	500	
Toxaphene	200	500	1000[5]	2000	5000	
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

[1] Standards may be split into an A and B mix if resolution of all compounds on both columns is not obtained.

[2] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.

[3] Compounds may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.

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TABLE B4	
Column Degradation Evaluation Mix ng/mL for Methods 8081A and 8081B	
Component	**Concentration**
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

TABLE B5			
Laboratory Control Sample (LCS)/Matrix Spike/Spike Duplicate (MS/MSD) and Surrogate Spike levels µg/L or µg/kg for Methods 8081A and 8081B			
	Aqueous	**Soil**	**Waste**
gamma BHC (Lindane)	1	33.3	1000
Aldrin	1	33.3	1000
Heptachlor	1	33.3	1000
Dieldrin	1	33.3	1000
Endrin	1	33.3	1000
Alpha BHC	1	33.3	1000
Beta BHC	1	33.3	1000
Delta BHC	1	33.3	1000
Gamma BHC	1	33.3	1000
4,4'DDD	1	33.3	1000
4,4'DDE	1	33.3	1000
4,4'DDT	1	33.3	1000
Endosulfan I	1	33.3	1000
Endosulfan II	1	33.3	1000
Endosulfan Sulfate	1	33.3	1000
Heptachlor Expoxide	1	33.3	1000
Methoxychlor	1	33.3	1000
Endrin Ketone	1	33.3	1000
Endrin Aldehyde	1	33.3	1000
Alpha-chlordane	1	33.3	1000
Gamma-chlordane	1	33.3	1000
Tetrachloro-m-xylene (Surrogate)	0.20	6.7	200
Decachlorobiphenyl (Surrogate)	0.20	6.7	200

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TABLE B6

Laboratory Control Sample (LCS)/Matrix Spike/Spike Duplicate (MS/MSD) and Surrogate Spike levels for TCLP µg/L or µg/kg for Methods 8081A and 8081B	
	Aqueous
Heptachlor	2
Heptachlor epoxide	2
Lindane	2
Endrin	2
Methoxychlor	4

TABLE B7
Suggested Analytical Sequence for Methods 8081A and 8081B

Initial Calibration

Solvent blank (optional)
Primer if needed
Breakdown Mix
Individual mix AB — All levels
ICV
Technical Chlordane — Level 3[1]
Toxaphene — Level 3[1]
Up to 20 samples unless 12 hours comes first)
Solvent blank (optional)
Individual mix AB — Mid level (Continuing calibration)
Samples
After 12 hours:
Breakdown mix
Individual mix AB
Any other single component analytes
Any multi-component analytes

[1] A curve with a minimum of five points for any of the multi-component analytes may be included.

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

Note: The initial primer is used if the instrument has been idle for a period of time.

12 -Hour Calibration

At least every 12 hours, counting from the start of the initial calibration or from the start of the last daily calibration, the retention time windows must be updated using the Individual mix AB and a PEM must be analyzed if the analysis is to continue.

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1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8082 or 8082A is applied to the analysis of polychlorinated biphenyls (PCB) by GC/ECD. This appendix is applicable to extracts derived from any matrix which are prepared according to the appropriate sample extraction SOPs. PCBs are determined and quantitated as Aroclor mixes.

1.2. Tables C1 and C5 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

Note: SW-846 method 8082 and 8082A provides incomplete guidance for determination of individual PCB congeners. This SOP does not include directions for congener specific analysis.

2. SUMMARY OF METHOD

2.1. This method presents conditions for the analysis of prepared extracts of PCBs. The PCBs are injected onto the column and separated and detected by electron capture detection. Quantitation is by external standard methods.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4. INTERFERENCES

4.1. Refer to the main body of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.

4.3. Sulfur will interfere and can be removed using procedures described in SOP NC-OP-025.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts. These cleanup procedures are included in SOP NC-OP-025.

5. SAFETY

5.1. Refer to the main body of this SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

5.3. All ^{63}Ni sources must be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.4. All ^{63}Ni sources must be inventoried every six months. If a detector is missing, the EH&S Director must be immediately notified and a letter sent to the NRC or local state agency.

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6. EQUIPMENT AND SUPPLIES

6.1. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table C2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.4 Autosampler vials, inserts, and caps

7. REAGENTS AND STANDARDS

7.1. Refer to the main body of this SOP for general requirements for reagents and supplies.

7.2. Refer to Table C3 for details of calibration standards. See the Standards Logbook for details on sample preparation.

7.3. Surrogate Standards

7.3.1. Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to Table C4 for details of surrogate standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The holding time for PCB extracts is 40 days from extraction to analysis. Samples must be refrigerated at $\leq 6^{o}$C.

9. QUALITY CONTROL

9.1. Refer to main body of this SOP for general quality control procedures, including batch definition, requirements for method blanks, Laboratory Control Sample (LCS), matrix spikes / spike duplicate (MS/MSD), surrogates, and control limits.

9.2 Refer to Table C4 for the components and levels of the Laboratory Control Sample (LCS) and matrix spikes / spike duplicates (MS/MSD) mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the main body of this SOP for general calibration requirements.

10.2 Update IV recommends analysis of DDT and analogs DDD and DDE prior to calibration to assure there is not interference with major 1254 peaks.

10.2. Initial Calibration

10.2.1. Refer to Table C6 for the initial calibration analytical sequence.

10.2.2. The response for each Aroclor must be calculated by the procedures described in the main body of this SOP with the following modifications.

10.2.3. A minimum five-point calibration of all Aroclors is generated. The average response factor is used to quantitate Aroclors. The low-level standard must be at or below the reporting limit. The other standards define the working range of the detector.

10.2.4. The high and low standards for the initial calibration of 1016 / 1260 define the acceptable quantitation range for the other Aroclors. If any Aroclor is determined above this concentration, the extract must be diluted and re-analyzed.

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Note: For Ohio VAP, Aroclor 1268 may be analyzed. In order to meet project-specific reporting limits, a lower concentration standard may be added to the calibration curve.

10.2.5. If the analyst knows that a specific Aroclor is of interest for a particular project, that Aroclor may be used for the calibration rather than the 1016 / 1260 mix.

10.2.6. The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

10.2.7. The following is used for the quantitation of all Aroclors. The same quantitation option must be used for standards and samples.

10.2.7.1. Multiple peak option.

10.2.7.2. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks.

10.3. Daily 12-Hour Continuing Calibration

10.3.1. The 12-hour calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration. For Method 8082A, the CCV acceptance criteria is ± 20%.

10.3.2. At a minimum, the 12-hour calibration includes analysis of the Aroclor 1260 / 1016 mix.

10.3.3. Other Aroclors are included in the daily calibration check.

10.3.4. The retention time windows for any analytes included in the daily calibration and CCVs are updated.

10.3.5. For this method, samples must be bracketed with successful calibration verification runs.

10.4. Initial Calibration Verification (ICV)

10.4.1. An initial calibration verification (ICV) standard, from a second source, is analyzed immediately following the initial calibration. Acceptance criteria is ± 20%. If this is not met, a new initial calibration curve is analyzed.

10.5. Daily Calibration Verification Standards

10.5.1. The Aroclor 1260/1016 calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples, including matrix spikes, Laboratory Control Sample (LCS), and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.)

10.5.2. A mid-level standard is used for the calibration verification.

11. PROCEDURE

11.1. Refer to the main body of this SOP for general procedural requirements.

11.2. Suggested gas chromatographic conditions are given in Table C2.

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11.3. Allow extracts to warm to ambient temperature before injection.

11.4. The suggested analytical sequence is given in Table C6.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Identification of Aroclors

12.1.1. Retention time windows are used for identification of Aroclors, but the "fingerprint" produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.1.2. A clearly identifiable Aroclor pattern serves as confirmation of single column GC analysis.

12.2. Quantitation of Aroclors

12.2.1. Use 3-10 major peaks or total area for quantitation

12.2.2. If the analyst believes that a combination of Aroclor 1254 and 1260 or a combination of 1242, 1248 and 1232 is present, then only the predominant Aroclor is quantitated and reported; but the suspicion of multiple Aroclors is discussed in the narrative. If well-separated Aroclor patterns are present, then multiple Aroclors may be quantitated and reported.

12.3. Second column confirmation of Aroclors will only be performed when requested by the client or regulatory program. The appearance of the multiple peaks in the sample usually serves as a confirmation of Aroclor presence. For Ohio VAP projects, both columns must meet criteria.

12.4. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits, or if one is <10%.

Note: For Ohio VAP samples and DoD projects, all surrogates must meet acceptance limits,unless the surrogate is biased high and the sample is ND.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced in the main body of this SOP.

13.2. Method detection limits (MDL) are determined for all Aroclors.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of main body of this SOP.

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the main body of this SOP

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8082

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16.2. SW846, Update IV, Revision 1, February 2007, Method 8082A

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Method 8082 and 8082A includes limited direction for congener specific quantitation. This is outside the scope of this SOP.

17.2. Tables

TABLE C1
Standard Analyte list and Reporting Limits for Methods 8082 and 8082A

	Reporting Limit, µg/L or µg/kg		
Compound	**Water**	**Soil**	**Waste**
Aroclor-1016	1.0	33	1000
Aroclor-1221	1.0	33	1000
Aroclor-1232	1.0	33	1000
Aroclor 1242	1.0	33	1000
Aroclor-1248	1.0	33	1000
Aroclor-1254	1.0	33	1000
Aroclor-1260	1.0	33	1000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	5 mL
Low-level Soil	30g	10 mL
High-level soil / waste	1g	10 mL

TABLE C2
Instrumental Conditions for Methods 8082 and 8082A

Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	70°C for 0.5min, 30°C/min to 190°C, 2.5°C/min to 225, 18°C/min to 280°C, 3 min hold
Column 1	DB-5 or Rtx-5 30m x 0.32mm id, 0.5µm, or CLPesticide I, 30m, 0.53mm id, 0.5um
Column 2	DB 1701 or Rtx 1701 30m x 0.32 mm id, 0.25µm, or CLPesticide II, 30m, 0.53 mm id, 0.5um
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	1µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

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TABLE C3 Calibration Levels ug/mL for Methods 8082 and 8082A						
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6[1]**
Aroclor 1016/1260	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1242	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1221 +1254	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1232	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1248	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1262	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1268	0.05	0.1	0.2	0.5	1.0	2.0
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	0.0025	0.005	0.01	0.025	0.05	0.1
Decachlorobiphenyl	0.0025	0.005	0.01	0.025	0.05	0.1

[1] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.

TABLE C4 Laboratory Control Sample (LCS) / Matrix Spike/Spike Duplicate (MS/MSD) and Surrogate Spike levels for Aroclor analysis mg/L or mg/kg for Methods 8082 and 8082A			
	Aqueous	Soil	Waste
Aroclor 1016/1260	10	333	10,000
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

TABLE C5 Michigan Analyte List and Reporting Limits[1] for Methods 8082 and 8082A			
	Reporting Limit		
Compound	**water (mg/L)**	**soil (mg/Kg)**	
Aroclor-1016	0.2	330	
Aroclor-1221	0.2	330	
Aroclor-1232	0.4	330	
Aroclor 1242	0.2	330	
Aroclor-1248	0.2	330	
Aroclor-1254	0.2	330	
Aroclor-1260	0.2	330	

[1] Reporting Limits are only for samples performed under the Michigan program.

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Table C6
Suggested Analytical Sequence for Methods 8082 and 8082A

Initial Calibration

Injection #		
1	Solvent blank (optional)	
2	Aroclor 1016/1260	Level 1
3	Aroclor 1016/1260	Level 2
4	Aroclor 1016/1260	Level 3
5	Aroclor 1016/1260	Level 4
6	Aroclor 1016/1260	Level 5
7	Aroclor 1232	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
8	Aroclor 1242	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
9	Aroclor 1248	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
10	Aroclor 1221/1254	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
11	Aroclor 1268 or 1262	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
12	ICV	
13-32	Sample 1-20 (or as many samples as can be analyzed in 12 hours	
33	Aroclor 1016/1260	Level 3

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

12-hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Aroclor 1260 / 1016 mix. Mid-level standards of any other Aroclors expected to be present in the samples are also injected.

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1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8151A or EPA Method 615 is applied to the analysis of chlorinated phenoxy acid herbicides in extracts prepared by SOP NC-OP-031.

1.2. Table D1 lists compounds, which are routinely analyzed by this method and give the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

2. SUMMARY OF METHOD

2.1. This method presents conditions for the analysis of prepared extracts of phenoxy acid herbicides by gas chromatography. The herbicides, as their methyl esters, are injected onto the column, separated, and detected by electron capture detectors. Quantitation is by external standard methods.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4. INTERFERENCES

4.1. Refer to the main body of this SOP for general information regarding chromatographic interferences.

4.2. Chlorinated acids and phenols cause the most direct interference with this method.

4.3. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples.

5. SAFETY

5.1. Refer to the main body of this SOP for general safety requirements.

5.2. All ^{63}Ni sources must be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.3. All ^{63}Ni sources must be inventoried every six months. If a detector is missing, the EH&S Director must be immediately notified and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

6.1. A Ni_{63} electron capture detector is required.

6.2. Refer to Table D2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.4 Autosampler vials, inserts, and caps

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7. REAGENTS AND STANDARDS

7.1. Refer to the main body of this SOP for general information on reagents and standards.

7.2. Refer to Table D4 for details of calibration standards. See the Standards Logbook for details on sample preparation.

7.3. Surrogate Standards

7.3.1. DCAA is the surrogate standard. Refer to Table D4 for details of surrogate standards.

8. SAMPLE PREPARATION, PRESERVATION, AND STORAGE

8.1. The holding time for herbicide extracts is 40 days from extraction to analysis. Samples must be refrigerated at $\leq 6^{o}C$.

9. QUALITY CONTROL

9.1. Refer to the main body of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes / spike duplicates (MS/MSD).

9.2. Refer to Table D3 for the components and levels of the Laboratory Control Sample (LCS) and matrix spikes / spike duplicates (MS/MSD) mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the main body of this SOP for general calibration requirements.

10.2. Refer to Table D2 for recommended instrument operating conditions.

10.3. Calibration standards are prepared from purchased standards in the methyl ester form.

10.4. The low-level standard must be at or below the laboratory reporting limit.

10.5. The response for each analyte must be calculated by the procedures described in the main body of this SOP

10.6. Initial Calibration Verification (ICV)

10.6.1. An initial calibration verification (ICV) standard, from a second source, is analyzed immediately following the initial calibration. Acceptance criteria is ± 20%. If this is not met, a new initial calibration curve is analyzed.

10.7 For method 615, the Ical and the CCV must both pass by 10%.

10.8 Daily Continuing Calibration

10.8.1 A mid-level calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spike / spike duplicate (ms/msd), Laboratory Control Sample (LCS), and method blanks. If 12 hours elapse, analyze the 12-hour standard sequence instead. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

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11. PROCEDURE

11.1. Refer to the main body of this SOP for procedural requirements.

11.2. Extraction

11.2.1. The extraction procedure is described in SOP NC-OP-032.

11.3. Analytical Sequence

11.3.1. The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.3.2. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence.

11.3.3. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.3.4. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the main body of this SOP for identification and quantitation of single component analytes.

12.2. The herbicides are analyzed as their methyl esters, but reported as the free acid. For this reason, it is necessary to correct the results for the molecular weight of the ester versus the free acid. This is achieved through the concentrations of the calibration standards. For example the 20µg/L calibration standard for 2,4-D contains 21.3 µg/L of the methyl ester. No further correction is necessary.

12.3. A routine 10X dilution occurs on final extracts for all samples. Due to a LIMSlimitation, the dilution factor field in LIMS cannot be used when a dilution is routine, because the dilution factor is automatically applied to all reference values creating reporting problems. For the herbicide analysis, the extract volume will be 10mL and an aliquot at 10X dilution will be analyzed. The final extract volume recorded on the laboratory bench sheet will be recorded as 100mL to avoid using the dilution factor field in LIMS.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced in the main body of this SOP.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of the main body of this SOP.

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the main body of this SOP.

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16. REFERENCES

16.1. Method 8151A, SW-846, Update III, December 1996

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. None

17.2. TABLES

TABLE D1
Standard Analyte List for Method 8151A

Compound	CAS Number	Reporting Limit, µg/L or µg/kg		
		Aqueous	Soil	Waste
2,4-D	94-75-7	4	80	4000
2,4-DB	94-82-6	4	80	4000
2,4,5-TP (Silvex)	93-72-1	1	20	1000
2,4,5-T	93-76-5	1	20	1000
Dalapon	75-99-0	2	40	2000
Dicamba	1918-00-9	2	40	2000
Dichloroprop	120-36-5	4	80	4000
Dinoseb	88-85-7	0.6	12	600
MCPA	94-74-6	400	8000	400,000
MCPP	93-65-2	400	8000	400,000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.	Dilution Factor
Ground water	1000 mL 10 mL	10	
Low-level Soil without GPC	50g	10 mL	10
High-level soil / waste	1g	10 mL	10

Specific reporting limits are highly matrix dependent. The reporting limits listed above are provided for guidance only and may not always be achievable. For special projects, the extracts may be analyzed without any dilution, resulting in reporting limits 20 times lower than those in Table D1.

TABLE D2
Instrumental Conditions for Method 8151A

PARAMETER	Recommended conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	80,2/30/170,0/1/180,1
Column 1	CLPesticideI, 30m, 0.53 mm id, 0.5 um
Column 2	CLPesticide II, 30m, 0.53 mm id, 0.5um
Injection	1-2µL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

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Recommended conditions must result in resolution of all analytes listed in Table D1.

The reporting limits listed in Table D1 will be achieved with these calibration levels and a 20-fold dilution of the sample extract. Lower reporting limits can be achieved with lesser dilutions of the sample extract.

TABLE D3

Laboratory Control Sample (LCS)/Matrix Spike/Spike Duplicate (MS/MSD) and Surrogate Spike levels mg/L or mg/kg[1] for Method 8151A

	Aqueous	Soil	Waste
2,4-D	40	400	20000
Silvex	10	100	5000
2,4,5-T	10	100	5000
2,4-DB	40	400	20000
Dalapon	20	200	10000
DCAA (surrogate)	40	400	20000
Dicamba	20	200	10000
MCPP	4000	40000	200000
MCPA	4000	40000	200000
Dichloroprop	40	400	2000
Pentachlorophenol	5	50	2500
Dinoseb	6	60	300

[1] Laboratory Control Sample (LCS), MS and SS spikes are as the free acid.
Note: Dinoseb is a poor performing analyte. No corrective action will be taken if recovery is outside acceptance limits.

TABLE D4

Calibration Levels for Methods 8151A and 615 (ng amount)

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
2,4 D	0.02	0.04	**0.08**	0.16	0.32	0.64
2,4 DB	0.02	0.04	**0.08**	0.16	0.32	0.64
2,4,5 T	0.005	0.01	**0.02**	0.04	0.08	0.16
2,4,5 TP (Silvex)	0.005	0.01	**0.02**	0.04	0.08	0.16
Dalapon	0.01	0.02	**0.04**	0.08	0.16	0.32
DCAA (surr)	0.02	0.04	**0.08**	0.16	0.32	0.64
Dicamba	0.01	0.02	**0.04**	0.08	0.16	0.32
Dichloroprop	0.02	0.04	**0.08**	0.16	0.32	0.64
Dinoseb	0.003	0.006	**0.012**	0.024	0.048	0.096
MCPA	2	4	**8**	16	32	64
MCPP	2	4	**8**	16	32	64
Pentachlorophenol	0.0005	0.005	**0.01**	0.02	0.04	0.08

Bold levels indicate CCV

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1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8015B or 8015C is applied to the determination of the concentration and **tentative** identification of extractable petroleum (diesel range) hydrocarbon mixes in waters, wastewaters, soils, and sludges. This Appendix is applicable to extracts derived from any matrix which are prepared according to the appropriate sample extraction SOPs.

1.2. Table E2 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed. Other analytes may be analyzed by this method if the quality control criteria in Section 9 and the initial demonstration of method performance in Section 13 are met. Reporting limits are also listed in Table E2. The laboratory carbon range for Ohio VAP and BUSTR projects is Middle Distillates (C10-C20) and Heavy Distillates (C20-C34).

2. SUMMARY OF METHOD

2.1 This method presents conditions for the analysis of total petroleum hydrocarbons by gas chromatography. The total petroleum hydrocarbon samples are injected into the column, separated, and detected by flame ionization detectors (FID). Quantitation is by external standard methods.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4. INTERFERENCES

4.1. Refer to the main body of this SOP for general information regarding chromatographic interferences.

4.2. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples.

5. SAFETY

5.1. Refer to the main body of this SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. A flame ionization detector (FID) is required.

6.2. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.3. Autosampler vials, inserts, and caps

7. REAGENTS AND STANDARDS

7.1. Refer to the main body of this SOP.

7.2. The petroleum hydrocarbons (Diesel Fuel) are purchased from a chemical supplier when available. When no chemical supplier is available, the fuels are purchased from public sources. See the Standards Logbook for details on sample preparation.

7.3. Refer to Table E3 for details of calibration standards.

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7.4. Surrogate Standards

7.4.1. Nonane (C9) is the surrogate standard.

8. SAMPLE PREPARATION, PRESERVATION, AND STORAGE

8.1. The holding time for semivolatile extracts is 40 days from extraction to analysis. Samples must be refrigerated at <6°C.

9. QUALITY CONTROL

9.1. Refer to the main body of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes / spike duplicates (MS/MSD).

9.2. Matrix spikes / spike duplicates (MS/MSD) recoveries are calculated from a diesel calibration.

9.3. Surrogates

9.3.1. Because of the nature of the TPH analysis, - certain petroleum mixtures can override the C9 (Nonane) surrogate.

Note: Ohio VAP rules require reanalysis when surrogate recoveries are outside of control limits. Re-extraction is required if surrogates are outside of control limits.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the main body of this SOP for general calibration requirements. The low-level calibration standard must be at, or below, the reporting limit.

10.2. Refer to Table E1 for recommended instrument conditions.

10.3. Initial Calibration

10.3.1. Prior to the initial calibration, a marker solution consisting of alkanes from C10-C44 is analyzed, If additional carbon ranges are needed, a separate solution with alkanes from C10-C50 can be analyzed with a modified instrument program. The marker solution establishes the retention time window.

10.3.2. Analyze a diesel calibration calibration, using a minimum of five points, referring to the recommended instrument conditions. The calibration concentrations are 100, 200, 500, 1000, and 2000 ng/uL. A 5000ng/uL standard may be analyzed if needed. The retention time window of C10-C32 must be used for the Diesel calibration. The low-level standard must be at or below the reporting limit. The other standards define the working range of the detector.

Note: For special projects, retention time windows can be customized to reflect additional carbon ranges. The additional carbon ranges are quantitated against the diesel calibration.

10.4. Initial Calibration Verification (ICV)

10.4.1. An initial calibration verification (ICV) standard, from a second source, is analyzed immediately following the initial calibration. Acceptance criteria is ± 20%. If this is not met, a new initial calibration curve is analyzed.

10.5. Daily Continuing Calibration

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10.5.1. Refer to Section 10 of the Method 8000B section of this SOP for general calibration requirements.

10.5.2. A mid-range standard of diesel, C10-20, and C20-34 is used, as appropriate, for the CCV. The acceptance criteria is ±15%. This marker solution must be analyzed at the beginning of each sequence. For Method 8015C, the CCV acceptance criteria is ± 20%

11. PROCEDURE

11.1. Refer to the main body of this SOP for procedural requirements.

11.2. A suggested analytical sequence is given in Table E4.

11.3. Petroleum Hydrocarbon Identification and/or Fingerprinting

11.3.1. To identify the type of petroleum hydrocarbon, compare the chromatographic peak pattern to the patterns of known petroleum hydrocarbons analyzed under identical chromatographic conditions. Samples are quantified against diesel, but fingerprinting may be done when client requested.

11.3.2. Positive matching may not be possible, even using site-specific hydrocarbons. Degradation of the pattern can occur during environmental exposure of the fuel. See Table E2 for possible fingerprints.

11.3.3. Samples are quantified against the initial calibration of diesel or DRO on a single column.

11.3.4. The total height or area of the hydrocarbon is determined in the same manner used for the hydrocarbon standard.

11.3.5. If the amount of sample injected into the GC exceeds the working range of the calibration curve, an appropriate dilution is performed before reanalysis.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the main body of this SOP for identification and quantitation of single component analytes.

12.2. Surrogate recovery results are calculated and reported for Nonane (C-9). The surrogate must be within QC criteria. Corrective action is only necessary if Nonane (C-9) is outside of acceptance limits, unless the surrogate is high and the sample is ND.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced in the main body of this SOP.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of the main body of this SOP.

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the main body of this SOP.

16. REFERENCES

16.1. SW846, Method 8015B, Nonhalogenated Organics Using GC/FID, Test Methods for Evaluating Solid Waste, Third Edition, USEPA

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16.2. SW846, Method 8015C, Nonhalogenated Organics by Gas Chromatography, Test Methods for Evaluating Solid Waste, Revision 3, February 2007.

TABLE E1
Recommended Instrument Conditions for Methods 8015B and 8015C

Parameter	Recommended Conditions
Column	RTX-5
Initial Temperature	40°C
Initial Hold Time	4 minutes
Temperature Program	10°C/minute
Final Temperature	280°C
Final Hold Time	10 minutes
Injection	1µL
Carrier Gas	Hydrogen carrier gas - flow rate 5 - 6 mL/min
Detector Gas Mixture	Air hydrogen mixture in a 10:1 ratio, air 80 - 120 mL/min, hydrogen 8 -12 mL/min

TABLE E2
Reporting Limits for TPH Analysis

Analyte	Reporting Limits		
	Water (mg/L)	Solids (mg/kg)	Waste Dilution (mg/kg)
TPH (as Diesel) or DRO	500	16.7	200
C10-C20 (OVAP & BUSTR -Middle Distillates)	500	16.7	
C20-C34 (OVAP & BUSTR – Heavy Distillates)	500	16.7	
Fingerprint Compounds [1]			
Mineral Spirits	Kerosene	Motor Oil	
Hydraulic Oil	Jet Fuel	Stoddard Solvent	

[1] This list represents most of the common petroleum hydrocarbons. The list may be expanded to include other petroleum hydrocarbons.

TABLE E3					
Calibration Levels for Methods 8015B and 8015C (ng/L)					
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**
TPH (as Diesel)	100	200	500	1000	2000

TABLE E4
Suggested Analytical Sequence for Method 8015B and 8015C

Initial Calibration

Solvent blank (optional)	
Primer if needed	
Marker Solution	
Diesel Standard	All levels
ICV	
Up to 20 samples unless 12 hours comes first)	
Mid level Diesel Standard (Continuing calibration)	

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

Note: The initial primer is used if the instrument has been idle for a period of time.

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Title: CONTINUOUS LIQUID / LIQUID EXTRACTION OF ORGANIC COMPOUNDS FROM WATERS BASED ON METHOD SW846 3520C AND 600 SERIES AND WASTE DILUTION BASED ON METHOD 3580A

[Methods: SW846 3520C and 600 Series and 3580A]

Approvals (Signature/Date):

Technology Specialist	02/16/11 Date	Health & Safety Coordinator	02/16/11 Date
Quality Assurance Manager	03/02/11 Date	Laboratory Director	02/27/11 Date
Technical Director	03/03/11 Date		

This SOP was previously identified as SOP No. NC-OP-032, Rev 1, dated 01/07/09, which is now split into four individual SOPs

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1. SCOPE AND APPLICATION

1.1 This SOP describes procedures for preparation (extraction) of semivolatile organic analytes in aqueous, and TCLP leachate, matrices for analysis by Gas Chromatography (GC) and Gas Chromatography / Mass Spectrometry (GC/MS) using Continuous Liquid/Liquid Extraction. The procedures are based on SW846 and 600 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1.1. Extraction procedures for the following determinative methods are covered: 8081A, 8081B, 8082, 8082A, 8270C, 8270D, 8015B, 8015C, 608, and 625.

1.1.2. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

2. SUMMARY OF METHOD

2.1. Continuous Liquid/Liquid Extraction

A measured volume of sample (typically 1 liter) is placed into a continuous liquid/liquid extractor, adjusted if necessary, to a specific pH, and extracted with the appropriate solvent for 18-24 hours.

2.2. Concentration

Procedures are presented for drying and concentration of the extract to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms and acronyms used in this SOP may be found in the glossary of the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

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5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded, other gloves must be cleaned immediately.

5.3. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds must be prepared in the hood.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3 2 mg/m^3-Ceiling	This material will cause burns if comes into contact with the skin or eyes. Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.

Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain, and severe tissue burns. Can cause blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride must be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of methylene chloride is spilled, evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints, which can become stuck. Technicians must use Kevlar or other cut/puncture-resistant gloves when separating stuck joints.

5.9. 3520 Extraction Continuous Liquid/Liquid

5.9.1. All personnel are to ensure liquid-liquid area is clear of unnecessary items. Heating mantles used with liquid-liquid extractions generate temperatures that could ignite some materials that come in contact with the heating mantles.

5.9.2. Ensure all solvents are away from liquid-liquid extractor. Increased temperatures near solvents can cause the pressure in the containers to increase.

5.9.3. Ensure all boiling flasks have cooled to room temperature before disconnecting liquid-liquid bodies from boiling flasks to prevent any burns.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware must be cleaned per Glassware Washing, SOP NC-QA-014.

6.2. Equipment and supplies for extraction procedures:

EQUIPMENT AND SUPPLIES	CLLE	Conc
pH Indicator paper, ranges: 0-14, 7.5-14, 0-6	Ö	
Graduated cylinder: 1 liter. (other sizes may be used as needed)	Ö	Ö
Methylene chloride collection tank	Ö	
Initial volume template	Ö	
Solvent dispenser pump or 100 mL graduated cylinder		Ö
Continuous liquid / liquid extractor	Ö	
Round or flat bottom: 250	Ö	
Boiling chips: contaminant-free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent)	Ö	Ö
Cooling condensers	Ö	
Heating mantle: rheostat controlled	Ö	
Auto-timer for heating mantle	Ö	
Beakers: 250 & 400 mL, graduated	Ö	Ö
Kuderna-Danish (K-D) apparatus: 500 mL		Ö
Concentrator tube: 10 mL, attached to K-D with clips		Ö
Snyder column: three-ball macro		Ö
Water bath: heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.		Ö
Vials: glass, 2 mL, 2.5mL, and 40 mL capacity with Teflon®-lined screw-cap		Ö
Nitrogen blowdown apparatus		Ö
Nitrogen: reagent grade.		Ö
Culture tubes: 10 mL, 16 mmx100 mm		Ö
Microliter pipette, syringe 1 mL	Ö	
Glass wool	Ö	
Glass funnel: 75 X 75 mm	Ö	Ö
Disposable pipettes, 5 ¾ in, and 9in.	Ö	Ö
Aluminum foil	Ö	Ö
Paper towels	Ö	Ö

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	CLLE	Conc
Sodium hydroxide (NaOH), pellets: reagent grade	Ö	
Sodium hydroxide solution, 10 N: dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	Ö	
Sulfuric acid (H_2SO_4), concentrated: reagent grade	Ö	
Sulfuric acid (1:1): carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	Ö	
Hydrochloric acid (HCl)	Ö	
Organic-free reagent water	Ö	
Sodium sulfate (Na_2SO_4), granular, anhydrous: purify by heating at 400°C a minimum of two hours	Ö	Ö
Extraction / exchange solvents: methylene chloride, hexane, acetonitrile, acetone, pesticide quality or equivalent	Ö	Ö
Acetone, methylene chloride: used for cleaning	Ö	Ö

7.2. Standards

7.2.1. Stock Standards

Stock standards are purchased as certified solutions. Semivolatile stock standards are stored at $\leq 6^{o}C$. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampoule is opened if purchased). Standards must be allowed to come to room temperature before use.

7.2.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 5. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3. Matrix Spiking and Laboratory Control Spiking Standards

The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 6. Spiking standards are purchased or prepared as dilutions of the stock standards.

Spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.4 See SOP NC-QA-017 for additional information on Standards and Reagents.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1 The holding time for aqueous samples is seven days from sampling to extraction.

8.3.2 For TCLP leachates, the holding time is 14 days from sampling to the leach process. The extraction holding time seven days from when the TCLP Leach tumbling has been completed, excluding the filtration step, to the extraction step.. If the filtration step requires extended times, this time counts as part of the seven-day holding time.

8.3.3. Analysis of the extracts is completed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS, and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS / MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See Policy QA-003 for further definition of the batch.

9.2. Sample Count

9.2.1. Laboratory-generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.3. Method Blank

9.3.1. A method blank consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. See Table 3 for the appropriate amount of surrogate to use for each analytical method. The method blank is used to identify any

background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.3.2. Aqueous Method Blanks use 1000 mL of reagent water spiked with the surrogates. The method blank goes through the entire analytical procedure.

9.3.3. TCLP method blanks use 250 mL of leachate fluid spiked with the surrogates. SPLP method blanks use 1000 mL of leachate fluid spiked with the surrogates The leachate may optionally be diluted to 1000 mL with reagent water. The method blank goes through the entire analytical procedure.

9.4. Laboratory Control Sample (LCS)

9.4.1. Laboratory Control Samples are well-characterized laboratory-generated samples used to monitor the laboratory day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure.

9.4.2. The LCS is made up in the same way as the method blank (see Sections 9.3.1 through 9.3.3), but spiked with the LCS standard and the surrogates. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method.

9.5. Surrogates

9.5.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.5.2. Each applicable sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. See Table 3 for the appropriate amount of surrogate spike to use for each analytical method.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method.

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9.7. Initial Demonstration of Capability

9.7.1. The initial demonstration and method detection limit studies described in Section 13 must be acceptable before analysis of samples may begin.

9.9 Control Limits

9.9.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.9.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs (QC Browser program).

9.10 Method Detection Limits (MDLs) and MDL Checks

9.10.1 MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.10.2 MDLs are accessible via LIMs (QC Browser program).

9.11 Nonconformance and Corrective Action

9.11.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volume of solvent into the appropriate size glass vial using a gastight syringe that is manufactured to a certified volume delivery tolerance of ± 0.01 mL. The "standard" glass vial is sealed, and the meniscus is marked by etching a line on the bottle. The glass vials containing the sample extracts are then compared against the "standard" glass vial to ensure the final volume is consistently 1.0 ± 0.01 mL. A log is kept of the glass vial lot number and preparation date.

11. PROCEDURE

Refer to SOP NC-QA-016 for information on DoD samples.

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC Manager. The Nonconformance memo will be filed in the project file. Procedural variations are not allowed for Ohio VAP projects.

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11.2. Continuous Liquid/Liquid Extraction from Water Samples

11.2.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.2.2 Assemble the apparatus. Add approximately 250 mL of methylene chloride to the extractor body. Add three to five boiling chips to the round-bottom distilling flask. Label the flask with an extraction ID label.

11.2.3 Measure the initial sample pH with wide-range pH by inserting a disposable pipette into the sample, and placing a drop of sample onto the wide range pH paper. Record on the extraction benchsheet.

11.2.4 Measure the initial volume using the volume template. Place the template next to the sample bottle and read the volume marking from the template. Record this volume on the benchsheet. Prepare a method blank, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method. Use 1 L of reagent water for method blanks and LCS. If the sample cannot be prepared using continuous liquid/liquid extraction due to matrix, a waste dilution may be required. Refer to Section 11.3 for the waste dilution procedure.

11.2.5 Use 250mL of leachate for TCLP semivolatiles and TCLP pesticides. Use 1000 mL of leachate for SPLP semivolatiles and SPLP pesticides. Dilute to about 1 liter with reagent water.

11.2.6 For a TCLP method blank, LCS, and LCS Dup, measure 250 mL of the buffer solution in a beaker and transfer to the continuous liquid/liquid extractor. Dilute to about 1 liter with reagent water. For an SPLP method blank, LCS, and LCS Dup, measure 1000 mL of the buffer solution using the volume template and transfer to the continuous liquid/liquid extractor. No dilution with reagent water is required.

11.2.7 Less than one liter of sample may be used for highly contaminated samples, or if the reporting limit can be achieved with less than one liter of sample. In this event, dilute the sample to about 1 liter with reagent water. This must be documented with a Non-Conformance Memo.

11.2.8 Add reagent water to the extractor body until approximately 150 mL of methylene chloride is pushed over into the round-bottomed flask to ensure proper operation and solvent cycling. Prime the extractor using reagent water. The method blank and samples are spiked with the surrogates, the LCS and matrix spikes with the surrogates, and matrix spiking solutions.

11.2.9 Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH, as necessary. Recheck the sample with pH paper. Record adjusted pH, spiking volumes and standard numbers on the

benchsheet. Return spiking solutions to the refrigerator as soon as possible. Attach cold condenser (about 10°C). Turn on heating mantle. Inspect joints for leaks once solvent has begun cycling. Extract for 18-24 hours (24 hours required for 600 series).

11.2.10 If extraction at a secondary pH is required (see Table 1), turn off the heating mantle and allow the extractor to cool. Detach the condenser and adjust the pH of the sample in the extractor body to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure by inserting a disposable pipette into the sample, and placing a drop of sample on the pH paper. Record the adjusted pH on the benchsheet. Re-attach the condenser, and turn on the heating mantle. Extract for 18-24 hours.

11.2.11 Turn off the heating mantle and allow the extractor to cool.

11.2.12 Cover with aluminum foil and refrigerate if the extract is not concentrated immediately. Refer to Section 11.4 for concentration.

11.3 Waste Dilution

11.3.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.3.2 Label the vial with the sample number. Tare the vial, then transfer approximately 1g of sample to the vial. Record the weight to the nearest ± 0.01g.

11.3.3 For the blank and LCS/LCSD, add a small amount of the appropriate solvent to the vial. Add appropriate volume of surrogate and spike solutions (Table 3).

11.3.4 Dilute to 10 mL with the appropriate solvent (Methylene Chloride for GC/MS Semi and GCS TPH). Add 10 mL of appropriate solvent (Hexane) for GCS pesticide and/or PCB analysis. Method 8015B and 8015C waste dilutions are diluted to approximately10 mL with DCM and are placed on the nitrogen evaporation unit to reduce to a 2 mL final volume.

11.3.5 Cap and shake or vortex each extract.

11.3.6 The sample is now ready for analysis.

11.4 Concentration

According to the type of sample, different solvents and final volumes will be required. Refer to Table 2 for the appropriate final volumes and concentrations.

11.4.1 Kuderna-Danish (KD) Method

11.4.1.1 Assemble a Kuderna-Danish concentrator by attaching a 10 mL

concentrator tube to the 500 mL K-D flask. Label the CT and K-D. Transfer the sample to the labeled K-D flask, filtering Continuous Liquid/Liquid and Soxhlet samples through funnels filled with sodium sulfate. Rinse the funnel with 20-30 mL of methylene chloride to complete the quantitative transfer.

11.4.1.2 Add one or two clean boiling chips to the KD flask and attach a three-ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column. (This is important to ensure that the balls are not stuck, and the column will work properly). Attach to the KD flask.

11.4.1.3 Place the KD apparatus on a water bath (90-98°C) so the tip of the concentrator tube is submerged. The water level must not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter; but the chambers should not flood.

11.4.1.4 Concentrate to 15-20 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent to the top of the Snyder Column, and then continue the water bath concentration back down to 5-8 mL. Refer to Table 2 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.4.1.5 Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.5 Nitrogen Evaporation to Final Concentration

11.5.1 Transfer the CT to the evaporation apparatus.

11.5.2 Place the tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but must not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.5.3 Refer to Table 1 to determine the final volume needed for a specific test method. Evaporate to slightly less than the required final volume.

Quantitatively transfer the extract to the appropriate final container and dilute to the appropriate final volume using the "standard" glass vial noted in Section 10.1. Cap the sample and affix the appropriate label. The sample is now ready for analysis.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.6 Analytical Documentation

11.6.1 Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.6.2 All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.6.3 Sample information and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which must contain all the analytes of interest. The spiking level must be equivalent to a mid-level calibration. (For certain tests, more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. See SOPs NC-GC-038, NC-MS-018, NC-MS-003, and NC-GC-007 for detailed information on the determinative methods.

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.2.2 Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files

14. POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention**."**

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Extracted aqueous samples contaminated with methylene chloride. This tank is then periodically rolled to the Tank Room, the pH is verified, contents are neutralized with sodium bicarbonate, pH re-verified, and Dichloromethane waste drained into a waste drum located outside the building. The wastewater is discharged to the POTW.

15.2.2. Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

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15.2.3. **Assorted flammable solvent waste from various rinses**. These wastes are put into the halogenated/non-halogenated 25 gallon solvent waste container located under the fume hood in extractions.

15.2.4. **Methylene chloride waste from various rinses:** These wastes are disposed of in the liquid-liquid separation unit.

15.2.5. **Hexane- Hexane waste:** These samples are to be disposed in the flammable waste.

15.2.6. **Waste Hexane in vials.** These vials are placed in the vial waste located in the GC prep laboratory.

15.2.7. **Waste Methylene Chloride sample vials**. These vials are placed in the vial waste located in the GC prep laboratory.

15.2.8. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCBs must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB, and PCB vial waste.

15.2.9. Solvent Recovery System Waste. Methylene Chloride waste from the Solvent Recovery System is collected and disposed of in the liquid-liquid separation unit. Acetone/Methylene Chloride waste from this system is disposed of in the flammable waste containers located in the laboratory.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B, 3520C, and 3580A

16.1.2 Federal Register - Environmental Protection Agency, 40 CFR, Part 136, Volume 49, No. 209, October 26, 1984, Method 625

16.1.3 EPA 600, Methods for Chemical Analysis of Water and Wastes, Method 608

16.1.2. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5. Revision History

Historical File:		Revision 3.4: 10/16/98		Revision 0: 03/12/08 (NC-OP-032)
(formerly CORP-OP-0001NC)		Revision 3.5: 04/22/99		Revision 1: 01/07/09 (NC-OP-032)
		Revision 3.6: 05/13/99		
		Revision 3.7: 03/20/01		
		Revision 3.8: 05/23/01		
		Revision 3.9: 04/22/02		
		Revision 4.0: 02/04/03		
		Revision 4.1: 10/07/03		
		Revision 4.2: 01/30/06		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work SOP, NC-QA-016

16.2.6. Gas Chromatographic Analysis based on Method 8000B, 8021B, 8081A, 8081B, 8082, 8082A, 8151A, 8015B, 8015C, and 615, NC-GC-038

16.2.7. GC/MS Analysis based on Method 8270C and 8270D, NC-MS-018

16.2.8. Analysis of Pesticides and PCBs by EPA Method 608, NC-GC-007

16.2.9. GC/MS Semivolatile Organic Compounds Capillary Column Technique Based on EPA Method 625, NC-MS-003

16.2.10. Standards and Reagents, NC-QA-017

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17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Spiking levels for method 608 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

17.2. Tables

TABLE 1
Liquid /Liquid Extraction Conditions

Determinative Method	Initial Ext. pH	Secondary Ext. pH
BNA: 8270C and 8270D[1]	1-2 (acid first), or 11-12 (base first)	11-12 (base), or 1-2 (acid)
625	11-12 (base first), or 1-2 (acid first)	1-2 (acid), or 11-12 (base)
Pest/PCB: 8081A, 8081B, 8082, 8082A and 608	5-9	None
Hydrocarbons: 8015B and 8015C	As received	None

[1] If the laboratory has validated acid only 8270 extraction for the target compound list required then the base extraction step may be omitted. The required validation consists of a four-replicate initial demonstration of capability and a method detection limit study (see Section 13). Additionally, either of the base or acid fractions of Method 8270 can be run first.

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TABLE 2

Final Volumes and Exchange Solvents

Type	Exchange Solvent for Analysis	Final Volume for Analysis in mL
Semivolatiles	N/A	2.0 mL
PCB	Approximately 18 mL hexane – water	5.0 for H2O 2.0 for H2O*
Pesticides	Approximately 18 mL hexane	5.0 for H2O 2.0 for H2O*
Pesticides/TCLP	Approximately 18 mL hexane	3.0 mL
BNA – SIM	N/A	2.0 mL
TPH	N/A	5 mL

* Michigan work requires a final volume of 2 mL.

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

TABLE 3

Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA	100/150 ppm BNA	0.2
BNA / SIM	100/150 ppm BNA	0.2 / 0.02
PEST	0.2 ppm DCB/TCX	1.0
TPH	40ng Nonane (C9)	1.0
PCB	0.2 ppm DCB/TCX	1.0

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TABLE 4
Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA	100 ppm BNA All-Analyte Spike and Restek Spike	0.2
BNA / SIM	100 ppm BNA All-Analyte Spike and Restek Spike	0.2 / 0.02
PEST	Pest NPDES Spike	1.0
PEST TCLP	Pest TCLP Spike	1.0
PCB	10 ppm PCB Spike	1.0
TPH	See Spike List – Table 6	1.0

TABLE 5
Surrogate Spike Components

Analyte Group	Compounds	Conc. (µg/mL)
BNA	2-Fluorobiphenyl	100
	Nitrobenzene-d5	100
	p-Terphenyl-d14	100
	2-Fluorophenol	150
	Phenol-d6	150
	2,4,6-Tribromophenol	150
	1,2-Dichlorobenzene-d4	100
	2-Chlorophenol-d4	150
PEST	Decachlorobiphenyl	0.2
PCB	Tetrachloro-m-xylene	0.2
TPH	Nonane (C9)	40.0

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	Acenaphthene	100
	4-Chloro-3-Methylphenol	100
	2-Chlorophenol	100
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	4-Nitrophenol	100
	Pentachlorophenol	150
	Phenol	100
	Pyrene	100
	1,2,4-Trichlorobenzene	100
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	Hexachlorobenzene	100
	Hexachlorobutadiene	100
	Hexachloroethane	100
	2-Methylphenol	100
	3-Methylphenol	100
	4-Methylphenol	100
	Nitrobenzene	100
	Pentachlorophenol	100
	Pyridine	100
	2,4,5-Trichlorophenol	100
	2,4,6-Trichlorophenol	100
	Acenaphthene	100
	Acenaphthylene	100
	Anthracene	100
	Benzo(a)anthracene	100
	Benzo(b)fluoranthene	100
	Benzo(k)fluoranthene	100
	Benzo(a)pyrene	100
	Benzo(ghi)perylene	100
	Benzyl butyl phthalate	100
	bis(2-chloroethyl)ether	100
	bis(2-chloroethoxy)methane	100
	bis(2-ethylhexyl)phthalate	100
	bis(2-chloroisopropyl)ether	100
	4-Bromophenyl phenyl ether	100
	2-Chloronaphthalene	100
	4-Chlorophenyl phenyl ether	100

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Type	Compounds	Conc. (µg/mL)
BNA	Chrysene	100
	Dibenzo(a,h)anthracene	100
	Di-n-butylphthalate	100
	1,3-Dichlorobenzene	100
	1,2-Dichlorobenzene	100
	3,3'-Dichlorobenzidine	100
	Diethyl phthalate	100
	Dimethyl phthalate	100
	2,4-Dinitrotoluene	100
	2,6-Dinitrotoluene	100
	Di-n-octylphthalate	100
	Fluoranthene	100
	Fluorene	100
	Hexachlorobenzene	100
	Hexachlorobutadiene	100
	Hexachloroethane	100
	Indeno(1,2,3-cd)pyrene	100
	Isophorone	100
	Naphthalene	100
	Nitrobenzene	100
	n-Nitrosodi-n-propylamine	100
	Phenanthrene	100
	1,2,4-Trichlorobenzene	100
	4-Chloro-3-methylphenol	100
	2-Chlorophenol	100
	2,4-Dichlorophenol	100
	2,4-Dimethylphenol	100
	2,4-Dinitrophenol	100
	2-Methyl-4,6-dinitrophenol	100
	2-Nitrophenol	100
	4-Nitrophenol	100
	Pentachlorophenol	100
	Phenol	100
	2,4,6-Trichlorophenol	100
	Acetophenone	100
	Atrazine	100
	Caprolactum	100
	Benzaldehyde	100
	1,1′-Biphenyl	100
	Safrole	100
	1,4-Dioxane	100

Type	Compounds	Conc. (µg/mL)
BNA	Pronamide	100
	p-Chlorobenzilate	100
	Phenacetin	100
	Ethyl methanesulfonate	100
	2-Picoline	100
	Phorate	100
	Quinoline	100
	Aniline	99.9
	Azobenzene	100
	Benzoic Acid	100
	bis-2-Ethylhexyl Adipate	100
	Carbazole	100
	Dibenzofuran	100
	Hexachlorocyclopentadiene	100
	n-Nitrosodimethylamine	100
	n-Nitrosodiphenylamine	100
	1-Methylnaphthalene	100
	1,2-Dinitroboenzene	100
	1,3-Dinitrobenzene	100
	1,4-initrobenzene	100
	2-Methylnaphthalene	100
	2-Nitroaniline	100
	2,3,4,6-Tetrachlorophenol	100
	2,3,5,6-Tetrachlorophenol	100
	3-Nitronaniline	100
	4-Chloroaniline	100
	4-Methylphenol	100
	4-Nitroaniline	100

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Type	Compounds	Conc. (µg/mL)
Pest TCLP	Heptachlor	0.5
	Heptachlor epoxide	0.5
	Lindane	0.5
	Endrin	0.5
	Methoxychlor	1.0
Pest NPDES/Pest	Alrin	1.0
	Alpha-BHC	1.0
	beta-BHC	1.0
	delta-BHC	1.0
	gamma-BHC (Lindane)	1.0
	4,4'-DDD	1.0
	4,4'-DDE	1.0
	4,4'-DDT	1.0
	Dieldrin	1.0
	alpha-Endosulfan	1.0
	beta-Endosulfan	1.0
	Endosulfan Sulfate	1.0
	Endrin	1.0
	Heptachlor	1.0
	Heptachlor Epoxide	1.0

Diesel Range Organics (8015B and 8015C) Spike	
Compound	**Final Concentration**
Diesel Fuel	500 ug/L

Title: SOXHLET (TRADITIONAL) EXTRACTION OF ORGANIC COMPOUNDS FROM SOILS BASED ON METHOD SW846 3540C AND WASTE DILUTION BASED ON METHOD 3580A

Approvals (Signature/Date):

Technology Specialist	03/23/11 Date	Health & Safety Coordinator	03/17/11 Date
Quality Assurance Manager	03/22/11 Date	Laboratory Director	03/16/11 Date
Technical Director	03/22/11 Date		

This SOP was previously identified as SOP No. NC-OP-032, Rev 1, dated 01/07/09, which is now split into four individual SOPs

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1. SCOPE AND APPLICATION

1.1 This SOP describes procedures for preparation (extraction) of semivolatile organic analytes in soil matrices for analysis by Gas Chromatography (GC) and Gas Chromatography / Mass Spectrometry (GC/MS) using Soxhlet Extraction. The procedures are based on SW846 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1.1. Extraction procedures for the following determinative methods are covered: 8081A, 8081B, 8082, 8082A, 8270C, and 8015B.

1.1.2. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

2. SUMMARY OF METHOD

2.1. Soxhlet Extraction (Accelerated and Traditional)

A 30 g sample is mixed with anhydrous sodium sulfate until free flowing. This is extracted with refluxing solvent.

2.2. Concentration

Procedures are presented for drying and concentration of the extract to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms and acronyms used in this SOP may be found in the glossary of the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health

and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded, other gloves must be cleaned immediately.

5.3. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds must be prepared in the hood.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride must be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of methylene chloride is spilled, evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints which can become stuck. Technicians must use Kevlar or other cut/puncture-resistant gloves when separating stuck joints.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware must be cleaned per Glassware Washing, SOP NC-QA-014.

6.2. Equipment and supplies for extraction procedures:

EQUIPMENT AND SUPPLIES	Sox	Conc
Graduated cylinder: 1 liter. (other sizes may be used as needed)		Ö
Erlenmeyer flask: 250 mL (other sizes optional)		Ö
Solvent dispenser pump or 100 mL graduated cylinder	Ö	Ö
Round or flat bottom: 250 mL	Ö	
Boiling chips: contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent)	Ö	Ö
Cooling condensers	Ö	
Heating mantle: rheostat controlled	Ö	
Auto-timer for heating mantle	Ö	
Soxgriddle	Ö	
Beakers: 450mL wide-mouth glass jars		Ö
Balance: >100 g capacity, accurate ±0.1 g	Ö	Ö
Soxhlet extractor	Ö	
Cellulose and glass thimbles Accelerated soxhlet extractor (Soxtherm™)	Ö Ö	
Kuderna-Danish (K-D) apparatus: 500 mL		Ö
Concentrator tube: 10 mL, attached to K-D with clips		Ö
Snyder column: three-ball macro		Ö

EQUIPMENT AND SUPPLIES	Sox	Conc
Water bath: heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.		Ö
Vials: glass, 2 mL and 40 mL capacity with Teflon®-lined screw-cap		Ö
Nitrogen blowdown apparatus		Ö
Nitrogen: reagent grade.		Ö
Culture Tubes: 10 mL, 16 mmx100 mm		Ö
Microliter pipette, syringe 1 mL	Ö	
Glass wool	Ö	
Glass funnel: 75 X 75 mm	Ö	Ö
Disposable pipettes, 5 ¾ in, and 9in.	Ö	Ö
Aluminum foil	Ö	Ö
Paper towels	Ö	Ö

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better, unless otherwise specified.

REAGENTS	Sox	Conc
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.	Ö	Ö
Magnesium sulfate	Ö	
Extraction/Exchange Solvents: Methylene chloride, hexane, acetonitrile, acetone, pesticide quality or equivalent	Ö	Ö
Acetone, Methylene Chloride: Used for cleaning	Ö	Ö

7.2. Standards

7.2.1. Stock Standards

Stock standards are purchased as certified solutions. Semivolatile stock standards are stored at $\leq 6^{o}$C. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampoule is opened if purchased). Standards must be allowed to come to room temperature before use.

7.2.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 4. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3. Matrix Spiking and Laboratory Control Spiking Standards

The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 5. Spiking standards are purchased or prepared as dilutions of the stock standards.

Spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.4 See SOP NC-QA-017 for additional information on Standards and Reagents.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1 The holding time for solid and waste samples is 14 days from sampling to extraction.

8.3.2 Analysis of the extracts is completed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS, and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS / MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See Policy QA-003 for further definition of the batch.

9.2. Sample Count

9.2.1. Laboratory-generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.3. Method Blank

9.3.1. A method blank consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at

the same level as the samples. See Table 2 for the appropriate amount of surrogate to use for each analytical method. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.3.2. Solid method blanks use approximately 30 g of sodium sulfate spiked with the surrogates. See Table 2 for the appropriate amount of surrogate to use for each analytical method. The method blank goes through the entire analytical procedure.

9.4. Laboratory Control Sample (LCS)

9.4.1. Laboratory Control Samples are well-characterized laboratory-generated samples used to monitor the laboratory day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure.

9.4.2. The LCS is made up in the same way as the method blank (see Sections 9.4.1 through 9.5.2), but spiked with the LCS standard and the surrogates. See Tables 2 and 3 for the appropriate amount of spike to use for each analytical method.

9.5. Surrogates

9.5.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.5.2. Each applicable sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. See Table 2 for the appropriate amount of surrogate spike to use for each analytical method.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike. See Tables 2 and 3 for the appropriate amount of spike to use for each analytical method.

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9.7. Initial Demonstration of Capability

9.7.1. The initial demonstration and method detection limit studies described in Section 13 must be acceptable before analysis of samples may begin.

9.8 Control Limits

9.8.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.8.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs (QC Browser program).

9.9 Method Detection Limits (MDLs) and MDL Checks

9.9.1 MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.9.2 MDLs are easily accessible via the LIMs (QC Browser program).

9.10 Nonconformance and Corrective Action

9.10.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volume of solvent into the appropriate size glass vial using a gastight syringe that is manufactured to a certified volume delivery tolerance of ± 0.01 mL. The "standard" glass vial is sealed, and the meniscus is marked by etching a line on the bottle. The glass vials containing the sample extracts are then compared against the "standard" glass vial to ensure the final volume is consistently 1.0 ± 0.01 mL. A log is kept of the glass vial lot number and preparation date.

11. PROCEDURE

Refer to SOP NC-QA-016 for information on DoD samples.

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC Manager. The

Nonconformance memo will be filed in the project file. Procedural variations are not allowed for Ohio VAP projects.

11.2 Soxhlet

11.2.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.2.2 Decant any water layer on a sediment/soil sample into the sample lid or mason jar and return it into the sample after removing the amount needed for the extraction unless there are specific instructions not to decant. Record and document if a water layer was present on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible, place the sample in a clean beaker and homogenize. Upon completion of homogenization in the beaker, return the sample to original container. Discard foreign objects such as sticks, leaves, and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials, consult with the client (via the Project Manager). If the sample cannot be prepared using a Soxhlet due to matrix issues, a waste dilution may be required. Refer to Section 11.3 for the waste dilution procedure.

11.2.3 Place approximately 200mL of solvent into a 250 mL flat bottom flask containing one or two clean boiling chips. Weigh 30g ± 0.5 g of sample into a thimble or in a beaker, recording the weight to the nearest 0.01g on the benchsheet. Sample weights less than 30g, but over 1g, may be used if the appropriate reporting limits can be met.

11.2.4 Prepare a method blank, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP, using sodium sulfate as the matrix for the LCS. The parent sample is used for the MS/MSD. The weight of sodium sulfate used must be approximately the weight of soil used in each sample.

11.2.5 Add anhydrous sodium sulfate to each sample and mix well. The mixture must have a free-flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to a soxhlet extractor thimble, but do not pack the thimble tightly. The soxhlet extractor or extraction thimble must drain freely for the duration of the extraction period. A glass wool plug below the sample in the soxhlet extractor is an acceptable alternative for the thimble.

11.2.6 Add the appropriate amount of surrogate and matrix spiking solution as indicated in Tables 2, 3, 4, and 5.

11.2.7 Attach the flask to the extractor and extract the sample for 16-24 hours at 4-6 cycles per hour. Check the system for leaks at the ground glass joints after it has warmed up.

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Note: If a reduced quantity of sample is extracted, it is usually necessary to increase the amount of sodium sulfate added or increase the solvent boiling rate to properly set the cycling rate.

Solvents:

Semivolatile GC/MS, TPH Organochlorine pesticides and PCBs	1:1 v/v Methylene Chloride / Acetone
8270 (MS) Concrete	Methylene Chloride

11.2.8 Allow the extract to cool after the extraction is complete, then disassemble by gently twisting the soxhlet from the flask.

11.2.9 The sample is now ready for the concentration step (Section 11.5).

11.2.10 Cover with aluminum foil and refrigerate if the extract is not concentrated immediately. Refer to Section 11.5 for concentration.

11.3 Waste Dilution

11.3.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.3.2 Label the vial with the sample number. Tare the vial, then transfer approximately 1g of sample to the vial. Record the weight to the nearest ± 0.01g.

11.3.3 For the blank and LCS, add a small amount of the appropriate solvent to the vial. Add appropriate volume of surrogate and spike solutions (Table 2).

11.3.4 Dilute to 10 mL with the appropriate solvent (Methylene Chloride for GC/MS Semi and GCS TPH). Add 10 mL of appropriate solvent (Hexane) for GCS pesticide and/or PCB analysis. Method 8015B and 8015C waste dilutions are diluted to approximately
10 mL with DCM and are placed on the nitrogen evaporation unit to reduce to a 2 mL final volume.

11.3.5 Cap and shake or vortex each extract.

11.3.6 The sample is now ready for analysis.

11.4 Concentration

According to the type of sample, different solvents and final volumes will be required. Refer to Table 1 for the appropriate final volumes and concentrations.

11.4.1 Kuderna-Danish (KD) Method:

11.4.1.1 Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to the 500 mL KD flask. Label the CT and KD. Transfer the sample to the labeled K-D flask, filtering Continuous Liquid/Liquid and Soxhlet samples through funnels filled with sodium sulfate. Rinse the funnel with 20-30 mL of methylene chloride to complete the quantitative transfer.

11.4.1.2 Add one or two clean boiling chips and the extract to be concentrated to the KD flask and attach a three-ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column. (This is important to ensure that the balls are not stuck, and the column will work properly). Attach to the KD flask.

11.4.1.3 Place the KD apparatus on a water bath (90-98°C) so the tip of the concentrator tube is submerged. The water level must not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter; but the chambers should not flood.

11.4.1.4 Concentrate to 15-20 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent to the top of the Snyder Column, and then continue the water bath concentration back down to 5-8 mL. Refer to Table 1 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.4.1.5 Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.5 Nitrogen Evaporation to Final Concentration

11.5.1 Transfer the CT to the evaporation apparatus.

11.5.2 Place the tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but must not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C

Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.5.3 Refer to Table 1 to determine the final volume needed for a specific test method. Evaporate to slightly less than the required final volume.

Quantitatively transfer the extract to the appropriate final container and dilute to the appropriate final volume using the "standard" glass vial noted in Section 10.1. Cap the sample and affix the appropriate label. The sample is now ready for analysis.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.6 Analytical Documentation

11.6.1 Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.6.2 All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.6.3 Sample information and associated QC are entered into LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which must contain all the analytes of interest. The spiking level must be equivalent to a mid-level calibration. (For certain tests, more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. See SOPs NC-GC-038, NC-MS-018, NC-MS-003, and NC-GC-007 for detailed information on the determinative methods.

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files

14. POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State, and local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention**."**

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.2. **Assorted flammable solvent waste from various rinses**. These wastes are put into the halogenated/non-halogenated 25 gallon solvent waste container located under the fume hood in extractions.

15.2.3. **Methylene chloride waste from various rinses:** These wastes are disposed of in the liquid-liquid separation unit.

15.2.4. **Hexane-Hexane waste:** These samples are to be disposed in the flammable waste.

15.2.5. **Waste Hexane in vials.** These vials are placed in the vial waste located in the GC prep laboratory.

15.2.6. **Waste Methylene Chloride sample vials**. These vials are placed in the vial waste located in the GC prep laboratory.

15.2.7. **Extracted solid samples contaminated with methylene chloride/acetone or acetone/hexane.** These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.8. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCBs must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB, and PCB vial waste.

15.2.9. Solvent Recovery System Waste. Methylene Chloride waste from the Solvent Recovery System is collected and disposed of in the liquid-liquid separation unit. Acetone/Methylene Chloride waste from this system is disposed of in the flammable waste containers located in the laboratory.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B, 3540C, and 3580A

16.1.2. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.4 Corporate Quality Management Plan (CQMP), current version16.1.5 Federal Register - Environmental Protection Agency, 40 CFR, Part 136, Volume 49, No. 209, October 26, 1984, Method 625

16.1.6 EPA 600, Methods for Chemical Analysis of Water and Wastes, Method 608

16.1.7 Revision History

Historical File:		Revision 3.4: 10/16/98		Revision 0: 03/12/08 (NC-OP-032)
(formerly CORP-OP-0001NC)		Revision 3.5: 04/22/99		Revision 1: 01/07/09 (NC-OP-032)
		Revision 3.6: 05/13/99		
		Revision 3.7: 03/20/01		
		Revision 3.8: 05/23/01		
		Revision 3.9: 04/22/02		
		Revision 4.0: 02/04/03		
		Revision 4.1: 10/07/03		
		Revision 4.2: 01/30/06		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work SOP, NC-QA-016

16.2.6. Gas Chromatographic Analysis based on Method 8000B, 8021B, 8081A, 8081B, 8082,8082A, 8151A, 8015B, 8015C, and 615, NC-GC-038

16.2.7. GC/MS Analysis based on Method 8270C and 8270D, NC-MS-018

16.2.8. Analysis of Pesticides and PCBs by EPA Method 608, NC-GC-007

16.2.9. GC/MS Semivolatile Organic Compounds Capillary Column Technique Based on EPA Method 625, NC-MS-003

16.2.10. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS

17.1. Modifications from Reference method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Spiking levels for method 608 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

17.2. Tables

TABLE 1

Final Volumes and Exchange Solvents

Type	Exchange Solvent for Analysis	Final Volume for Analysis in mL
Semivolatiles	N/A	2.0 mL
PCB	Approximately 18 mL Hexane – water Approximately 36 mL Hexane - solid	10.0 for solids
Pesticides	Approximately 18 mL Hexane	10.0 for solids
BNA – SIM	N/A	2.0 mL - Solids & H2O
TPH	N/A	5.0

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

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TABLE 2

Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA	100/150 ppm BNA	0.2
BNA / SIM	100/150 ppm BNA	0.2 / 0.02
BNA Waste Dilution	100/150 ppm BNA	0.5
PEST	0.2 ppm DCB/TCX	1.0
TPH	40ng Nonane (C9)	1.0
PCB	0.2 ppm DCB/TCX	1.0

TABLE 3
Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA	100 ppm BNA All-Analyte Spike and Restek Spike	0.2
	Waste Dilution	0.5
BNA / SIM	100 ppm BNA All-Analyte Spike and Restek Spike	0.2 / 0.02
PEST	Pest NPDES Spike	1.0
PCB	10 ppm PCB Spike	1.0
TPH	See Spike List – Table 5	1.0

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TABLE 4
Surrogate Spike Components

Analyte Group	Compounds	Conc. (µg/mL)
BNA	2-Fluorobiphenyl	100
	Nitrobenzene-d5	100
	p-Terphenyl-d14	100
	2-Fluorophenol	150
	Phenol-d6	150
	2,4,6-Tribromophenol	150
	1,2-Dichlorobenzene-d4	100
	2-Chlorophenol-d4	150
PEST	Decachlorobiphenyl	0.2
PCB	Tetrachloro-m-xylene	0.2
TPH	Nonane (C9)	40.0

TABLE 5
Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	Acenaphthene	100
	4-Chloro-3-Methylphenol	150
	2-Chlorophenol	150
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	4-Nitrophenol	150
	N-Nitroso-Di-n-Propylamine	100
	Pentachlorophenol	150
	Phenol	150
	Pyrene	100
	1,2,4-Trichlorobenzene	100
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	Hexachlorobenzene	100

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Type	Compounds	Conc. (µg/mL)
BNA	Hexachlorobutadiene	100
	Hexachloroethane	100
	2-Methylphenol	100
	3-Methylphenol	100
	4-Methylphenol	100
	Nitrobenzene	100
	Pentachlorophenol	100
	Pyridine	100
	2,4,5-Trichlorophenol	100
	2,4,6-Trichlorophenol	100
	Acenaphthene	100
	Acenaphthylene	100
	Anthracene	100
	Benzo(a)anthracene	100
	Benzo(b)fluoranthene	100
	Benzo(k)fluoranthene	100
	Benzo(a)pyrene	100
	Benzo(ghi)perylene	100
	Benzyl butyl phthalate	100
	Bis(2-chloroethyl)ether	100
	Bis(2-chloroethoxy)methane	100
	Bis(2-ethylhexyl)phthalate	100
	Bis(2-chloroisopropyl)ether	100
	4-Bromophenyl phenyl ether	100
	2-Chloronaphthalene	100
	4-Chlorophenyl phenyl ether	100
	Chrysene	100
	Dibenzo(a,h)anthracene	100
	Di-n-butylphthalate	100
	1,3-Dichlorobenzene	100
	1,2-Dichlorobenzene	100
	1,4-Dichlorobenzene	100
	3,3'-Dichlorobenzidine	100
	Diethyl phthalate	100
	Dimethyl phthalate	100
	2,4-Dinitrotoluene	100
	2,6-Dinitrotoluene	100
	Di-n-octylphthalate	100

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Type	Compounds	Conc. (µg/mL)
BNA	Fluoranthene	100
	Fluorene	100
	Hexachlorobenzene	100
	Hexachlorobutadiene	100
	Hexachloroethane	100
	Indeno(1,2,3-cd)pyrene	100
	Isophorone	100
	Naphthalene	100
	Nitrobenzene	100
	N-Nitrosodi-n-propylamine	100
	Phenanthrene	100
	Pyrene	100
	1,2,4-Trichlorobenzene	100
	4-Chloro-3-methylphenol	100
	2-Chlorophenol	100
	2,4-Dichlorophenol	100
	2,4-Dimethylphenol	100
	2,4-Dinitrophenol	100
	2-Methyl-4,6-dinitrophenol	100
	2-Nitrophenol	100
	4-Nitrophenol	100
	Pentachlorophenol	100
	Phenol	100
	2,4,6-Trichlorophenol	100
	Acetophenone	100
	Atrazine	100
	Caprolactum	100
	Benzaldehyde	100
	1,1′-Biphenyl	100
	Safrole	100
	1,4-Dioxane	100
	Pronamide	100
	p-Chlorobenzilate	100
	Phenacetin	100
	Ethyl methanesulfonate	100
	2-Picoline	100
	Phorate	100
	Quinoline	100

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Type	Compounds	Conc. (µg/mL)
Pest NPDES/Pest	Alrin	1.0
	Alpha-BHC	1.0
	beta-BHC	1.0
	delta-BHC	1.0
	gamma-BHC (Lindane)	1.0
	4,4'-DDD	1.0
	4,4'-DDE	1.0
	4,4'-DDT	1.0
	Dieldrin	1.0
	alpha-Endosulfan	1.0
	beta-Endosulfan	1.0
	Endosulfan Sulfate	1.0
	Endrin	1.0
	Heptachlor	1.0
	Heptachlor Epoxide	1.0

Diesel Range Organics (8015B) (8015C)Spike	
Compound	**Final Concentration**
Diesel Fuel	500 ug/L

APPENDIX B

Data Validation Checklist

Data Validation Completeness Worksheet
Level II

LDC#: _____
SDG#: _____
Laboratory: TestAmerica Dayton

Date: _____
Page _____
Reviewer: _____
2nd Reviewer: _____

Method:

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area	Code	Comments	Qualifier
I				
II				
III				
IV				
V				
VI				
VII				
VIII				
IX				

Codes: A = Acceptable
NA = Not provided/applicable
SW = See Worksheet
ND = No compounds detected
R = Rinsate
FB = Field Blank
D = Duplicate
TB = Trip Blank
EB = Equipment Blank

Validated Samples:

Sample ID	Q	Sample ID	Q	Sample ID	Q

Dick's Creek Data Review Summary Worksheet

SDG #	Laboratory: TestAmerica Dayton	Validator:	Validation Date:
Site:	AR/COC# na		Validation Level:
Matrix:	# of Samples:	Case Narrative Present:	Sample Receipt Docs Present:
COCs present:	COCs signed:	COCs dated:	Sample Container Integrity:
Analysis:			

Requested Analyses Not Reported			
Client Sample ID	**Lab Sample ID**	**Analysis**	**Comments**

Hold Time/Preservation Outliers						
Client Sample ID	Lab Sample ID	Analysis	Preservation	Collection Date	Preparation Date	Analysis Date

Dick's Creek PCB Worksheet

SDG:	Method:	Matrix:	Laboratory Sample IDs:
Batch #s:			

Analyte (outliers)	Method Blank	5X MB	LCS %R	MS %R	MSD %R	MS/MSD RPD	MS% R	MS/MSD RPD	LAB DUP RPD	LAB DUP RPD	LAB DUP RPD	FD RPD	FD RPD

Surrogate Recovery Outliers

NFG Limits (30-150); Lab Limits (TCMX 15-150; DCB 10-150)

Sample ID	TCMX %R	DCB %R	Sample ID	TCMS %R	DCB %R	Sample ID	TCMS %R	DCB %R	

ATTACHMENT C

DATA MANAGEMENT PLAN

SWMU 23 and Additional Areas 02 through 09
Data Management Plan
AK Steel Corporation
Middletown, Ohio

Prepared for:

AK Steel Corporation
9227 Centre Pointe Drive
West Chester, OH 45069

Prepared by:

KEMRON Environmental Services, Inc.
2343-A State Route 821
Marietta, OH 45750

November 2012

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TABLES

6-1 Conceptual Data Management Flow Scheme

FIGURES

1-1 Site Location
2-1 Project Organization Chart

ACRONYMS

AA	Additional Area
CCR	Current Conditions Report
CLP-SOW	Contract Laboratories Program - Statement of Work
DMP	Data Management Plan
EDD	electronic data deliverable
ENVIRON	ENVIRON International Corporation
GC	gas chromatograph
GIS	geographic information system
GPS	global positioning system
IM SOW	Interim Measures Scope of Work
MS/MSD	matrix spike/matrix spike duplicate
KEMRON	KEMRON Environmental Services, Inc.
NRDC	Natural Resources Defense Council
Ohio EPA	Ohio Environmental Protection Agency
PCBs	polychlorinated biphenyls
PFE	pressurized fluid extraction
PIP	Public Involvement Plan
PMP	Project Management Plan
QA	quality assurance
QAM	Quality Assurance Manual
QAPP	Quality Assurance Project Plan
QC	quality control
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
RPD	relative percent difference
SDG	sample delivery group
SOP	Standard Operating Procedure
SVOCs	semi-volatile organic compounds
TestAmerica	TestAmerica Analytical Testing Corporation
TCLP	Toxicity Characteristic Leaching Procedure
United States et al.	United States, State of Ohio, Sierra Club, and NRDC
USEPA	United States Environmental Protection Agency
VOCs	volatile organic compounds
%R	percent recovery

1.0 PROJECT DESCRIPTION

This *RFI Data Management Plan* (DMP) for SWMU 23 and Additional Areas of potential concern AA02-AA09 addresses the procedures necessary to document and track data and results associated with the RFI Work Plan – SWMU 23 and Additional Areas AA02-AA09, Middletown, Ohio in accordance with the requirements of the Consent Decree entered May 15, 2006. The Consent Decree was signed by United States et al. (i.e., United States, State of Ohio, Sierra Club, and Natural Resources Defense Council [NRDC]) and AK Steel (Case No. C-100530) and entered on May, 15, 2006.

This *DMP* describes the project; project organization and responsibility; sample identification; chain-of-custody guidelines; and data flow, transformation, reduction, transfer, validation, analysis, reporting, tracking, and security. All quality assurance/quality control (QA/QC) procedures will be structured in accordance with applicable United States Environmental Protection Agency (USEPA) and Ohio Environmental Protection Agency (OEPA) requirements, regulations, guidance, and technical standards, as documented in the SWMU 23 and Additional Areas *Quality Assurance Project Plan RFI* (QAPP) (KEMRON, 2012).

1.1 Objectives

The objective of this *DMP* is to provide an accurate and readily accessible method of efficient data management for the activities associated with the Additional Areas AA02-AA09 and SWMU23 for the Dicks Creek Study Area. This *DMP* describes the people, processes, and procedures required to establish and maintain a complete, accurate, user-friendly, and timely project database. The investigation will be conducted in accordance with this *DMP* are described in the SWMU 23 and Additional Areas RFI Work Plan (KEMRON, 2012)

The constituents of interest for the Additional Areas are polychlorinated biphenyls (PCBs) and RCRA metals and Volatile Organic Compounds (VOCs) at SWMU 23. Analytical data will be generated and documented in accordance with the RFI Quality Assurance Project Plan (QAPP) (KEMRON, 2012).

1.2 Study Area Description

The Current Conditions Report (CCR, Arcadis, 2012) documents current site conditions for AK Steel Middletown Works and surrounding properties. Information from this document has been excerpted and is presented in this section.

The AK Steel Middletown Works facility is located in southwestern Ohio, in Butler County, in the City of Middletown (Figure 1-1). Middletown Works manufactures steels, which includes carbon steel melting, casting, hot and cold rolling, and finishing operations. The entire Middletown Works facility covers an area of approximately 2,600 acres with each area involved in various aspects of iron and steel manufacturing and processing.

Several environmental investigations have taken place at various locations at the Middletown Works over the years. A CCR (ARCADIS, May 2012) has been developed which summarizes the history of environmental investigation programs at the Middletown Works. This document includes a summary review of historical aerial photographs, Sanborn fire insurance maps, and other historical data resources used for determining the industrial developmental history of AK Steel in Middletown. The CCR, PMP, supported with other AK

Steel environmental data reports and information from personal interviews and historic information regarding the placement of materials from historic dredging of Dicks Creek all were used in development of this RFI Work Plan.

SWMU 23 is a closed, unlined set of three (3) wastewater lagoons located on a parcel outside of Middletown Works facility, known as the former Coil Paint area. The former Coil Paint area is situated west of Middletown Works.

Additional Areas of Potential Concern AA02 through AA09 consist of off-site properties (not owned by AK Steel) that are a mixture of single family residential properties and small to medium-sized commercial/industrial properties located adjacent to Dicks Creek toward the southwest side of the Middletown Works. The table below provides a brief description of the individual AA parcels.

LOCATION	DESCRIPTION
ADDITIONAL AREAS OF POTENTIAL CONTAMINATION	
AA02	Former Glenn Cartage property (NS of DC, East of Yankee Rd)
AA03	Sturgell Property (NS of DC, W of RR bridge, E of Yankee Rd)
AA04	Back half of properties between G. Cartage and Sturgell Properties (NS of DC)
AA05	Old Armco Lot, NS DC, North of big meander
AA06	Former Burridge Machine Shop Property, NS DC (now a dairy outlet)
AA07	Former Cecil Osburn Lot (located between Station 12-18, NS of DC)
AA08	Pipeline Fill, Stations 32-36, along Oxford S.R., N of Outfall 002
AA09	Former Orman's Welding Property

NS=North Side DC=Dicks Creek

Additional information regarding the parcels/areas is detailed in the RFI Work Plan (KEMRON, 2012).

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

AK Steel has responsibility for all phases of the investigation. AK Steel has selected KEMRON Environmental Services, Inc. (KEMRON) to conduct the RFI. KEMRON will manage the project, direct the field investigations, manage all incoming data, develop and populate the project database, prepare the report, and perform any subsequent studies, as appropriate. Specific individuals will be designated to each responsibility upon implementation of the project. KEMRON will maintain consistent contact with the AK Steel Project Manager and will perform work in accordance with AK Steel direction and authorization.

2.1 Management Responsibility

Management responsibility, including the AK Steel Project Coordinator, AK Steel Project Manager, and the KEMRON Project Manager are described below.

2.1.1 AK Steel Project Coordinator

The primary function of the AK Steel Project Coordinator (Patrick Gallo) is to ensure that technical, financial, and scheduling objectives are achieved successfully. The Project Coordinator will approve all external reports (deliverables) before their submission to the United States et al.

2.1.2 AK Steel Project Manager

The AK Steel Project Manager (James Kemp) is responsible for implementing the project and has the authority to commit the resources necessary to meet project objectives and requirements. The AK Steel Project Manager will work directly with the KEMRON Project Manager to monitor and direct daily activities; communicate health and safety information to project personnel during daily safety tailgate meetings; regulate site admittance to personnel; and serve as a liaison between KEMRON, subcontractor, and regulatory personnel and the AK Steel Project Coordinator.

2.1.3 KEMRON Project Manager

The KEMRON Project Manager (Mary Lou Rochotte) has responsibility for ensuring that the project meets the objectives of the United States et al. and KEMRON's quality standards. The KEMRON Project Manager will provide assistance to the AK Steel Project Manager and Project Coordinator in terms of writing and distributing the RFI Work Plan and other key project documents to all those parties connected with the project. The KEMRON Project Manager will report directly to the AK Steel Project Coordinator and is responsible for technical quality control and project oversight.

2.1.4 KEMRON Database Manager

The KEMRON database manager (Dave Pitzer) will be responsible for the following:

- Setting up the project database as specified on a company server;
- Creating and maintaining lists of synonyms and valid values for data fields;
- Controlling access to the database;
- Performing routine maintenance on the database including backup and performance refinement;

- Protecting the usability and integrity of the database;
- After reviewing electronic data deliverables (EDDs), uploading the data into the database and verify the new data is formatted correctly and complete;
- Overseeing post-import quality control;
- Verifying that all data values are valid;
- Updating data records based on the data validation report, including the documentation of these changes; and
- Creating queries, data exports, and summary tables from the database to be used to produce text, tables, graphs, maps, isopleth plots, models, and other products used to describe the data. Assigning staff to quality control evaluation of summary tables, queries, and database export routines.

2.2 Field Responsibilities

The KEMRON field team leader (Sarah Burch or other designee) will support the AK Steel Project Manager. She is responsible for leading and coordinating the day-to-day activities of the various resource specialists under her supervision. Specific field team leader responsibilities include:

- Providing day-to-day coordination with the KEMRON Project Manager on technical issues in specific areas of expertise;
- Developing and implementing field-related work plans, assurance of schedule compliance, and adherence to management-developed study requirements;
- Coordinating and managing field activities, including sampling and drilling, and supervising field staff;
- Implementing QC for technical data provided by the field staff, including field measurement data;
- Adhering to work schedules provided by the AK Steel and/or KEMRON Project Manager;
- Authoring and approving of text and graphics required for field team efforts;
- Coordinating and overseeing technical efforts of subcontractors assisting the field team;
- Identifying problems at the field team level, resolving difficulties in consultation with the AK Steel and KEMRON Project Managers, implementing and documenting corrective action procedures, and providing communication between team and upper management; and
- Participating in preparation of the final report.

Field Technical Staff

The technical staff for this project will be drawn from KEMRON's pool of corporate resources and, as necessary, subcontractors. The technical staff will be utilized to gather and analyze data and to prepare various task reports and support materials. All of the designated technical team members are experienced professionals who possess the degree of specialization and technical competence to effectively and efficiently perform the required work.

2.3 Laboratory Responsibilities

The constituents of analytical interest for environmental samples and waste material are PCBs at AA02 through AA09 and RCRA metals and VOCs at SWMU 23. All laboratory data generation and project management responsibilities are detailed in the RFI QAPP (KEMRON, 2012).

2.3.1 TestAmerica Analytical Testing Corporation

TestAmerica is responsible for all environmental sample analyses including PCBs, VOCs and RCRA metals analyses of investigative samples (as applicable), and all waste material, including fluids generated during equipment decontamination, residual solids collected during sampling procedures.

TestAmerica Analytical Testing Corporation
3601 South Dixie Drive
Dayton, Ohio 45439
Phone: (937) 294-6856

If a different laboratory is determined to be appropriate, the change in laboratory will be addressed in an RFI QAPP addendum or revision.

Laboratory Project Manager
The TestAmerica laboratory Project Manager (Debbie Olszowska) will serve as the liaison to the KEMRON Project Manager and will be responsible for the following:

- Ensuring client specifications are met by communicating project and quality assurance requirements to the laboratory;
- Notifying laboratory personnel of incoming projects and sample delivery schedules;
- Monitoring the status of all projects in-house to ensure timely delivery of reports;
- Informing the client of project-related problems, resolving service issues, and coordinating technical issues with the laboratory staff;
- Coordinating client requests for sample containers and other services;
- Scheduling sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples;
- Coordinating subcontract work;
- Preparing data packages;
- Reviewing laboratory data reports, quotes, and sample login sheets;
- Reviewing and approving data reports prior to their release to the client; and
- Ensuring client specific reporting and quality control requirements are met**.**

Laboratory Division Manager
The TestAmerica laboratory division manager (Chris Weathington) provides the resources necessary to implement and maintain an effective and comprehensive quality assurance program and will be responsible for the following:

- Providing technical guidance to the analytical staff. The Division Manager may be the source point for technical help or designate an individual(s) to fill this role. A Division Manager appoints the technical directors for the appropriate fields of testing. The names of the technical director will be included in the national database.
  If a technical director is absent for a period of time exceeding 15 consecutive calendar days, the Division Manager must designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function (this can be one of the other technical directors). If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing;

- Ensuring that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented;
- Ensuring that personnel are free from any commercial, financial, and other undue pressures that might adversely affect the quality of their work;
- Ensuring TestAmerica's human resource policies are adhered to and maintained;
- Ensuring that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the laboratory specific Quality Assurance Manual (QAM) or laboratory SOPs may be suspended by the Division Manager;
- Reviewing and approving all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to;
- Establishing and maintaining a laboratory information system for tracking all samples in the laboratory and utilizes the system to ensure all sample holding times are met; and
- Assuming the responsibilities of the QA Officer in the temporary absence of the QA Officer.

Laboratory Operations Manager

The TestAmerica laboratory operations manager (Chris Weathington) will be responsible for the following:

- Monitoring standards of performance in quality control and quality assurance;
- Monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data;
- Ensuring that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory;
- Providing training direction to laboratory staff;
- Monitoring production efforts of the lab. Looking for ways to enhance production and improve quality through technical advances and improved Laboratory Information Management System utilization; and
- Coordinating and reviewing preparation of all test method SOPs.

Laboratory Quality Assurance Officer

The TestAmerica laboratory QA Officer (James Davis) has the overall responsibility for ensuring that systems are maintained to produce data that are technically sound, legally defensible, and of consistent quality in line with the laboratory specific QAM and SOPs. Specifically, the laboratory QA Officer will be responsible for the following:

- Serving as the focal point for QA/QC and be responsible for the oversight and/or review of Quality Control Data;
- Maintaining and updating the laboratory specific QAM. Ensure that the manual accurately reflects the procedures of the laboratory;
- Monitoring and communicating regulatory changes that may affect the laboratory to management, marketing, and the Corporate Director of Quality Assurance;

- Pursuing and maintaining appropriate laboratory certification and contract approvals. Arranging for the analysis of Performance Testing samples necessary to satisfy certification requirements;
- Training and advising the laboratory staff on QA/QC procedures that are pertinent to their daily activities;
- Functioning independent from laboratory operations for which he/she has quality assurance oversight;
- Possessing a general knowledge of the analytical test methods for which data audit/review is performed (and/or have the means of getting this information when needed);
- Arranging for or conduct internal audits; and
- Notifying laboratory management of deficiencies in the quality system and ensure corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs must be temporarily suspended or restricted following the procedures outlined in the laboratory specific QAM.

Final responsibility for project quality rests with KEMRON's Project Manager. Independent QA will be provided by the laboratory Project Manager and QA Officer prior to release of all data to KEMRON and AK Steel.

Laboratory Technical Staff
The TestAmerica laboratory technical staff will be responsible for sample analyses and identification of necessary corrective actions. The staff will report directly to the laboratory operations manager.

2.4 Quality Assurance Responsibilities

2.4.1 Data Validation

The data validator for the laboratory (identified in Section 2.3) will report directly to the KEMRON Project Manager and will be responsible for the following:

- Verifying complete chain-of-custody documentation;
- Comparing blank values to sample values (equipment blanks, lab blanks, and method blanks);
- Verifying sample analysis dates and preparation dates with respect to sample holding times;
- Evaluating data on matrix spike/matrix spike duplicate (MS/MSD) samples;
- Reviewing field documentation (where necessary);
- Summarizing and assigning validation codes to the sample data; and
- Documenting data that do not meet validation criteria.
- Independent data validation will be performed as specified in the RFI QAPP.

2.4.2 Data Assessment

The KEMRON Project Manager (Mary Lou Rochotte) is responsible for the assessment of data to assure the adherence to high scientific and technical standards.

2.5 Project Organization Diagram

Figure 2-1 presents a diagram illustrating the project organization.

3.0 SAMPLE IDENTIFICATION AND TRANSPORT

Sample labels are necessary to definitively identify the samples. Labels will be affixed to the sample container (not the caps) prior to or at the time of sampling. Pertinent information on the labels will be filled out in waterproof ink at the time of collection and will include the following information:

- Sample number/identification code;
- Name (initials) of collector;
- Date and time of collection;
- Project title and/or project number;
- KEMRON;
- Required analyses;
- Analytical method;
- Sample matrix; and
- Preservative.

After collection, all samples will be handled as little as possible. Field personnel will use extreme care to maintain the integrity of the samples. If samples are placed in a cooler with wet ice, personnel will package the ice so that melted ice will not leak into the sample containers or cause cross contamination. A complete description of sample identification and transport requirements can be found in the RFI *QAPP* (KEMRON, 2012).

4.0 CUSTODY GUIDELINES

Custody is a vital aspect of sampling procedures. The data and samples must be traceable from the time of sample collection, through sample analyses, data generation, and report preparation.

All containers will be shipped from the supplier to the KEMRON office or a location designated by the KEMRON Project Manager by common carrier in sealed containers or picked up in person from the laboratory. The supplier will include a shipping form or laboratory chain-of-custody listing all containers shipped and the purpose of each container. It is KEMRON's policy that all containers are considered to be in the custody of the supplier until the sample containers are received by KEMRON. Upon receipt, sample containers will be checked to determine if any breakage occurred during shipment. The containers will be maintained in the custody of the receiver in a clean, secure area until they are used for sample collection. Once samples have been collected, they will be maintained in the custody of the sampling personnel. The procedure for maintaining and documenting chain-of-custody in the field will be as follows:

1. The sample label that is affixed to the container will be inspected to confirm that all of the required information has been provided (see Section 3).

2. The sample container will be sealed in a zip-lock plastic bag or wrapped in bubble pack, and packed in a cooler chest in a manner that will minimize movement. All samples will be cooled with double-bagged wet ice.

3. For each cooler chest that is sent to the laboratory, a Chain-of-Custody Record will be completed. All information on the Chain-of-Custody Record and the sample container labels will be checked against field records and the samples will be recounted.

4. If a commercial courier service (e.g., Federal Express) is being used to transport the samples to the laboratory, the Chain-of-Custody Record will be signed by a member of the field team, and a copy of the Record will be retained by the field team. The remaining copies of the Record will be sealed in a zip-lock plastic bag and placed in the cooler chest with the samples. The cooler chest will be sealed with packaging tape and two custody seals that have been signed and dated by a member of the field team. Because commercial couriers will not sign chain-of-custody forms, the package routing documentation maintained by the courier service during shipping serves as chain-of-custody documentation for the samples.

All laboratory analysts and technicians must follow chain-of-custody protocols described in the respective laboratory's QAPP, which will be provided upon request.

5.0 DATA ACQUISITION AND RECORDING

5.1 Data Acquisition Requirements

Data may be acquired from previously collected sources (e.g., literature, databases, spreadsheets). Acceptance criteria for previously compiled data will be such that qualifying standards will allow for the combination of new and old data sets. Observations and transformation equations of the previous project(s) will be evaluated to assure that the assumptions of the previous source(s) are consistent with the current project.

5.2 Field Data Recording

All field activities will be recorded at the time of the activity. Several different types of records may be created during the sampling event. All records will be prepared in indelible ink, will be dated and signed by the author, and any errors will be corrected by drawing a single line through the error and initialing and dating the error. No pages should be removed from the field notebook. All records will be maintained in the project file as a permanent record.

Project field activities will be documented on daily work description logs. Subsurface lithology will be documented on boring logs. Standard log sheets for soil and sediment sampling will be completed, as appropriate, for each sample collected. These logs will be completed as samples are collected. Records of photo documentation and sample location description (i.e., distance measurement, latitude and longitude coordinates) will be maintained. Field notes will be signed at the end of each day. All field data records, including sampling logs, equipment calibration specifications, laboratory task orders (if needed), and chain-of-custody forms, will be reviewed for accuracy and completeness.

5.3 Laboratory Data Recording

For analytical data quality to be adequately assessed, it is imperative that expected reporting levels are communicated to the laboratory. The laboratory must supplement analytical results with all supporting QC data required for effective validation of the analytical data. Additional documentation of analytical QC data will be available upon request from the laboratories to support validation conclusions and data usability determinations.

Laboratory data reports are described in detail in the RFI QAPP (KEMRON, 2012).

Paper data deliverables will be generated after all samples in the sample delivery group (SDG) have been completed. EDDs will be produced in a format mutually agreed upon by the laboratory and the KEMRON Project Manager. The laboratory is responsible for maintaining consistency between EDDs and hardcopy data reports. A complete description of laboratory data recording can be found in the RFI *QAPP* (KEMRON, 2012).

5.4 Geospatial Data Recording

The location of all sampling and monitoring stations will be recorded using a global positioning system (GPS) unit, in addition to traditional surveying methods if required. In some cases, GPS satellites may be temporarily out of range, such that sufficient precision cannot be achieved at the time of sampling. In these cases, the station will be marked with a stake, and coordinates will be determined and recorded at a later time.

6.0 DATA FLOW

The flow of all data produced by this investigation will be overseen by the KEMRON Project Manager and is conceptualized in Table 6-1. After data are generated, they will be verified and validated, if appropriate, for accuracy before finalization. All data produced during this investigation will be maintained electronically both as individual EDDs and in a project database under the control of the database manager, as well as hard copy.

A project file will be developed for data generated during this investigation and will include the following items: all reports, field notes, laboratory reports, signed chain-of-custody forms, sampling procedures, and any other pertinent documents. These items shall be stored in a central location with access limited to select project personnel. The file custodian will be the KEMRON Project Manager or her designee. The project file will be maintained for a period of at least five (5) years after termination of the Consent Decree.

7.0 DATA TRANSFORMATION AND REDUCTION

Data transformation/reduction is the process of converting analytical data from instrument form into digital form and correcting for all sample weight, dilution factors, and percent solids calculations that may be applicable. This process is performed in the field and in the laboratory.

7.1 Field Data Reduction Procedures

Raw data from field measurements and sample collection activities will be recorded in the field records and on applicable field log sheets. Instrument readings will be recorded directly into the field records. Field personal will submit all field notes and field logs to the Project Manager at the end of each work week. These documents include daily logs, soil/sediment sampling logs, boring logs, and laboratory chain-of-custody documents. Errors will be corrected by drawing a single line through the error and initialing and dating the error. Recorded field data will be reviewed for accuracy and completeness by the KEMRON Project Manager or designee. Example field logs and other field records are contained in KEMRON SOPs, which are provided in the RFI QAPP (KEMRON, 2012).

7.2 Laboratory Data Reduction Procedures

Raw data from analytical laboratory measurements may or may not be generated by digital read-out instruments. In addition, due to dilution and concentration factors often developed during the analysis of the sample, careful data reduction is required to convert raw data to useable digital analytical data. All data reductions will be performed according to procedures presented in the laboratory's QAPP.

8.0 DATA TRANSFER

8.1 Field Measurement Data

Raw handwritten field measurement data will be transferred from the original log sheets to electronic data forms. The person who performed the analysis will either enter the data into the electronic forms or they will carefully review the forms if a surrogate enters the data. These data will then be uploaded in to the project database by the database manager into the "FieldData" table in the project database. Measurements will include: sample number, sample location, date, sample time, sample depths, sample lithology, sampling personnel, any additional collection data, and a memo for any other information related to sample collection. The KEMRON Project Manager or designee will review the transferred data for completeness and notify the source of any content or format problems. Records will be kept within the database of any changes made to the data after it has been entered into the electronic forms.

8.2 Laboratory Measurement Data

Laboratory data will be transferred from the Laboratory Project Manager to the KEMRON Project Manager electronically as EDDs as they are processed and as a hard copy data package after completion of the SDG. EDDs will be assigned unique identifiers and a date to allow for data tracking. Once received, each EDD and the data package will be added to the project data file by the database manager. The KEMRON Project Manager or designee will review the data package for completeness and notify the laboratory of any content or format problems. Records will be kept within the data file of any changes made to the data.

Laboratory data will be entered into a project database as they are received and will include sample results and data qualifiers. Sample results will include: sample ID, reach, area, location, location ID, date sampled, depth sampled, analytical method, compound name, laboratory data qualifiers, results, and units. Additional QC requirements will be added to the database including laboratory duplicate results, reagent water blank spike duplicate results, and matrix spike duplicates results.

8.3 Geospatial Data

Coordinates of all field sampling and monitoring locations will be uploaded from the GPS unit (or manually entered) into the "GISData" table in the project database. Additional analysis may be required to fully populate the database (e.g., river name and river mile for sediment and surface water samples). Traditional surveying measurements will be manually entered into the project database. Geospatial measurements will include location code and latitude and longitude coordinates of each sample location.

8.4 Electronic Data Edits

All edits and updates to data after they have been uploaded into the project database will be recorded in the database. The "Edits" table will be populated by the database manager or designee at the time of all data edits to the three primary data tables (GISData, FieldData, LabData). The following information will be recorded: table name, file name, original value, updated value, date, rationale, and a memo for any additional information that may be relevant. Updates to queries and valid value tables will not be tracked.

9.0 DATA VALIDATION

Data validation is a systematic process for reviewing a body of assembled analytical results against a set of criteria to determine if the data are adequate for the intended use. Data validation involves (1) data editing; (2) data screening and checking; (3) data auditing and verification; (4) data certification; and (5) final data review and informed decision-making whereby data are accepted, qualified, or rejected.

All analytical data developed from the RFI investigation will be validated according to criteria specified in the RFI QAPP (KEMRON, 2012). The data validation procedures employed for this project will include an evaluation of the field data packages, an evaluation of the laboratory's analytical data packages, and documentation of data that do not meet validation criteria.

Analytical data will be submitted to KEMRON in both hard copy and computerized format, organized to facilitate data management. The laboratory-provided data flags may include such items as estimated concentrations, concentrations below required detection limits, and concentrations of chemicals also found in blanks. All data will then be processed through a Data Validation Checklist for Level II reporting requirements. The checklist and other validation specifications are detailed in the RFI QAPP (KEMRON, 2012).

The database manager will be responsible for uploading all data validation changes into the project database and maintaining a record of all such changes in the database.

10.0 DATA ANALYSIS AND REPORTING

Project data will be processed in order to create data products for use in decision-making and reporting. Analyses may include statistical interpretation of results, risk assessments, and other approaches. Analysts may compile, separate, or summarize data in their analysis, so as to best utilize field measurements, laboratory measurements, and geospatial data. These data will be subsequently used to produce text, tables, graphs, maps, isopleth plots, models, and other products used to describe the data. Results of such analyses may lead to additional analyses.

All data obtained during RFI activities will be submitted to USEPA, OEPA, and Intervenors in the RFI Report. All locational and analytical data will be submitted in an electronic database compatible with standard geographic information system (GIS) viewers/software. Deliverables will include, but not be limited to, monthly progress reports, data summary reports, and RFI Report.

11.0 PUBLIC INVOLVEMENT

The RFI *Public Involvement Plan* (KEMRON, 2012) addresses the tools necessary to keep the public informed regarding activities associated with investigation, remediation, and restoration of the Dicks Creek Study Area. Consistent with the RFI *PIP*, the following measures will be taken.

11.1 Information Repository

An Information Repository will be established at the Middletown Public Library to ensure that site-related information is available to the public. All work plans and reports containing site-related data will be available within 30 days of approval. The address, phone number, and hours of operation for the repository are provided below.

MidPointe Middletown Public Library
125 S. Broad Street
Middletown, OH 45044
513-424-1251

Hours of Operation:
Monday through Thursday 9:00 a.m. – 7:00 p.m.
Friday 9:00 a.m. – 7:00 p.m.
Saturday 9:00 a.m. – 5:00 p.m.
Sunday 1:00 p.m. – 5:00 p.m.

The repository is handicapped accessible and contains photocopying capabilities.

11.2 Additional Public Involvement

Various letters, fact sheets, meetings and other public involvement activities may be conducted as described in the RFI PIP. All information and records generated and issued to the public will be retained in the project record.

12.0 DATA TRACKING AND SECURITY

Processed data will be tracked from receipt of the QA field and analytical data to the creation of the final RFI report. All data processing tracking records will be stored in the project file.

Data security measures help ensure that critical data are not lost, destroyed, or altered without supporting documentation. Measures include preserving both electronic and hard copy forms of all original data and chain-of-custody forms, maintaining the project database with routine backups, and limiting access to the project file to select personnel.

Within KEMRON, the project database is maintained on a company server. Interconnected personal workstations and laptops allow access to the database through a wide area network. Use of a server allows for central data storage and management of data set access. Access is limited to select KEMRON project personnel. All hard copy data generated through the current project will be stored in a project file maintained at KEMRON. Documents used by USEPA et. al and AK Steel to make project decisions will be retained by AK Steel as required by the Consent Decree.

13.0 REFERENCES

ENVIRON. 2006e. Interim Measures Data Management Plan. June.

KEMRON. 2012, RCRA Facility Investigation Work Plan, SWMU 23 and Additional Areas AA02-AA09. November.

USEPA. 2000. Guidance for Data Quality Assessment: Practical Methods for Data Analysis. EPA/600/R-96/084. U.S. Environmental Protection Agency. July.

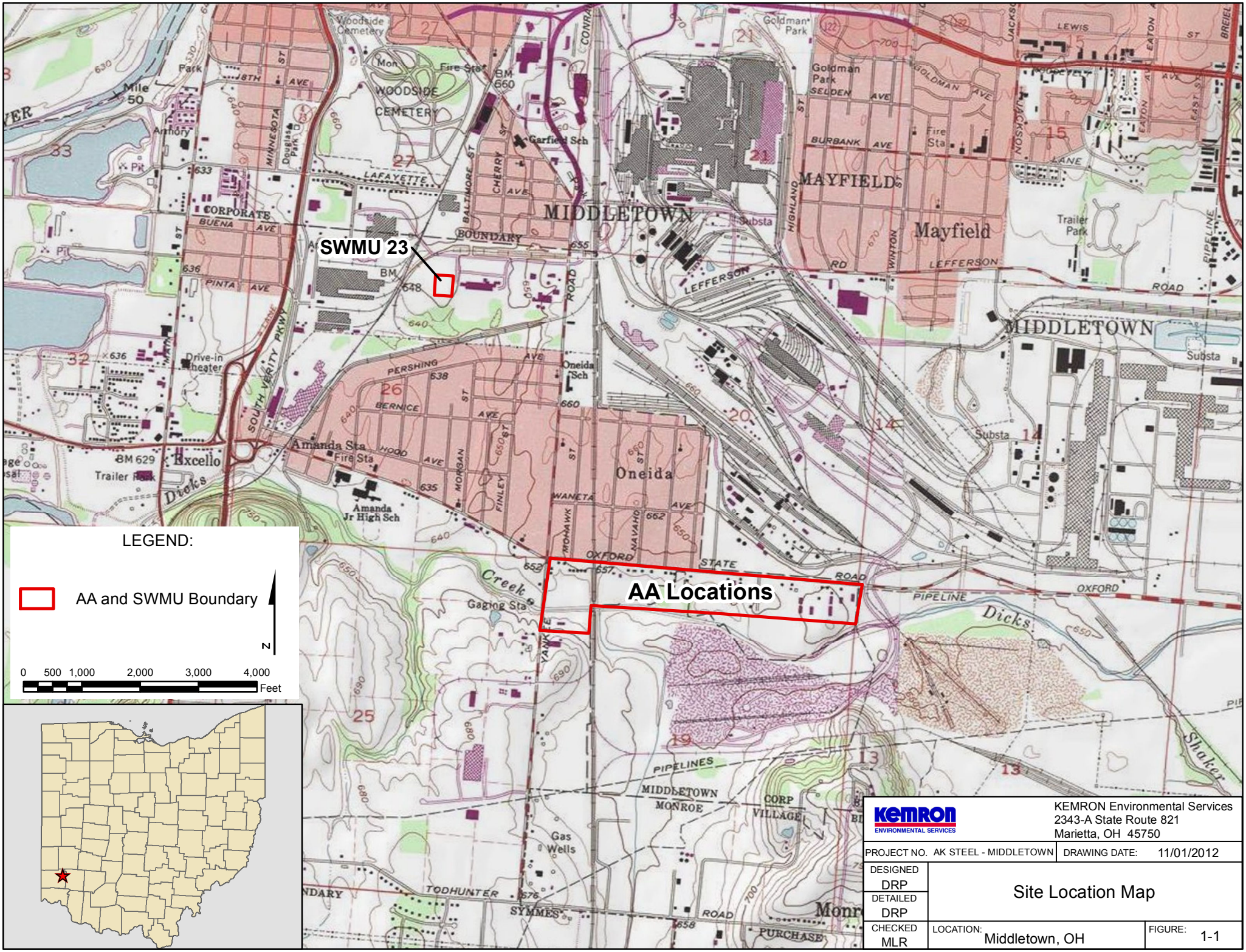
USEPA. 2002b. Guidance for Quality Assurance Project Plans. EPA/240/R-02/009. U.S. Environmental Protection Agency. December.

TABLE 6-1

Table 6-1. Conceptual Data Management Flow Scheme
SWMU 23 and Additional Areas 02 through 09, Middletown, OH

Data Flow Direction	Responsible Party	Receives	Transmits
	Field Technical Staff KEMRON, others	• Collects field data • Collect environmental media samples	• Field data sheets and samples to Field Team Leader
	Field Team Leader KEMRON	• Field data sheets from Field Technical Staff • Samples from Field Team Staff	• QA Field data to KEMRON Project Manager • Samples to Sample Custodian
	Laboratory Sample Custodian	• Samples from Field Team Leader	• Samples to Laboratory Technical Staff
	Laboratory Analysts	• Samples Laboratory Analysts	• Sample analyses to Laboratory Operations Manager
	Laboratory Operations Manager	• Sample analyses from Laboratory Analysts	• Analytical report to Laboratory Project Manager
	Laboratory Project Manager	• Analytical report from Laboratory Operations Manager	• Analytical report to Laboratory QC Officer
	Laboratory QA Officer	• Analytical report from Laboratory Project Manager	• QA analytical report to Laboratory Project Manager
	Laboratory Project Manager	• QA analytical report from Laboratory QA officer	• Finalized QA analytical report to KEMRON Database Manager
	Database Manager KEMRON	• QA data from Field Team Leader and Laboratory Project Manager	• Finalized QA analytical data to KEMRON Project Manager or designee
	Independent Data Validator	• Final analytical data from database manager	• Independent data review and validation results to KEMRON database manager
	Database Manager KEMRON	• Independent data review and validation results	• Finalized electronic data to KEMRON Project Manager
	Project Manager KEMRON	• QA field data from KEMRON Database Manager • Finalize QA analytical report from Laboratory Project Manager	• Finalized QA data report to AK Steel Project Manager • All data generated to project file
	Project Coordination AK Steel	• Finalized QA data report from KEMRON Project Manager	*End of External data flow*

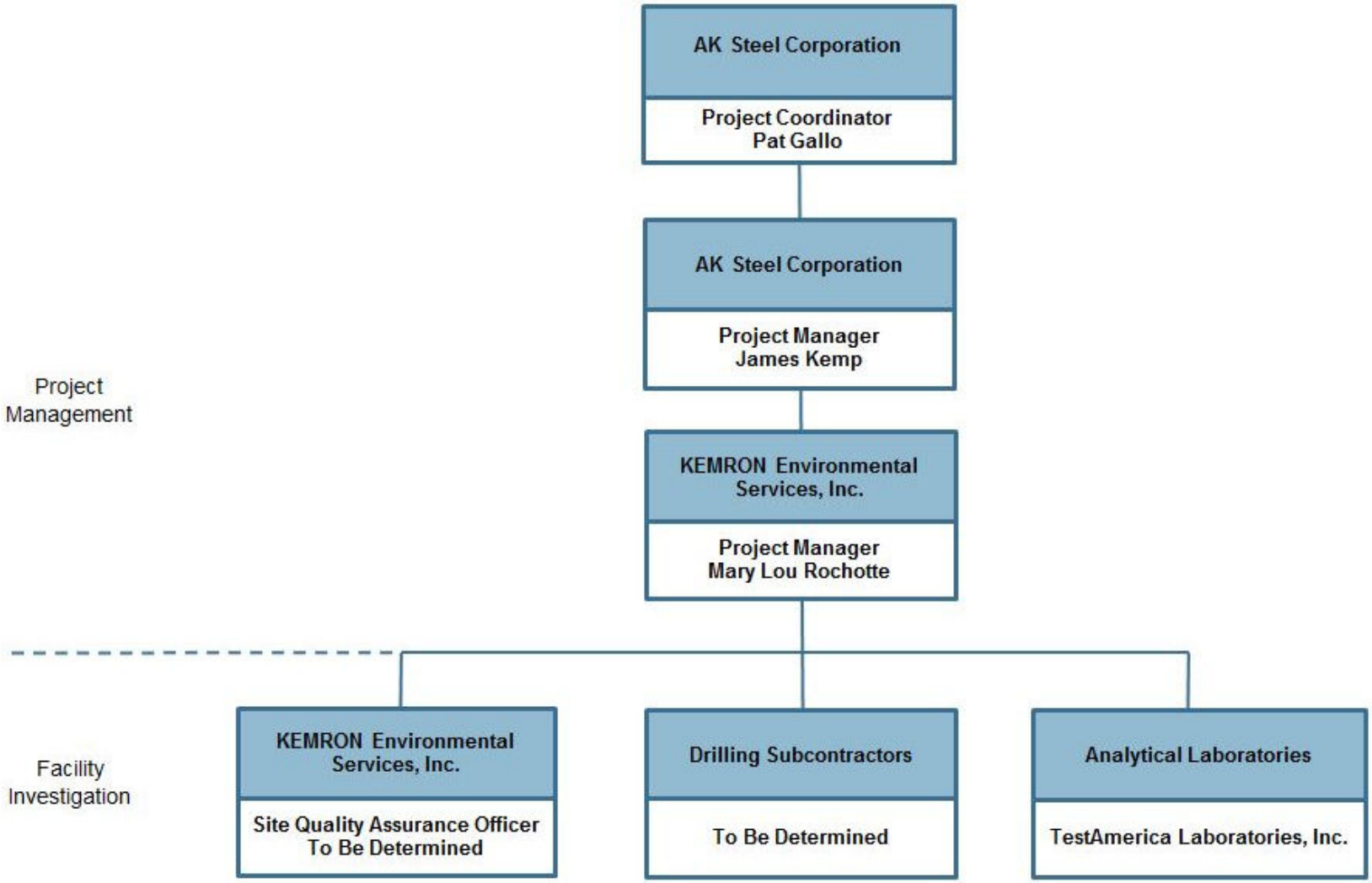
FIGURES



KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO.	AK STEEL - MIDDLETOWN	DRAWING DATE:	11/01/2012
DESIGNED	DRP	Site Location Map	
DETAILED	DRP		
CHECKED	MLR	LOCATION: Middletown, OH	FIGURE: 1-1



KEMRON ENVIRONMENTAL SERVICES	KEMRON Environmental Services 2343-A State Route 821 Marietta, OH 45750	
PROJECT NO. AK STEEL - MIDDLETOWN	DRAWING DATE: 11/01/2012	
DESIGNED DRP DETAILED DRP	Project Organization Chart	
CHECKED MLR	LOCATION: Middletown, OH	FIGURE: 2-1

ATTACHMENT D

PUBLIC INVOLVEMENT PLAN

RCRA Facility Investigation Public Involvement Plan
SWMU 23 and Additional Areas AA02-AA09
AK Steel Corporation
Middletown, Ohio

Prepared for:

AK Steel Corporation
9227 Centre Pointe Drive
West Chester, OH 45069

Prepared by:

KEMRON Environmental Services, Inc.
2343-A State Route 821
Marietta, OH 45750

November 2012

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ATTACHMENTS

Attachment 1 Example Letter
Attachment 2 Example Fact Sheet
Attachment 3 Example Press Release

ACRYONMS

AA	Additional Area of Potential Concern
AK Steel	AK Steel Corporation
AOC	Area of Concern
APR	Accredited in public relations
ARMCO	American Rolling Mill Co.
CMS	Corrective Measures Studies
ETC	Environmental Technologies & Communications, Inc.
HWMU	Hazardous Waste Management Unit
IMS	International Mill Services
KEMRON	KEMRON Environmental Services, Inc.
NRDC	Natural Resources Defense Council
OEPA	Ohio Environmental Protection Agency
PCB	Polychlorinated biphenyl
PIP	Public Involvement Plan
PRSA	Public Relations Society of America
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
SOW	Scope of Work
SWMU	Solid Waste Management Unit
United States et al.	United States, State of Ohio, Sierra Club, and NRDC
U.S.	United States
USEPA	United States Environmental Protection Agency
VOCs	Volatile Organic Constituents

1.0 INTRODUCTION

This *RFI Public Involvement Plan for SWMU 23 and Additional Areas 02 through 09* details communications and activities that AK Steel Corporation (AK Steel) will use to keep the public informed and involved during activities associated with the Resource Conservation and Recovery Act (RCRA) Facilities Investigation (RFI) described in the Consent Decree (Attachment 2, RFI Scope of Work [SOW]) for Solid Waste Management Unit (SWMU) 23, Former Coil Paint Area and eight Additional Areas. The Consent Decree was signed by United States et al. (i.e., United States [U.S.], State of Ohio, Sierra Club, and Natural Resources Defense Council [NRDC]) and AK Steel (Case No. C-100530) and entered on May, 15, 2006.

This Public Involvement Plan (PIP) is divided into the following sections:

Project Background - describes the project area and events leading up to the Consent Decree and RFI;

RFI Work - describes the progress made on the RFI for SWMU 23 and Additional Areas;

Community Profile - presents a profile of the Middletown community surrounding the portions of the AK Steel Middletown Works where SWMU 23 and Additional Areas are located;

Communication Goals, Objectives, and Strategies - outlines the major communication goal of the project and the objectives and strategies that will be used to help achieve this goal;

Public Outreach and Involvement Activities - discusses the communications and activities that will be used to keep the public informed and involved during the RFI process; and

Schedule - describes the schedule for specific public involvement activities.

2.0 PROJECT BACKGROUND

2.1 General Project Area

The AK Steel Middletown Works is a major steel making facility located in southwestern Ohio, in Butler County, in the City of Middletown, Ohio (Figure 1). The facility, which covers approximately 2,600 acres, is dedicated to iron and steel making and processing. Parts of the facility have been in operation since 1912 when AK Steel's predecessor, the American Rolling Mill Co. began production in this location.

Solid Waste Management Unit (SWMU) 23 is a closed, unlined set of three (3) wastewater lagoons on a parcel west of the Middletown Works facility. It is located in an area known as the former Coil Paint area. The Coil Paint area formerly was an active AK Steel operational unit that ceased operation in the 1980's; AK Steel no longer owns or operates manufacturing facilities at this site. The property that includes the former lagoons remains in AK Steel ownership. The location of SWMU 23 is illustrated by Figure 2.

The eight Additional Areas of Potential Concern (AA), AA02 through AA09, consist of off-site properties (not owned by AK Steel) that are a mixture of single family residential properties and small to medium-sized commercial/industrial properties located adjacent to Dicks Creek toward the southwest side of the Middletown Works. Figure 2 illustrates their locations to the southwest of Middletown Works.

Additional details regarding the background and history of SWMU 23 and the eight AAs are presented in the RFI Work Plan (KEMRON, 2012).

2.2 Lawsuit and Administrative Order

In June 2000, the U.S. Department of Justice, on behalf of the U.S. Environmental Protection Agency (USEPA) sued AK Steel under the Clean Water Act over polychlorinated biphenyl (PCB) contamination in Dicks Creek. Shortly thereafter, the USEPA issued an Administrative Order requiring the same actions from AK Steel that it sought through the lawsuit. AK Steel sought relief from the Order, based on a human health assessment (ARCADIS, 2004a) and an ecological risk assessment (ARCADIS, 2004b) indicating that no imminent threat to human health or the environment was present. In January 2003, the USEPA withdrew the Administrative Order but continued the lawsuit. Expert reports were submitted to the court in 2004, after which the parties entered settlement negotiations. These negotiations culminated in a Consent Decree that became effective on May 15, 2006 between AK Steel and the United States, State of Ohio, Sierra Club, and NRDC (United States et al.).

2.3 Consent Decree

Signed in April 2006 and effective on May 15, 2006, the Consent Decree between AK Steel and the United States et al. calls for AK Steel to conduct Resource Conservation and Recovery Act (RCRA) Facility Investigations and Corrective Measures Studies (RFI) at the Hazardous Waste Management Units (HWMUs), Solid Waste Management Units (SWMUs), Areas of Concern (AOCs), spill areas, and other suspected source areas listed in Exhibit A to Attachment 2 of the Consent Decree. The Consent Decree also requires AK Steel to conduct 12 Interim Measures to remediate PCBs and other contaminants in soil, sediment, and groundwater. The Decree includes a Supplemental Environmental Project that serves the public interest.

This Public Involvement Plan only discusses the RFI for SWMU 23 and Additional Areas (Figure 2); however, it is anticipated this PIP will be amended in the future to include other RFI and/or CMS work performed under the Consent Decree.

3.0 RFI WORK

The RFI process is an investigation to collect and analyze data necessary for evaluating the nature and extent of contamination at specific known or suspected affected areas, and assessing the risk to human health and environment posed by the contamination. The tasks comprising the RFI phase are based on the existing available information and data. Where data already exists, it may be used in the RFI as long as it meets quality assurance standards. Data gaps are identified and investigation conducted to fill the gap. The corrective measures phase (or CMS phase) will follow the RFI phase if unacceptable contamination levels are identified. A CMS phase is designed to identify, evaluate, and select appropriate corrective measures alternatives (if any) for the contamination. Many plans must be developed, reviewed by USEPA et. al, and approved by USEPA before most of the work can go forward. Progress to date on development of these plans is summarized in Table 1.

3.1 SWMU 23

SWMU 23 consists of three (3) closed wastewater treatment lagoons at the west end of the former Coil Paint plant building, located west of Middletown Works and outside the current plant boundaries. The parcel on which the lagoons were located remains in AK Steel Asset Management ownership, while the surrounding parcels are owned by others. The unlined lagoons were in operation from 1971 to 1978. Two (2) of the lagoons were used to remove suspended solids from the Coil Paint wastewater stream. The third lagoon was used to dry the solids from the other two lagoons. Water from dewatering the solids was allowed to percolate through a sand and gravel bed into the underlying soil. The lagoons were closed in the mid-1980s and capped with clay and a vegetated soil cover. The capped lagoons remain in this state today.

Records indicate that the wastewater discharged into the lagoons contained metals constituents. Additionally, since painting was conducted at the Coil Paint plant area, USEPA and OEPA have indicated that volatile organic constituents (VOCs) should be evaluated in the investigation of this site.

3.2 ADDITIONAL AREAS

Additional Areas of Potential Concern AA02 through AA09 are parcels located adjacent to Dicks Creek. Historic records indicate that sediment removed from Dicks Creek in the 1960's and/or 1980's may have been placed on these eight (8) parcels. Two of the properties are in residential use, while the remaining six (6) are currently commercial or industrial use properties.

3.3 Previous Investigations

Several investigations have been conducted in and around SWMU 23 and Additional Areas. These investigations are summarized in the following documents:

1. *Investigation of Ground Water Flow Conditions at the Armco Plant, Middletown, Ohio*, Geraghty & Miller, 1989;

2. *Data Summary Report: Floodplain Soil (March 7-June 15, 2005), Dicks Creek Study Area, Middletown Ohio,* ENVIRON, August 2005;

3. *Data Summary Report. Phase II Floodplain Soil, Dicks Creek Study Area, Middletown,*

Ohio, ENVIRON, December 2006.

4. *Data Summary Report. Phase III Floodplain Soil (October 9-December 20, 2007), Dicks Creek Floodplain Soil, Middletown Ohio,* ENVIRON, March 2008;

5. *Current Conditions Report, AK Steel Corporation, Middletown Works, Middletown Ohio*, Version 5, ARCADIS, May 11, 2012.

These previous investigations were evaluated to be potential sources of laboratory analytical data relevant to the project areas.

Groundwater data currently available from monitoring wells near SWMU 23 do not indicate impacts from the historic operations. However, additional investigation of groundwater and soil in and immediately adjacent to the former lagoons will be conducted to further assess site conditions.

A limited amount of soil data related to the Additional Areas of Potential Concern has indicated low level PCB concentrations in soil samples collected on a limited area. Additional soil sampling and analyses will be conducted during the RFI to determine the PCB concentrations present on each parcel.

For both SWMU 23 and the Additional Areas of Potential Concern, the data from the investigation will be used to determine whether any further evaluation is necessary of any of the parcels based on potential risk to human health and the environment.

4.0 COMMUNITY PROFILE

Land use in Middletown is mixed, as shown in Table 2. The two largest land uses are residential (29%) and industrial (17.0%). About 12% is still farmed. Only 6% is used commercially. Industrial land use dominates the portion of Middletown south of Roosevelt Boulevard (a major east-west road) and along Oxford State Road. Major industrial entities include AK Steel, MADE, Greentree, and Midd-Cities. AK Steel is the single largest land user in Middletown, with approximately 2,600 acres under common ownership. Residential land use dominates the area north of Roosevelt Boulevard and east of University Boulevard. Public, institutional, and commercial land uses are also located adjacent to primary thoroughfares.

Middletown is an incorporated city in Butler County, Ohio with a population of approximately 48,500 people, according to the United States Census Bureau (2006-2008 community survey, U.S. Census Bureau, 2010). The population is relatively stable; it has neither grown nor declined much over the last 10 to 12 years, and is not expected to grow much in the future. Community data from the 2000 U.S. Census and a 2006-2008 U.S. Census Bureau community survey are provided in Table 3. These data reflect the distribution of age, race, housing, education, income, occupation, and other metrics. The following salient points can be made from the community data:

- Approximately 25% of the population of Middletown is under the age of 18, which is slightly lower than that of the general U.S. population (25.7%), according to the U.S. Census Bureau.
- The median age of Middletown was 36.2 years in 2000 and 36.4 years from 2006 to 2008. The median age is comparable to the national median age of 35.3 years in 2000 and 36.7 from 2006 to 2008.
- Approximately 84% to 87% of the Middletown population is Caucasian, slightly higher than the national average. Approximately 10.6% to 11.8 % of the Middletown population is African American, slightly lower than the national average.
- Of the population in Middletown aged 25 years or more, 76.9% to 80.6 % have a high school degree or higher, while only 13.1% to 13.5% have a bachelor's degree or higher. These education rates are slightly lower than the national average of approximately 80.4% to 84.5% with a high school degree or higher and approximately 24.4% to 27.4% with a bachelor's degree or higher.
- Census data indicated that about 27% of the Middletown population is employed in manufacturing. Other local business types include retail, health care, food service, wholesale, and finance.
- In 2000, the percentage of family living below the poverty level was the same (9.2%) for the City of Middletown and the U.S. However from 2006 to 2008, the City of Middletown had a higher percentage of families living below the poverty level (13.5%) in comparison to the national average (9.6%).
- In 2000 and from 2006 to 2008, the city of Middletown has more civilian veterans (14% and 11.1%, respectively) than the national average (12.7% and 10.10%, respectively).

These community data were taken into consideration during the development of the public involvement plan.

5.0 COMMUNICATION GOALS, OBJECTIVES, AND STRATEGIES

5.1 Goals

The primary communication goal for the SWMU 23 and Additional Areas RFI work is to keep the public informed and involved throughout the RFI process.

5.2 Objectives

To achieve this goal, the following key objectives have been identified:

1. Increase public awareness of the RFI and its progress.

2. Increase public knowledge of how to get more information about the RFI and its progress.

5.3 Strategies

To meet the above objectives, the following strategies will be employed:

1. Communicate regularly and openly with the public and the media.

2. Provide clear, timely, and understandable information about the process, progress, and schedule.

3. Use existing Interim Measures public involvement tools and techniques to build on current information exchanges.

5.4 Tactics

For specific communications and activities during the RFI work, please see Section 6.0.

5.5 Target Audiences

The target audiences for the RFI work include the following:

- Citizens who live near the AK Steel Middletown Works and the RFI areas;
- Business owners/managers with businesses nearby the AK Steel Middletown Works and the RFI areas;
- Places of worship, schools and other organizations nearby the AK Steel Middletown Works and the RFI areas;
- Middletown, Butler County, Ohio, and U.S. elected and appointed officials;
- Middletown fire and police; and,
- Local news media.

6.0 PUBLIC OUTREACH AND INVOLVEMENT ACTIVITIES

This section details the specific communications and activities that will be used during the RFI work to keep the public informed and involved.

To best serve the public interest, and to ensure that the goals and objectives of this plan are met, AK Steel has retained the services of Environmental Technologies & Communications, Inc. (ETC) of Cincinnati, Ohio, a professional independent environmental community relations firm to assist with the day-to-day public outreach and involvement activities.

6.1 Public Relations Coordinator

AK Steel has designated Ms. Bethany Dale of ETC as the Public Relations Coordinator for the RFI work. She serves as the primary point of contact for all questions, concerns, comments, etc. from the public related to the project. Her contact information is:

Bethany Dale
ETC, Inc.
4000 Executive Park Drive, Suite 400
Cincinnati, Ohio 45241
Toll-Free: 866-902-4257
Fax: 513-772-7904
Email: bethany.dale@etc-online.com

Ms. Dale is accredited in public relations (APR) by the Public Relations Society of America (PRSA). She is a Vice President and Project Manager for ETC and has more than 14 years of professional experience in environmental community relations.

All materials distributed to the public or media (e.g., letters, fact sheets, press releases) will contain the toll-free Environmental Information Line number, mailing address, fax and e-mail address for the Public Relations Coordinator.

Ms. Dale (or designated ETC staff in her absence) will answer calls from the public on the toll-free Environmental Information Line, respond to letters, faxes and e-mails from the public, coordinate public meetings, manage the Information Repository, write and design fact sheets, and handle other communications activities listed in this section.

6.2 Toll-free Environmental Information Line

A telephone hotline was established in June 2006 to allow for the public to contact the Public Relations Coordinator with questions, concerns and comments about the Interim Measures remediation (e.g., current and upcoming activities, Information Repository). This toll-free information line will also be used to allow the public to contact the Public Relations Coordinator with questions, concerns, and comments about the RFI work. Anyone wishing to add or delete his or her name to the mailing list can also use this number for that purpose. The number is:

1-866-902-4AKS or 1-866-902-4257

The hotline will be answered during normal business hours, 8:30 a.m. to 4:30 p.m., Monday through Friday by Ms. Bethany Dale, the Public Relations Coordinator or an ETC designee in

her absence. After-hours callers can leave a message, which will be returned by Ms. Dale or designee by the next business day. A log of all calls will be maintained.

6.3 Information Repository

A public Information Repository was established in the Middletown Public Library to ensure that the public has access to all the project reports, data, and documents regarding the Interim Measures remediation. A similar but separate Information Repository will be established to address the RFI reports, data, and documents. The RFI Repository will include copies of the Consent Decree and attachments, documents that have been approved by the USEPA, Ohio Environmental Protection Agency (OEPA) and the Sierra Club, monthly progress reports, correspondence with the regulators, AK Steel fact sheets, AK Steel press releases and Middletown Journal newspaper stories. ETC will maintain a master list of the documents available in the Repository, updating the list on a monthly basis as newly approved documents are added to the repository.

The Repository will be housed at the MidPointe Middletown Public Library. The address, phone number, and hours of operation for the repository are provided below.

MidPointe Middletown Public Library
125 S. Broad Street
Middletown, OH 45044
513-424-1251

Hours of Operation:
Monday through Thursday 9:00 a.m. – 9:00 p.m.
Friday 9:00 a.m. – 7:00 p.m.
Saturday 9:00 a.m. – 5:00 p.m.
Sunday 1:00 p.m. – 5:00 p.m.

Visitors to the Information Repository should go to the Reference Desk to sign the visitors log book and review the current master list of documents. Visitors will then be directed to the area of the library where the documents are kept. No documents from the AK Steel repository may leave the library. Visitors may make copies of any document in the repository for a fee using the library copier equipment. The library is handicapped accessible and has a photocopying machine that charges $0.15/copy.

All materials distributed to the public or media (e.g., letters, fact sheets, press releases) will contain the location, phone number, and address for the Information Repository at the MidPointe Middletown Public Library.

6.4 Mailing List

In order to disseminate information easily and effectively to interested parties, the mailing list that was prepared for the Interim Measures work will also be used for the RFI work. This list currently includes over 700 entries, including property owners along Dicks Creek; households, businesses, churches, and organizations along Oxford State Road, Yankee Road, and Main Street in the vicinity of Dicks Creek; staff and families who attend Amanda Elementary School on Oxford State Road; and other interested citizens and key stakeholders (e.g., regulators, AK Steel, Middletown officials, Butler County officials, area environmental groups, and Middletown fire and police chiefs).

The mailing list will be maintained to keep contact information current. It will be revised based on calls made to the Environmental Information Line, sign-in sheets from public meetings, and coordination with elected officials and public organizations. The list will be revised through time based on citizen's requests to be added or removed, and as the Consent Decree work locations change. The mailing list will not be made available to the public to address privacy concerns.

6.5 Letters and Fact Sheets

As the RFI work progresses, AK Steel will prepare and mail letters and an informational fact sheet to everyone on the mailing list to communicate various aspects of the project. At a minimum, these mailings will include:

- Letter announcing the beginning of investigative work. These mailings will be sent out after the RFI Work Plan is approved, including establishment of a final schedule for initiation of field work.

- A cover letter and fact sheet describing the results of the RFI will be sent out after approval of the RFI report by United States et al.

The letters and the fact sheet will be clear and easy to read. The letters will usually be no longer than one page, and the fact sheets will be no longer than one page, front and back. All correspondence will contain information on how to contact the Public Relations Coordinator and access the Information Repository. They will be printed and distributed to members of the mailing list, handed out at public meetings, and placed in the Information Repository. The fact sheet will be submitted to USEPA for review and comment before it is finalized and released to the public. See Attachment 1 and Attachment 2 for an example letter and an example fact sheet, respectively.

6.6 Media Relations

AK Steel will prepare and issue press releases or fact sheets whenever a major event occurs during the RFI work. At a minimum, these press releases or fact sheets will include:

- Details of public meetings hosted by AK Steel.
- Beginning work of investigative work and, following completion of the schedule for such releases or fact sheets will be determined on a case by case basis.

In general, the press releases or fact sheets will be issued in concert with mailings to the mailing list.

Press releases will be developed by ETC, but Mr. Barry Racey, Director, Government and Public Relations for AK Steel, will issue the releases and serve as the company's official media spokesperson.

All media contacts will be local and include the following news organizations:

- Middletown Journal – Daily newspaper providing the most thorough coverage of AK Steel news (circulation 19,300 on weekdays and 19,900 on Sunday);
- Channel 24 TV Middletown – Local cable TV station; and,
- WPFB Radio Middletown – Local radio station.

If reporters from the media call the Environmental Information Line, ETC will coordinate with AK Steel including Mr. Racey. Mr. Racey will obtain assistance as needed from ETC for arranging press interviews and conferences. Attachment 3 provides an example press release.

6.7 Public Meetings and Availability Sessions

Public meetings and availability sessions may be held during the RFI work to provide additional information to the public about progress being made. These meetings will also provide the community an opportunity to talk directly to AK Steel representatives about the issues that concern them. If USEPA and OEPA attend the meetings, AK Steel will provide an opportunity for citizens to approach the Agencies' representatives with questions.

If necessary, AK Steel may hold a public meeting and/or availability session within 45 days of final approval of the RFI Work Plan to discuss site investigation activities conducted to date and the anticipated future schedule.

Anyone who cannot attend a public meeting or availability session due to illness or disability can contact the Public Relations Coordinator via the toll-free Environmental Information Line, letter, e-mail, or fax to request an opportunity to communicate directly with a representative of AK Steel. AK Steel will also provide the person(s) with copies of the meeting agenda and handouts.

An agenda will be prepared for each public meeting, which will include presentations, posters, handouts, and/or demonstrations. The information will be clear and understandable. The availability sessions will be small group question and answer sessions with an AK Steel representative(s). There will be no formal agenda, but visual information will also be prepared.

Invitations to the public meetings and availability sessions will be sent out two weeks prior to each event to everyone on the mailing list, via a press release to the three Middletown media outlets, and through postings at local gathering places (e.g., library, local churches). The announcements will give the meeting location, time, and topic. The meetings will be professionally facilitated and conducted in a respectful and orderly manner. The meetings will be held at a neutral and convenient public location.

In addition, AK Steel will be available to assist USEPA and Ohio EPA if the agencies decide to hold their own public meetings or availability sessions. AK Steel will provide reasonable support, upon request, for these meetings.

AK Steel and its consultants will not speak for or in any way indicate they represent the USEPA or the OEPA when interacting with the public, government officials, or media.

6.8 Public Input

Since there is no formal Public Comment Period during the RFI work, AK Steel will document all public input (e.g., comments and questions) received via the toll-free Environmental Information Line, e-mails, letters, faxes, public meetings and availability sessions. These comments and inquiries and any necessary resolution will be included in the Monthly Progress Reports, which are prepared by AK Steel and submitted to USEPA, et al. The Monthly Progress Reports will be placed in the Information Repository on a monthly basis. To protect the identity of the person making comment or inquiry, the progress reports will include the date the public input was received, the city where the citizen currently resides, and the comment or question from the

citizen. If USEPA requests the identity of the person making the comment, AK Steel will provide the Environmental Information Line call log, e-mail, fax, letter, or summary from public meetings/availability sessions.

7.0 SCHEDULE

Public involvement activities already initiated or scheduled for future activity are outlined in Table 4. The schedule for the conduct of the RFI for SWMU 23 and the AAs is provided in the *SWMU 23 and Additional Areas of Potential Concern RFI Work Plan* (KEMRON, 2012).

8.0 REFERENCES

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TABLES

Table 1. Progress on RFI Work Plans for SWMU 23 and Additional Areas

Work Plan	Progress or Status
Current Conditions Report (CCR)	Approved
Project Management Plan (PMP)	Approved
Draft RFI Work Plan for SWMU 23 and Additional Areas	Submitted November 21, 2012 for EPA review and approval
RFI Data Management Plan	Submitted November 21, 2012 for EPA review and approval
RFI Public Involvement Plan	Submitted November 21, 2012 for EPA review
RFI Quality Assurance Project Plan	Submitted November 21, 2012 for EPA review and approval
RFI Health and Safety Plan	Submitted November 21, 2012 for EPA review

RFI: Resource Conservation and Recovery Act (RCRA) Facility Investigation
SWMU: Solid Waste Management Unit
EPA: U.S. Environmental Protection Agency

Table 2. Land Use by Area, Middletown, Ohio

Land Use	Acres	Percent
Office	46	0%
Mobile Home	98	1%
Multiple Family	440	3%
Park	497	3%
Semipublic	572	4%
Commercial	1,026	6%
Vacant	1,254	8%
Public	1,458	9%
Not Classified	1,778	11%
Agriculture	1,964	12%
Industrial	2,691	17%
Single Family	4,033	25%
Total	***15,857***	

Source: 2005-2010 City of Middletown Master Plan

Table 3. Census and Community Survey Statistics for Middletown, Ohio

		2000 Census			2006-2008 Community Survey		
		Middletown (Number)	Middletown (Percent)	U.S. (Percent)	Middletown (Number)	Middletown (Percent)	U.S. (Percent)
Middletown Population	**Total**	51,605	(X)	(X)	48,500	(X)	(X)
	Male	24,647	47.8	0.491	22,864	47.1	49.30%
	Female	26,958	52.2	50.90%	25,636	52.9	50.70%
Age	Median age (years)	36.2	(X)	35.3	36.4	(X)	36.7
	Under 5 years	3,707	7.2	6.80%	3,348	6.9	6.90%
	Under 18 years	NR	25	25.7	NR	NR	NR
	18 years and over	38,693	75	74.30%	36,624	75.5	75.50%
	18 to 24 years	NR	9.3	NR	NR	10	NR
	25 years and over	34,115	(X)	(X)	31,953	(X)	(X)
	25 to 44 years	NR	29.2	NR	NR	NR	NR
	45 to 64 years	NR	21.6	NR	NR	NR	NR
	65 years and over	7,702	14.9	12.40%	6,589	13.6	12.60%
Race or Ethnic Group	One race	50,874	98.6	97.60%	47,254	97.4	97.80%
	Caucasian	44,886	87	75.10%	40,743	84	74.30%
	African American	5,467	10.6	12.30%	5,743	11.8	12.30%
	American Indian and Alaska Native	128	0.2	0.90%	137	0.3	0.80%
	Asian	190	0.4	3.60%	218	0.4	4.40%
	Native Hawaiian and Other Pacific Islander	18	0	0.10%	0	0	0.10%
	Some other race	185	0.4	5.50%	413	0.9	5.80%
	Two or more races	731	1.4	2.40%	1,246	2.6	2.20%
	Hispanic or Latino (of any race)	460	0.9	12.50%	789	1.6	15.10%
Household/Family Size	Average household size	2.38	(X)	2.59	2.44	(X)	2.61
	Average family size	2.94	(X)	3.14	3.07	(X)	3.2

		2000 Census			2006-2008 Community Survey		
		Middletown (Number)	Middletown (Percent)	U.S. (Percent)	Middletown (Number)	Middletown (Percent)	U.S. (Percent)
Housing	Total housing units	23,144	(X)	(X)	23,114	(X)	(X)
	Occupied housing units	21,469	92.8	91.00%	19,697	85.2	88.00%
	Owner-occupied housing units	12,895	60.1	66.20%	11,647	59.1	67.10%
	Renter-occupied housing units	8,574	39.9	33.80%	8,050	40.9	32.90%
	Vacant housing units	1,675	7.2	9.00%	3,417	14.8	12.00%
Education (Population 25 years and over)	High school graduate or higher	26,233	76.9	80.40%	(X)	80.6	84.50%
	Bachelor's degree or higher	4,460	13.1	24.40%	(X)	13.5	27.40%
Labor/Occupation	In labor force (population 16 years and over)	25,098	62.4	63.90%	23,749	62.8	65.20%
	Management, professional, and related	NR	24.3	NR	NR	NR	NR
	Service	NR	16.3	NR	NR	NR	NR
	Sales and office	NR	25.5	NR	NR	NR	NR
	Farming, fishing, and forestry	NR	0.1	NR	NR	NR	NR
	Construction, extraction, and maintenance	NR	9.6	NR	NR	NR	NR
	Production, transportation, and material moving,	NR	24.2	NR	NR	NR	NR
	Agriculture, forestry, fishing and hunting	NR	0.1	NR	NR	NR	NR
	Manufacturing	NR	27	NR	NR	NR	NR
	% government workers (local, state, or federal	NR	8.3	NR	NR	NR	NR

		2000 Census			2006-2008 Community Survey		
		Middletown (Number)	Middletown (Percent)	U.S. (Percent)	Middletown (Number)	Middletown (Percent)	U.S. (Percent)
Income and Poverty Level	Median household income in 1999 (dollars)	36,215	(X)	41,994	39,600	(X)	52,175
	Median family income in 1999 (dollars)	43,867	(X)	50,046	49,513	(X)	63,211
	Per capita income in 1999 (dollars)	19,773	(X)	21,587	20,543	(X)	27,466
	Families below poverty level	1,292	9.2	9.20%	(X)	13.5	9.60%
	Individuals below poverty level	6,444	12.6	12.40%	(X)	19	13.20%
Other Metrics	Civilian veterans (civilian population 18 years	5,458	14	12.70%	4,072	11.1	10.10%
	Disability status (population 5 years and	10,976	23.1	19.30%	(X)	(X)	(X)
	Foreign born	521	1	11.10%	933	1.9	12.50%

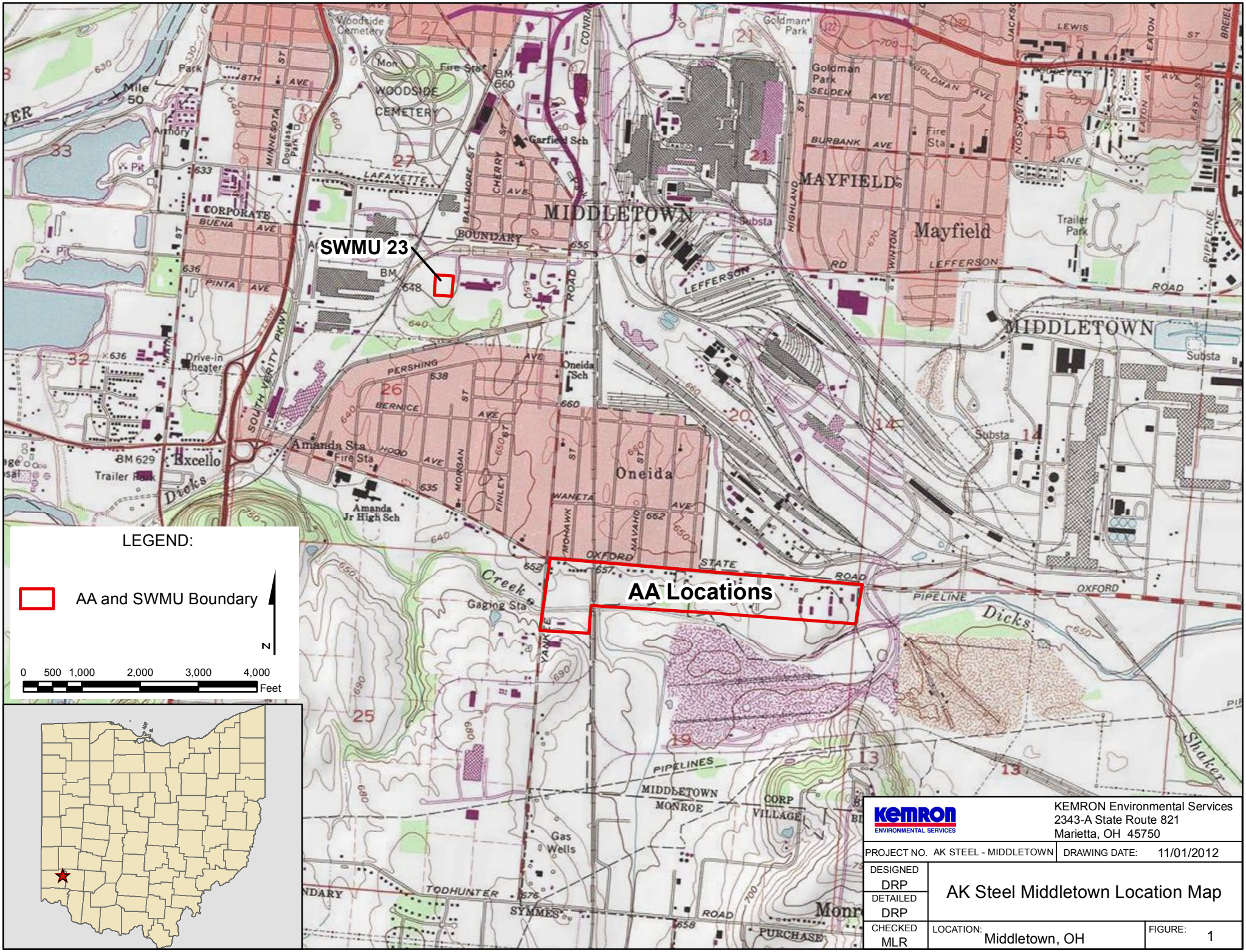
(X) Not applicable.
NR: Not Reported
Source: U.S. Census

Table 4. Proposed Public Involvement Activity Schedule

Public Involvement Activity	Timeline
Toll-free Environmental Information Line	Established in June 2006 for Interim Measures work, and publicly announced in October 2006. Answered weekdays 8:30 a.m. to 4:30 p.m., except for holidays.
Public Relations Coordinator	Established in June 2006 for Interim Measures work, and publicly announced in October 2006. Available weekdays 8:30 a.m. to 4:30 p.m., except for holidays.
Information Repository	Will be established following approval of this plan. Updated monthly as work plans, fact sheets, etc. are approved or reviewed by USEPA, et al.
Mailing List	Prepared in October 2006 for Interim Measures work; contains over 700 entries, including residents, businesses, churches and key stakeholders. Will be kept updated.
Letters	Will be issued as necessary for public meetings, summary of past and current site investigation activities, beginning and end of various phases of work, completion of cleanup, etc. Letters will be distributed to the public via the mailing list.
Press Releases or Fact Sheets	Will be issued for public meetings, the beginning of investigative work and, following completion of the investigative work,
Public Input	To be reported monthly, as part of the Monthly Progress Report to USEPA, et al.
Public Meetings and Availability Sessions AK Steel	If necessary, within 30 days of final approval of the RFI Work Plan.

RFI: Resource Conservation and Recovery Act (RCRA) Facility Investigation
USEPA: U.S. Environmental Protection Agency

FIGURES



KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO.	AK STEEL - MIDDLETOWN	DRAWING DATE:	11/01/2012
DESIGNED	DRP	AK Steel Middletown Location Map	
DETAILED	DRP		
CHECKED	MLR	LOCATION: Middletown, OH	FIGURE: 1

KEMRON ENVIRONMENTAL SERVICES

KEMRON Environmental Services
2343-A State Route 821
Marietta, OH 45750

PROJECT NO. AK STEEL - MIDDLETOWN		DRAWING DATE: 11/01/2012	
DESIGNED DRP	General Location Map; SWMU 23 and Additional Areas		
DETAILED DRP			
CHECKED MLR	LOCATION: Middletown, OH	FIGURE: 2	

Attachment 1

Example Letter

AK Steel Corporation
9227 Centre Pointe Drive
West Chesler, Ohio *45069*

RECEIVED

JUN 20 2012

KEMRON Environmental Services, Inc.

June 15, 2012

To Our Neighbors and Middletown-Area Community:

Enclosed is Fact Sheet No. 14, which provides an overview of the upcoming Year 2 Dicks Creek remediation work. Equipment mobilization and preparation of the staging area is expected to begin in late June, with cleanup work anticipated to occur from June through November 2012.

If you have any questions about the Dicks Creek cleanup, please call our toll-free environmental information line at 1-866-902-4AKS (4257) or visit the Information Repository housed in the MidPointe Middletown Library.

AK Steel appreciates your ongoing patience, support and cooperation with the cleanup.

Sincerely,

Barry L. Racey
Director, Government and Public Relations

Enclosure: Fact Sheet No. 14

Attachment 2

Example Fact Sheet

AK Steel

Middletown Works
Middletown, Ohio

FACT SHEET No. 14

Dicks Creek Year 2 Remediation

June 2012

Introduction

AK Steel completed the Year 1 remediation work in 2010. This included work on Monroe Ditch, a portion of Dicks Creek Reach 1, and installation of a groundwater and free product containment system extending about 500 feet along Monroe Ditch.

As additional information was required to be collected as part of the permitting process for the in-stream work, no work was performed in 2011. AK Steel has now received approval to begin the Year 2 remediation work. This year's work will include the remainder of Reach 1 and a portion of Reach 2 as shown on the map below.

Year 2 Remediation & Restoration

The Year 2 work is expected to begin in June 2012 with the mobilization of equipment and preparation of the related staging area. The actual remediation should occur from June through fall 2012. This work is formally outlined in the **Dicks Creek Year 2 Sediment and Floodplain Soil Remediation Design Document,** which was approved by the USEPA in 2011, after consultation with Ohio EPA and Sierra Club.

In summary, the Year 2 remediation will include:

Excavation and proper disposal of floodplain soil from Dicks Creek that exceeds the floodplain soil cleanup goal ("Action Lever) for PCBs;

Excavation and proper disposal of creek sediment and other materials in Reach 1 that exceed the PCB Action Level;

Removal of all material sand-size or finer from the channel of Dicks Creek Reach 2; and

Remediation of disturbed floodplain areas in Reach 2.

The cleanup work will be done "in the dry" by damming the creek and pumping the water around the work area, as it was done in Year 1. Excavation equipment will then dig up the contaminated material for off-site disposal, as shown on the photo on page 2. The excavated material with lower PCB concentrations will be sent to the Rumpke landfill in Colerain Township, Ohio, whereas the material with higher PCB concentrations will go to Heritage Environmental landfill near Roachdale, Indiana.

Map of Dick's Creek Remediation Areas

A portion of the 2012 work will occur on either side of the Yankee Road bridge. The equipment staging area will be located on the west side of Yankee Road, adjacent to the bridge (near the intersection of Yankee and Oxford State Roads). The City of Middletown is also doing some road work in this area over the summer. AK Steel is coordinating its work schedule and activities with the City to minimize traffic impacts to the extent possible.

Restoration

Following remediation, the creek and adjacent floodplain will be restored with clean soil and fill materials, and re-planted with native vegetation to reduce erosion and improve fish and wildlife habitat to the maximum extent possible. The area will be restored to equal to or better than pre-remediation conditions. This will likely be completed in November. An example of the restoration from the Year 1 cleanup is shown in the second photo below.

Above: Dredging work in progress along Dicks Creek in 2010.

Below: The same general area of Dicks Creek shown above post dredging and restoration. This photo was taken in May 2012. (The Sun Coke facility is visible in the background.)

Temporary Inconveniences & Safety Reminders

Increased construction traffic may cause delays in the vicinity of the Yankee and Oxford State Road intersection. Please follow posted speed limits. Plan travel time accordingly and use alternate routes, if possible.

- Children are often curious about construction. Please keep them away from the site, as you would any construction site.

- Increased noise from equipment operations along the creek is expected during daylight hours.

Year 3 Schedule

The remaining portion of Dicks Creek, where the Year 2 work ends in Reach 2, will be cleaned up in Year 3, which is expected to occur in 2013. The permit AK Steel just received covers the Year 2 and 3 work; therefore, the company does not anticipate any future delays.

Additional information is available from the following sources:

Interim Measures
Public Relations Coordinator

Bethany Dale
ETC, Inc.
4000 Executive Park Drive, Suite 400
Cincinnati, Ohio 45241
Email: bethany.dale@etc-online.com
Fax: 513-772-7904
Toll-Free Environmental Information Line:
1-866-902-4257 (4AKS)

AK Steel
Information Repository

MidPointe Middletown Library
125 S. Broad Street
Middletown, Ohio 45044
Hours of operation:
Monday - Thursday: 9:00 am – 9:00 pm
Friday: 9:00 am – 7:00 pm
Saturday: 9:00 am – 5:00 pm
Sunday: 1:00 pm – 5:00 pm

Attachment 3

Example Press Release

News Release

Contact: Media - Alan H. McCoy, Vice President, Government and Public Relations (513) 425-2826

AK Steel To Host Public Meeting On Dicks Creek Cleanup Progress And Plans

WEST CHESTER, OH, February 19, 2009 – AK Steel will conduct a public meeting on Thursday, March 5, 2009 to update the Middletown-area community about the ongoing cleanup of Dicks Creek. The meeting will be held from 6:30 p.m. to 7:30 p.m. in the Community Room at the Middletown Public Library, 125 S. Broad Street. Those with questions about the meeting may call the Dicks Creek toll-free environmental information line at 866-902-4257.

Under an agreement reached between AK Steel, the U.S. EPA, Ohio EPA, Sierra Club and the Natural Resources Defense Council, AK Steel is removing PCB-contaminated sediment and soil from affected areas of the creek, and preventing PCBs from reaching the creek. This work began in 2007. Additional information regarding the cleanup of Dicks Creek is available at the AK Steel Information Repository, located at the Middletown Public Library.

###

ATTACHMENT E

PHOTOGRAPHS SWMU-23 EXISTING SITE CONDITIONS

Photo 1 - Access to SWMU 23 Area

Photo 2 – Access to SWMU 23 Area

Photo 3 – Access to SWMU 23

Photo 4 - Northeast of SWMU 23

Photo 5 - Gated access to SWMU 23

Photo 6 - Gated access toward SWMU 23

Photo 7 - Northeast of SWMU 23

Photo 8 – Northeast of SWMU 23

Photo 9 - Interior SWMU 23

Photo 10 - View through Gate toward SWMU 23

Photo 11 - Interior of SWMU 23

ATTACHMENT F

PHOTOGRAPHS ADDITIONAL AREAS
AA02 – AA09 EXISTING SITE CONDITIONS

Photo 1 - AA02 View to the Northeast

Photo 2 - AA02 View to the North

Photo 3 - AA04 View to the North

Photo 4 - AA04 View to the North

Photo 5 - AA09 View to the East

Photo 6 - AA03 View to the Northwest

Photo 7 - AA07 View to the North

Photo 8 - AA05 View to the North

Photo 9 - AA05 View to the Northwest

Photo 10 - AA05 View to the North

Photo 11 - AA06 View to the South

Photo 12 - AA06 View to the North

ATTACHMENT G

HEALTH AND SAFETY PLAN

HEALTH AND SAFETY PLAN

FOR

RCRA FACILITY INVESTIGATION OF ADDITIONAL AREAS 02 THROUGH 09 AND SWMU 23

AT THE

AK STEEL MIDDLETOWN WORKS
MIDDLETOWN, OHIO

PREPARED FOR:

AK Steel Corporation
9227 Centre Pointe Drive
West Chester, OH 45069

PREPARED BY

2343-A State Route 821
Marietta, OH 45750

November 2012

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Acronyms

Acronym	Definition
AAs	Additional Areas
ACGIH	American Conference of Governmental Industrial Hygienists
APR	Air Purifying Respirator
BBP	Bloodborne Pathogens
CFR	Code Of Federal Regulations
CGI	Combustible Gas Indicator
CMS	Corrective Measures Studies
CPR	Cardiopulmonary Resuscitation
CRZ	Contamination Reduction Zone
EPA	Environmental Protection Agency
EZ	Exclusion Zone
H&S	Health and Safety
HASP	Health and Safety Plan
HSC	Health and Safety Coordinator
HSM	Health and Safety Manager
HSO	Health and Safety Officer
HSP	Health and Safety Plan
HWMU	Hazardous Waste Management Unit
KEMRON	KEMRON Environmental Services, Inc.
LEL	Lower Exposure Limit
PCBs	Polychlorinated Biphenyls
PEL	Permissible Exposure Limits
PID	Photoionization Detector
PM	Project Manager
PMP	Project Management Plan
PPE	Personal Protective Equipment
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
SSO	Site Safety Officer
SWMU	Solid Waste Management Unit
TCL	Target Compound List
USEPA	United States Environmental Protection Agency
VOCs	Volatile Organic Compounds
WWTP	Waste Water Treatment Plant

SECTION 1.0 INTRODUCTION

This Health and Safety Plan (HSP) has been developed to minimize potential risk to personnel who will perform Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) tasks at Solid Waste Management Unit (SWMU) 23 and Additional Areas (AAs) 02 through 09 at the AK Middletown Works Site (Site) in Middletown. Tasks to be performed include drilling, soil and groundwater sampling, and surveying.

The provisions of this plan apply to all contractor and subcontractor personnel who will potentially be exposed to safety and/or health hazards during the performance of site activities.

This HSP has been written to comply with the requirements of the Occupational Safety and Health Administration's (OSHA's) Hazardous Waste Operations and Emergency Response Standard (29 CFR 1910.120), the KEMRON Health and Safety Policy Manual and AK Steel health and safety requirements for this site (See Section 8.1, below). All activities covered by this HSP must be conducted in complete compliance with this HSP and with all applicable federal, state, and local health and safety regulations. Specific reference is given to the OSHA General Industry Standards (29 CFR 1910), including the Hazardous Waste Operations and Emergency Response Standard and the OSHA Construction Industry Standards (29 CFR 1926). Personnel covered by this HSP who cannot or will not comply with these requirements will be excluded from site activities by the on-site Project Manager.

SECTION 2.0 SITE DESCRIPTION AND SCOPE OF WORK

2.1 Site Description and Objective

AK Steel is under a Consent Decree to perform Resource Conservation and Recovery Act (RCRA) Corrective Action with the USEPA, Ohio EPA, and others. The Consent Decree became effective on May 15, 2006, and requires AK Steel to implement RCRA Corrective Actions and/or IMs at a number of locations on or surrounding the Middletown facility. RCRA Corrective Actions consist of one or more of the following investigation types: a RCRA Facility Investigation (RFI), IMs and Corrective Measures Studies (CMS), as necessary to evaluate individual site conditions.

The Consent Decree identifies a preliminary list of Hazardous Waste Management Units (HWMUs), Solid Waste Management Units (SWMUs), Areas of Concern (AOCs), Spill areas, and Additional Areas (AAs) of Potential Concern for evaluation under the Consent Decree. The CCR and PMP further refine the sites that require RFI work.

The following areas will be evaluated during performance of work under this HASP:

LOCATION	DESCRIPTION
FORMER COIL PAINT AREA	
SWMU 23	Former Coil Paint Wastewater Treatment Lagoons
ADDITIONAL AREAS OF POTENTIAL CONTAMINATION	
AA02	Former Glenn Cartage property (North Side of DC, East of Yankee Rd)
AA03	Sturgell Property (North Side of DC, W of RR bridge, E of Yankee Rd)
AA04	Back half of properties between G. Cartage and Sturgell Properties (North Side of DC)
AA05	Old Armco Lot, NS DC, North of big meander
AA06	Former Burridge Machine Shop Property, North Side DC (now a dairy outlet)
AA07	Former Cecil Osburn Lot (located between Station 12-18, North Side of DC)
AA08	Pipeline Fill, Stations 32-36, along Oxford S.R., N of Outfall 002
AA09	Former Orman's Welding Property

SWMU 23 is an inactive site, which contained three (3) unlined wastewater lagoons/ponds associated with the former Coil Paint Wastewater Treatment Plant (WWTP). The unlined ponds were in operation from 1971 to 1978, and installed in a sand and gravel area west of the Coil Paint Building. Two (2) of the ponds were used to remove suspended solids from the Coil Paint wastewater stream. The third pond was used to dry the solids from the other two ponds, where the water from dewatering the solids was allowed to percolate through sand and gravel bed into

the underlying groundwater. During operation, wastewater containing metals associated with the Coil Paint operations were discharged into the ponds. The ponds were backfilled in the mid-1980s and capped with a vegetated soil cover.

Evaluation information available currently indicates that the ponds were unlined, and that there is an identified release potential for metal constituents and Volatile Organic Compounds (VOCs) into the environment from historical operations. Local groundwater flow and monitoring data from nearby monitoring wells do not currently indicate detectable levels of VOCs.

Additional Areas AA02 through AA09 are characterized as parcels where dredged spoils from previous channelization of Dicks Creek may have been placed, potentially creating an unacceptable PCB exposure risk to human and/or ecological receptors. There is some potential that if dredged materials were placed in these areas, that the spoil materials may contain Polychlorinated Biphenyls (PCBs).

The key objectives of the RFI of Additional Areas AA02 through AA09, and SWMU 23 are:

- Characterize the nature and extent of contamination both within and migrating beyond the boundaries of the subject parcels of the RFI;
- Gather environmental data necessary to support an assessment of risk to human health and the environment posed by any identified releases of contaminants;
- As appropriate, identify, develop, evaluate, and recommend appropriate corrective measures alternatives to protect human health and the environment.

2.2 Scope

In accordance with the requirements of the Consent Decree and Work Plan, the following tasks may be performed during implementation of the Work Plan:

1. Soil Boring Installation
2. Soil Sampling
3. Groundwater Monitoring Well Installation
4. Groundwater Sampling
5. Surveying

SECTION 3.0 HAZARD ASSESSMENT

This section addresses the potential chemical, physical, and biological hazards involved with the work activities of this project. The hazards for each task are listed in Table 3.1, and the protective measures are listed in Table 5.2, and discussed in the body of the HSP.

3.1 Task-Specific Hazard Analysis

The following table indicates potential hazards involved with each, specific task:

Table 3-1 Task Specific Hazards

Task	Chemical Exposure	Slips, Trips, Falls	Mechanical Trauma	Thermal Stress	Noise	Plant/Animal Hazards
Mobilization		√	√	√	√	
Soil Boring Installation – using sonic drilling	√	√	√	√	√	√
Soil Sampling	√	√	√	√	√	√
Groundwater Monitoring Well Installation and Development	√	√	√	√	√	√
Groundwater Sampling	√	√	√	√		√
Surveying		√		√		√
Demobilization		√	√	√	√	

3.2 Hazards Associated with the Materials of Concern

Potential materials of concern on this site are Target Compound List (TCL) Volatile Organic Compounds (VOCs), RCRA Metals, and Polychlorinated Biphenyls (PCBs). Although these are non-specific materials, the constituents of primary importance based upon exposure standards in each group are discussed below, along with their hazardous properties, are:

- VOCs (benzene and vinyl chloride)
- RCRA Metals (arsenic, cadmium, chromium, mercury, and lead)
- PCBs - Arochlors

Table 3-2 Hazardous Properties of Materials of Concern

Compound Name	Ionization Potential (eV)	PEL (8hr TWA)	TLV (8hr TWA)	Affected Media
Volatile Organic Compounds (VOCs)				
Acetone	9.69	1000 ppm (2400 mg/m^3)	500 ppm	Soil / Groundwater
Benzene	9.24	1 ppm	0.5 ppm	Soil / Groundwater
Bromobenzene	8.98	NE	NE	Soil / Groundwater
Bromochloromethane	10.77	200 ppm (1050 mg/m^3)	200 ppm	Soil / Groundwater
Bromodichloromethane (Dichlorobromomethane)	10.88	NE	NE	Soil / Groundwater
Bromoform	10.51	0.5 ppm (5 mg/m^3)	0.5 ppm	Soil / Groundwater
Bromomethane (Methyl bromide)	10.53	20 ppm (80 mg/m^3)	1 ppm	Soil / Groundwater
2-Butanone (MEK)	9.53	200 ppm (590 mg/m^3)	200 ppm	Soil / Groundwater
tert-Butylbenzene	8.68	NE	NE	Soil / Groundwater
sec-Butylbenzene	8.68	NE	NE	Soil / Groundwater
n-Butylbenzene	8.69	NE	NE	Soil / Groundwater
Carbon disulfide	10.08	20 ppm	10 ppm	Soil / Groundwater
Carbon tetrachloride	11.47	10 ppm	5 ppm	Soil / Groundwater
Chlorobenzene	9.07	75 ppm	10 ppm	Soil / Groundwater
Chloroethane	10.97	1000 ppm	100 ppm	Soil / Groundwater
Chloroform	11.42	50 ppm (240 mg/m^3)	10 ppm	Soil / Groundwater
Chloromethane (Methyl chloride)	11.28	100 ppm	50 ppm	Soil / Groundwater
4-Chlorotoluene	8.69	NE	NE	Soil / Groundwater
2-Chlorotoluene	8.83	NE	50 ppm	Soil / Groundwater
Dibromochloromethane (Chlorodibromomethane)	10.59	NE	NE	Soil / Groundwater
Dibromomethane	10.19	NE	NE	Soil / Groundwater
1,2-Dichlorobenzene	9.07	50 ppm (300 mg/m^3)	25 ppm	Soil / Groundwater
1,4-Dichlorobenzene	8.94	75 ppm (450 mg/m^3)	10 ppm	Soil / Groundwater
1,3-Dichlorobenzene	9.12	NE	NE	Soil / Groundwater
Dichlorodifluoromethane	11.75	1000 ppm (4950 mg/m^3)	1000 ppm	Soil / Groundwater
1,1-Dichloroethane	11.06	100 ppm (400 mg/m^3)	100 ppm	Soil / Groundwater
1,2-Dichloroethane	11.04	50 ppm	10 ppm	Soil / Groundwater
cis-1,2-Dichloroethene	9.65	200 ppm (790 mg/m^3)	200 ppm	Soil / Groundwater

Compound Name	Ionization Potential (eV)	PEL (8hr TWA)	TLV (8hr TWA)	Affected Media
trans-1,2-Dichloroethene	9.66	200 ppm (790 mg/m^3)	200 ppm	Soil / Groundwater
1,1-Dichloroethene	9.60	NE	5 ppm	Soil / Groundwater
1,3-Dichloropropane	10.85	NE	NE	Soil / Groundwater
2,2-Dichloropropane	10.87	NE	NE	Soil / Groundwater
1,2-Dichloropropane	10.87	75 ppm (350 mg/m^3)	10 ppm	Soil / Groundwater
1,1-Dichloropropene	NR	200 ppm (260 mg/m^3)	200 ppm	Soil / Groundwater
cis-1,3-Dichloropropene	NR	NE	NE	Soil / Groundwater
trans-1,3-Dichloropropene	NR	NE	NE	Soil / Groundwater
Ethylbenzene	8.76	100 ppm (435 mg/m^3)	100 ppm	Soil / Groundwater
Hexachlorobutadiene	NR	NE	0.02 ppm	Soil / Groundwater
n-Hexane	10.18	500 ppm (1800 mg/m^3)	50 ppm	Soil / Groundwater
2-Hexanone	9.34	100 ppm (410 mg/m^3)	5 ppm	Soil / Groundwater
Isopropylbenzene (Cumene)	8.75	50 ppm (245 mg/m^3)	50 ppm	Soil / Groundwater
p-Isopropyltoluene	NR	NE	NE	Soil / Groundwater
Methyl tert-butyl ether	10.00	NE	50 ppm	Soil / Groundwater
Methylene chloride	11.35	25 ppm	50 ppm	Soil / Groundwater
4-Methyl-2-pentanone (MIBK)	9.32	100 ppm (410 mg/m^3)	50 ppm	Soil / Groundwater
n-Propylbenzene	8.72	NE	NE	Soil / Groundwater
Styrene	8.47	100 ppm	20 ppm	Soil / Groundwater
1,1,1,2-Tetrachloroethane	NR	NE	NE	Soil / Groundwater
1,1,2,2-Tetrachloroethane	11.10	5 ppm (35 mg/m^3)	1 ppm	Soil / Groundwater
Tetrachloroethene	9.32	100 ppm	25 ppm	Soil / Groundwater
Toluene	8.82	200 ppm	50 ppm	Soil / Groundwater
1,2,4-Trichlorobenzene	9.37	NE	5 ppm	Soil / Groundwater
1,1,1-Trichloroethane	11.25	350 ppm (1900 mg/m^3)	350 ppm	Soil / Groundwater
1,1,2-Trichloroethane	NR	10 ppm (45 mg/m^3)	10 ppm	Soil / Groundwater
Trichloroethene	9.45	100 ppm	50 ppm	Soil / Groundwater
Trichlorofluoromethane	NR	1000 ppm (5600 mg/m^3)	1000 ppm	Soil / Groundwater
1,2,4-Trimethylbenzene	8.27	NE	25 ppm	Soil / Groundwater
1,3,5-Trimethylbenzene	8.39	NE	25 ppm	Soil / Groundwater
Vinyl Acetate	9.19	NE	10 ppm	Soil / Groundwater
Vinyl chloride	10.00	1 ppm	1 ppm	Soil / Groundwater

Compound Name	Ionization Potential (eV)	PEL (8hr TWA)	TLV (8hr TWA)	Affected Media
m-Xylene	8.56	100 ppm (435 mg/m^3)	100 ppm	Soil / Groundwater
o-Xylene	8.56	100 ppm (435 mg/m^3)	100 ppm	Soil / Groundwater
p-Xylene	8.45	100 ppm (435 mg/m^3)	100 ppm	Soil / Groundwater
Polychlorinated Biphenyls (PBCs)				
Aroclor-1016	NA	1 mg/m^3 for 42% chlorine 0.5 mg/m^3 for 54% chlorine	1 mg/m^3 for 42% chlorine 0.5 mg/m^3 for 54% chlorine	Soil
Aroclor-1221	NA			Soil
Aroclor-1232	NA			Soil
Aroclor-1242	NA			Soil
Aroclor-1248	NA			Soil
Aroclor-1254	NA			Soil
Aroclor-1260	NA			Soil
Aroclor-1268	NA			Soil
RCRA Metals				
Arsenic	NA	0.010 mg/m^3	0.010 mg/m^3	Soil / Groundwater
Barium	NA	NE	0.5 mg/m^3	Soil / Groundwater
Cadmium	NA	0.005 mg/m^3	0.002 mg/m^3	Soil / Groundwater
Chromium	NA	1 mg/m^3	0.5 mg/m^3	Soil / Groundwater
Mercury	NA	0.1 mg/m^3	0.025 mg/m^3	Soil / Groundwater
Lead	NA	0.050 mg/m^3	0.050 mg/m^3	Soil / Groundwater
Selenium	NA	0.2 mg/m^3	0.2 mg/m^3	Soil / Groundwater
Silver	NA	0.01 mg/m^3	0.1 mg/m^3	Soil / Groundwater

3.3 Volatile Organic Compounds (VOCs)

Benzene, and vinyl chloride are examples of volatile organic compounds (VOCs). They are colorless, aromatic liquids. In moderate to heavy exposures, they may produce headache, eye and throat irritation, narcosis, and anesthesia. Excessive exposures can also produce liver and kidney damage, and bone marrow depression. Benzene is of more concern chronically. Benzene is a known carcinogen, associated with leukemia and aplastic anemia in chronic exposures and exposures should be avoided. Vinyl chloride is a known carcinogen associated with liver cancer. Exposure to vinyl chloride should be avoided and inhalation of vapors may cause dizziness, drowsiness, and headache. Unconsciousness For this reason, Drager tubes, or other specific monitoring methods, may be used to verify that benzene is not present at concentrations greater than 1 ppm.

The OSHA PEL for benzene is 1 ppm, with a STEL of 5 ppm. Its olfactory detection level is 5 ppm. Benzene is very flammable, with a flash point of -11 °C.

The OSHA PEL for vinyl chloride is 1 ppm, with a STEL of 5 ppm. Its olfactory detection level is 5 ppm. Vinyl chloride is an extremely flammable gas with a flashpoint of -77° C.

3.4 Polychlorinated Biphenyls (PCBs)

PCBs are mixtures of 209 individual compounds. In the United States, mixtures of PCBs carried the trademark "Aroclor" followed by a four-digit number that indicated the percent chlorine content by weight. In analyses of environmental samples from the site, PCBs have been quantified as a variety of Aroclors, primarily Aroclor 1248 and Aroclor 1254. PCBs are generally clear, colorless to light yellow, viscous liquids or solids with a mild hydrocarbon odor. They have a low vapor pressure, and thus a low volatility. The low volatility of PCBs makes the release of potentially significant levels of airborne vapor extremely unlikely, so that the most probable inhalation exposure would be to PCB-containing dusts generated during excavation.

The PCB-containing dust action level that will be followed during site work is based on the constituent (i.e., Aroclor 1254) with the lowest TLV listed in this HASP (see Table 1), the maximum total PCB concentration determined from on-site soil and sediment samples, and a safety factor based on the adequacy of site characterization. Using a maximum soil/sediment PCB concentration of 570 mg/kg and a safety factor of 10, an action level for PCB-containing dust is calculated using the following formula:

$$\textit{Action Level} = \frac{\textit{EL (mg/ m}^3\textit{) (10}^6\textit{)}}{\textit{Soil Conc. (mg/kg) (Safety Factor)}}$$

where: EL = exposure limit (Aroclor 1254 TLV)
Safety Factor = 10 (conservative assumption)

Substituting the values into the formula yields the following exposure limit:

$$\frac{\textit{0.5 mg/ m}^3\ (10^6)}{\textit{570 mg/kg (10)}} = \textit{88 mg/m}^3$$

From this equation, total dust concentrations in air would need to reach 88 mg/m^3 to create an airborne concentration of PCBs that would equal the TLV. Since the air monitoring equipment will measure respirable dust particles, it has been assumed, based on prior experience, approximately 50 percent of dust generated would be in the respirable range (less than ten microns in diameter). Therefore, the calculated exposure limit is divided by 2 to yield a PCB-containing dust action level of 44 mg/m^3. Dust generation of this magnitude would not occur under the type of work anticipated to be conducted during the RFI (e.g., drilling soil borings and installing monitoring wells). This action level is more than four times higher than the total nuisance particulate standard of the ACGIH (TLV-TWA = 10 mg/m^3); therefore, the nuisance particulate standard will be adequate to address any PCB-containing dust inhalation exposure. This action level is expected to be conservative for both workers and residents, especially given the short term nature of the potential exposure.

3.5 RCRA Metals

These metals include lead, arsenic, barium, cadmium, chromium, mercury, lead, selenium and silver. Even though the samples will be analyzed for these metals, the initial hazard analysis indicates that concentrations of metals in soil and water are expected to be below those concentrations that would pose a hazard to workers. For instance, lead in soil at 1000 mg/kg would require 50 mg/m^3 of total dust (a large amount of dust) to present an airborne lead hazard to workers. Nevertheless, care will be undertaken to prevent contaminant contact by wearing gloves and avoiding dust. Heavy metals are known to cause neurologic effects (lead, mercury), kidney damage (cadmium), and respiratory damage (arsenic, cadmium). Oral and respiratory exposures should be minimized. The PEL for inorganic lead is 0.01 mg/m^3.

3.6 Physical Hazards

Potential physical hazards associated with this project include the use of heavy equipment to construct borings (drill rigs), and to perform test-pitting (backhoe). Energized heavy equipment poses potential trauma and crushing hazards to employees. Inclement weather may quickly occur in the area. Removing soil samples from the split spoons can present ergonomic hazards.

Safety precautions for well installation hazards are addressed in Section 6. These activities must comply with the health and safety practices specified in 29 CFR 1926 (the OSHA Construction Standards).

Noise may also be a concern around energized equipment. If speech becomes difficult to understand at 5 feet, hearing protection will be worn.

Specific precautions for drilling and excavating are listed in 6 below.

3.6.1 Lifting Hazards and Back Safety

Using the proper techniques to lift and to move heavy pieces of equipment (e.g., generators or drums of investigation-derived wastes) is important to reduce the potential for back injury. The following precautions should be implemented when lifting or moving heavy objects.

- Use mechanical devices to move objects, such as drums of investigation derived wastes or generators, that are too heavy to be moved manually
- If mechanical devices are not available, ask another person to assist you.
- Bend at the knees, not the waist. Let your legs do the lifting.
- Do not twist while lifting.
- Bring the load as close to you as possible before lifting.
- Be sure the path you are taking while carrying a heavy object is free of obstructions and slip, trip and fall hazards.

3.6.2 Insects

Ticks are bloodsuckers, attaching themselves to warm-blooded vertebrates to feed. Deer ticks are the most common carriers of Lyme Disease, a bacterial infection that is transmitted to humans through the bite of the tick.

Personnel should carefully inspect themselves each day for the presence of ticks or any rashes.

This is important since prompt removal of the tick can prevent disease transmission. Female deer ticks are about one-quarter inch in length and are black and brick red in color. Males are smaller and all black.

Removal of the tick is important in that the tick should not be crushed and care must be taken so that the head is also removed. If the head is not completely removed or if the tick is allowed to remain for days feeding on human blood, a condition known as tick paralysis can develop, which is due to a neurotoxin that the tick apparently injects. This neurotoxin acts upon the spinal cord causing lack of coordination, weakness and paralysis.

One characteristic symptom of Lyme Disease is a bulls-eye rash that develops around the bite site. The rash appears in about 60-80% of all Lyme disease cases. Contact your Regional Health and Safety Manager (RHSM) immediately if you develop such a rash.

Tick season lasts from April through October; peak season is May through July. Wear light-colored clothing (easier to spot ticks) with long sleeves and make sure that shirts are tucked into pants and pants are tucked into socks or boots. Ticks have a tendency to crawl upwards. These procedures will make it more difficult for a tick to reach your skin.

Studies have determined that repellants containing DEET as a main ingredient are most effective against mosquitoes and ticks. DEET can be directly applied to the exposed skin of adults and/or clothing. Permanone® is another repellent however, it can only be directly applied to clothing. These repellants are also effective against chiggers, which are very common on this site.

3.6.3 Poisonous Plants

When working at sites that are heavily vegetated or located near wetlands, employees should be aware of the possible presence of poisonous plants and insects.

Poison oak and poison ivy occur in the eastern United States. Poison Ivy has its leaves divided into three leaflets; It may have white, berries, and in the Fall, the leaves may be red.

The leaves, roots, stems and fruit of poison ivy and poison oak contain an oil called urushiol. Contact with the irritating oil causes an intensely itching skin rash and characteristic blister-like lesions. The oil can be transmitted on soot particles when burned and may be carried on the fur of animals, equipment and apparel.

Proper identification of these plants is the key to preventing contact and subsequent dermatitis. Wear long sleeves and pants when working in wooded areas. In areas of known infestation, wear Tyvek coveralls and gloves. Oils are easily transferred from one surface to another. Wash all contaminated clothing and equipment promptly.

If you come in contact with these poisonous plants, wash all exposed areas immediately with cool water to remove the oils. Some commercial products such as Tecnu's Poison Oak-n-Ivy Cleanser claim to further help with the removal of oils.

3.6.4 Inclement Weather

The project locations may be subject to severe weather conditions including severe thunderstorms, and tornadoes. A weather radio will be on-site at these project locations for the duration of the project to monitor severe weather. If severe weather warnings are issued for the site location or surrounding county, work will cease. The site will be secured to the extent possible and employees will seek immediate refuge as directed by the On-Site Health and Safety Coordinator (HSC). Minimally, all drill rig masts, crane booms and the like shall be lowered, especially in the event of lightning.

3.6.5 Pressure Washing / Steam Cleaning

Pressure washing and steam cleaning of equipment present potential trauma hazards if a worker is hit by the steam or water blast, or if pressurized hoses / equipment get loose in the work area. During steam cleaning / pressure washing, employees other than the operator shall remain clear of the area. Anyone who must remain nearby will pre-arrange communication with the operator to avoid accidental spraying. Cleaning will not be directed toward areas in which workers are located. Personal Protective Equipment (PPE) will be worn by the operator which is protective from burns, trauma, and overspray.

3.6.6 Traffic Hazards

The site may present the hazards of incoming and outgoing traffic. When working in these traffic areas, the following precautions should be implemented.

- Wear a traffic safety vest or other high visibility garment. If work is being performed at dawn, dusk or evening the vests must have reflective tape. (If working around rotating equipment, loose clothing, including traffic vests, must be restrained or removed. Drillers and others working in such tasks may wear fluorescent clothing or reflective tape in order to avoid loose traffic vests.)
- Set up traffic cones or "Men at Work" signs in front of the work area.

Investigative activities may occur along or in a public roadway. If such work is scheduled, the Project Manager (PM) will contact the local police or authorities to determine if a traffic control plan must be filed or if a police detail is required.

3.6.7 Railroad Right-of-way Work

The railroad will be contacted prior to work within the right-of-way (ROW) of the rail lines. The following will be required within the ROW:

- The right-of-way is 100 feet (50 feet each side from the center of the track) along the length of the track.
- No equipment will be put within 25 feet of the track; to install wells in the ROW requires permission from the rail company.
- Any wells to be installed must be flush mounts.

- There may be other utilities within the ROW, which must be cleared for any invasive work.
- A spotter will be required to monitor all our activities while on the ROW.

Minimum PPE required within the ROW includes a hard hat, safety boots, safety glasses, and a high visibility traffic vest.

Review and comply with the applicable portions of AK Steel's General Safety Order # 3, working on or About RR Tracks.

SECTION 4.0 AIR AND RADIOLOGICAL MONITORING

4.1 Radiological Hazards

There is no information to indicate radiological hazards at this site.

4.2 Gases and Vapors

Daily airborne contaminant monitoring for VOCs (as benzene) will be performed on an hourly basis using a photoionization detector (PID) calibrated with isobutylene, until data establish that action levels for respiratory protection are not exceeded. There is no real-time monitor for PCBs or for the metal constituents which have been identified at this Site. A MiniRam airborne dust monitor will be used to evaluate total levels of airborne dust, since airborne particulates are the most likely exposure route for PCBs and metals.

A PID, such as a RaeSystems MiniRae 2000 ® equipped with a 10.6 eV lamp and calibrated to isobutylene, will be used to screen the breathing zone of employees during the advancement of soil borings, installation of monitoring wells, excavation of test pits and soil/groundwater sampling activities. If breathing zone concentrations are sustained (15 minutes) at 5 units above background, Level C respiratory protection, as described in Section 7.2, will be donned.

Excessive concentrations of flammable vapors are not anticipated in this work. However, should the PID read extremely high levels of vapors (1000 to 2000 units) in the work area, work must be stopped or a combustible gas indicator (CGI) must be acquired to assure concentrations are not exceeding 5.0% of the LEL. If there is ANY knowledge that saturated hydrocarbons may be encountered in the borings, a CGI is highly recommended for this work.

Since there is no monitor for PCBs or metals, dust levels must be controlled to minimize any potential exposure to these non-volatile substances.

SECTION 5.0 PERSONAL PROTECTIVE EQUIPMENT (PPE)

5.1 Respiratory Protection

Table 5-1 Respiratory Protection

	PID / MiniRam Units	Respirator
Level D	< 5, or <8 mg/m^3 dust	No respiratory protection.
Level C, ½ Mask	> 5, or 8 mg/m^3 dust	Half Mask, Air Purifying Respirator (APR), with Combination Cartridges (organic vapor, acid gas, high efficiency particulate).
Level C, Full-face[1].	> 500, or > 8 mg/m^3 dust	Full Face, Air Purifying Respirator (APR), same cartridges.

1. Level B, supplied air respiratory protection is not expected. In the unlikely event that the limitations of Level C, Full-face mask, (1000 units) are exceeded, personnel will retreat and revise the H&S procedures, per the situation.

Drilling, excavating, and other activities creating airborne organic vapors or dust require the use of respiratory protection when the above action levels are exceeded. During periods of respirator use, cartridges are to be changed each day of use, whenever breakthrough is detected, or when dust loading makes breathing difficult, whichever occurs first.

Response

When the PID yields persistent breathing-zone readings at or above the lower action limit, or when significant levels of potentially contaminated dust are being generated, workers in the affected area will don respirators. Air sampling will then continue on a more frequent basis. If readings are persistent at or above the upper limit, workers shall back off from the immediate work area until measured breathing-zone concentrations fall below the lower limit, at which time operations will resume and normal air monitoring will continue. If breathing zone levels do not fall below the upper limit, workers are to leave the work area. If necessary, engineering controls (ventilation, vapor suppression, etc.) will be instituted to maintain vapor concentrations below the upper limit or arrangements will be made to upgrade to supplied air protection.

Airborne contaminant monitoring will be performed using a PID calibrated with isobutylene, and a MiniRam airborne dust monitor, factory calibrated for respirable dust.

5.2 Protective Clothing and Equipment

AK's minimum PPE requirements for the jobsite include: Hard hat, safety glasses with side shields, long pants, long sleeve shirt, Advance Technology Armor (ATA) sleeves shall be worn over long sleeves of shirts, safety toe work boots with metatarsal protection. Task specific PPE are listed in Table 5-2

Table 5-2 Personal Protective Equipment by Task*

PPE Item	Task 1	Task 2	Task 3	Task 4	Task 5	Task 6	Task 7
Hard Hat	√	√	√	√	√	√	√
Safety Glasses		√	√	√	√	√	
Goggles / Face Shield		√[3]	√[3]	√[3]	√[3]		

PPE Item	Task 1	Task 2	Task 3	Task 4	Task 5	Task 6	Task 7
Safety Boots	√	√	√	√	√	√	√
Metatarsal Foot wear	√	√	√	√	√	√	√
Long Sleeves	√	√	√	√	√	√	√
Inner Gloves		√	√	√	√		
Outer Gloves		√	√	√	√		
Work Gloves				√			
Respirator[1]	1	1	1	1	1	1	1
Traffic Vest		√	√	√	√	√	
Ear Plugs/Muffs[2]	2	2	2	2	2	2	2
Sun glasses and sun screen[4]	4	4	4	4	4	4	4
Body Harness/Safety Rope		5				5	

[1]See 5.1
[2]Hearing protection must be worn above 85 decibels or when speech becomes difficult at 5 ft.
[3]If a splash hazard exists, i.e., using a pressure washer, mixing grout, etc.
[4]If necessitated by weather.
[5] If working on steep banks or slopes.

*Tasks

1. Mobilization
2. Soil Boring Installation – using sonic drilling
3. Soil Sampling
4. Groundwater Monitoring Well Installation
5. Groundwater Sampling and Water Level Data Collection
6. Surveying
7. Demobilization

5.3 Engineering Controls

Engineering controls will be implemented when necessary to lower potential concentrations of COCs to acceptable exposure levels. Should airborne dust be above target limits, and where feasible, fine water sprays or other engineering controls, will be used for minimizing dust when required.

5.4 Sanitation

If facilities are not available on site, a portable toilet will be provided adjacent to the contamination reduction zone (CRZ). A hand-wash station will also be located adjacent to the CRZ.

5.5 Spill Prevention Plan/Containment Program

Drums and containers of hazardous substances are not expected to be handled during this project. Therefore, a situation where a spill of hazardous substances would occur, which would warrant a spill containment plan is not anticipated. Drums containing investigation-derived wastes will be segregated from other materials and stored in secure areas for subsequent waste determination and disposal.

5.6 Site Control and Decontamination

Persons working in the exclusion zones (EZs), and who have come in contact with potentially contaminated soil, water, dust, or other materials at the Site will thoroughly decontaminate their equipment and themselves prior to leaving, eating, smoking, or drinking (exceptions include seriously injured employees).

A three basin contamination reduction (CRZ, decontamination zone) station will be established between the Exclusion Zone and the Support Zone. Entrance to, and exit from the EZ will be via the CRZ.

Any contaminated equipment will be left at an equipment drop, or decontaminated in the CRZ. Contaminated boots will be scrubbed clean in the first basin of detergent solution, rinsed clean, followed by a second water rinse; then outer gloves, will be rinsed free of gross contamination, and removed / disposed in the PPE drum. Other disposable PPE, such as Tyvek coveralls will be removed and disposed of, followed by removal and disposal of respirator cartridges. Disposables will be disposed of as contaminated waste, or in the sanitary refuse, if non-hazardous. Respirators, if used, will be cleaned after each use with soap and water or wipe pads, dried, and stored in plastic bags.

Figure HSP-1 provides a site map with proposed site work zones. The actual configuration of the work zones may change slightly once investigative activities begin at the Site.

5.7 Fire Safety

At least one fire extinguisher, rated 10 BC, meeting the requirements of OSHA in 29 CFR 1926 will be on site. Employees who may use extinguishers must be trained in the safe operation and use of portable fire extinguishers.

5.8 Thermal Stress

Investigation may be performed during any month of the year. As such, the hazards associated with heat and cold stress are addressed in this HASP.

5.8.1 Heat Stress

Types of Heat Stress

Heat related problems include **heat rash, fainting, heat cramps, heat exhaustion and heat stroke**. **Heat rash** can occur when sweat isn't allowed to evaporate, leaving the skin wet most of the time and making it subject to irritation. **Fainting** may occur when blood pools to lower parts of the body and as a result, does not return to the heart to be pumped to the brain. Heat related fainting often occurs during activities which require standing erect and immobile in the heat for long periods of time. **Heat cramps** are painful spasms of the muscles due to excessive salt loss associated with profuse sweating. **Heat exhaustion** results from the loss of large amounts of fluid and excessive loss of salt from profuse sweating. The skin will be clammy and moist and the affected individual may exhibit giddiness, nausea and headache. **Heat stroke** occurs when the body's temperature regulatory system has failed. The skin is hot, dry, red and spotted. The affected person may be mentally confused and delirious. Convulsions could occur. EARLY RECOGNITION AND TREATMENT OF HEAT STROKE ARE THE ONLY MEANS OF PREVENTING BRAIN DAMAGE OR DEATH. A person exhibiting signs of heat stroke should be removed from the work area to a shaded area. The person should be soaked with water to promote evaporation. Fan the person's body to increase cooling. Increased body temperature and physical discomfort also promote irritability and a decreased attention to the performance of hazardous tasks.

Early Symptoms of Heat-Related Health Problems:

- decline in task performance
- incoordination
- decline in alertness
- unsteady walk
- reduced vigilance
- excessive fatigue
- muscle cramps
- dizziness

Susceptibility to Heat Stress Increases due to:

- lack of physical fitness
- lack of acclimation
- increased age
- dehydration
- obesity
- drug or alcohol use
- sunburn
- infection

People unaccustomed to heat are particularly susceptible to heat fatigue. First timers in PPE need to gradually adjust to the heat.

The Effect of Personal Protective Equipment
Sweating normally cools the body as moisture is removed from the skin by evaporation. However, the wearing of certain personal protective equipment (PPE), particularly chemical protective coveralls (e.g., Tyvek), reduces the body's ability to evaporate sweat and thereby regulate heat buildup. The body's efforts to maintain an acceptable temperature can therefore become significantly impaired by the wearing of PPE.

Measures to Avoid Heat Stress:
The following guidelines should be adhered to when working in hot environments:

- Establish work-rest cycles (short and frequent are more beneficial than long and seldom).
- Identify a shaded, cool rest area.
- Rotate personnel, alternative job functions.
- Water intake should be equal to the sweat produced. Most workers exposed to hot conditions drink less fluids than needed because of an insufficient thirst. DO NOT DEPEND ON THIRST TO SIGNAL WHEN AND HOW MUCH TO DRINK. For an 8-hour work day, 50 ounces of fluids should be drunk.
- Eat lightly salted foods or drink salted drinks such as Gatorade to replace lost salt.
- Save most strenuous tasks for non-peak heat hours such as the early morning or at night.
- Avoid alcohol during prolonged periods of heat. Alcohol will cause additional dehydration.
- Avoid double shifts and/or overtime.

The implementation and enforcement of the above mentioned measures will be the joint responsibility of the project manager, on-site field coordinator, and health and safety officer. Potable water and fruit juices should be made available each day for the field team.

Heat Stress Monitoring Techniques
Site personnel should regularly monitor their heart rate as an indicator of heat strain by the

following method:

Check radial pulse rates by using fore-and middle fingers and applying light pressure to the pulse in the wrist for one minute at the beginning of each rest cycle. If the pulse rate exceeds 110 beat/minute, shorten the next work cycle by one-third and keep the rest period the same. If, after the next rest period, the pulse rate still exceeds 110 beats/minute, shorten the work cycle again by one-third.

5.8.2 Cold Stress

Types of Cold Stress
Cold injury is classified as either localized, as in frostbite, frostnip or chilblain; or generalized, as in hypothermia. The main factors contributing to cold injury are exposure to humidity and high winds, contact with wetness and inadequate clothing.

The likelihood of developing frostbite occurs when the face or extremities are exposed to a cold wind in addition to cold temperatures. The freezing point of the skin is about 30° F. The fluids around the cells of the body tissue freeze, causing the skin to turn white. This freezing is due to exposure to extremely low temperatures. As wind velocity increases, heat loss is greater and frostbite will occur more rapidly.

Symptoms of Cold Stress
The first symptom of frostbite is usually an uncomfortable sensation of coldness, followed by numbness. There may be a tingling, stinging or aching feeling in the affected area. The most vulnerable parts of the body are the nose, cheeks, ears, fingers and toes.

Symptoms of hypothermia, a condition of abnormally low body temperature, include uncontrollable shivering and sensations of cold. The heartbeat slows and may become irregular, the pulse weakens and the blood pressure changes. Pain in the extremities and severe shivering can be the first warning of dangerous exposure to cold.

Maximum severe shivering develops when the body temperature has fallen to 95° F. This must be taken as a sign of danger and exposure to cold must be immediately terminated. Productive physical and mental work is limited when severe shivering occurs.

Methods to Prevent Cold Stress
When the ambient temperature, or a wind chill equivalent, falls to below 40° F (American Conference of Governmental Industrial Hygienists recommendation), site personnel who must remain outdoors should wear insulated coveralls, insulated boot liners, hard hat helmet liners and insulated hand protection. Wool mittens are more efficient insulators than gloves. Keeping the head covered is very important, since 40% of body heat can be lost when the head is exposed. If it is not necessary to wear a hard hat, a wool knit cap provides the best head protection. A face mask may also be worn.

Persons should dress in several layers rather than one single heavy outer garment. The outer piece of clothing should ideally be wind and water proof. Clothing made of thin cotton fabric or synthetic fabrics such as polypropylene is ideal since it helps to evaporate sweat. Polypropylene is best at wicking away moisture while still retaining its insulating properties. Loosely fitting clothing also aids in sweat evaporation. Denim is not a good protective fabric. It is loosely woven which allows moisture to penetrate. Socks with a high wool content are best. If two pairs of socks

are worn, the inner sock should be smaller and made of cotton, polypropylene or a similar type of synthetic material that wicks away moisture. If clothing becomes wet, it should be taken off immediately and a dry set of clothing put on.

If wind conditions become severe, it may become necessary to shield the work area temporarily. The SSO and the PM will determine if this type of action is necessary. Heated break trailers or a designated area that is heated should be available if work is performed continuously in the cold at temperatures, or equivalent wind chill temperatures, of 20° F.

Dehydration occurs in the cold environment and may increase the susceptibility of the worker to cold injury due to significant change in blood flow to the extremities. Drink plenty of fluids, but limit the intake of caffeine.

SECTION 6.0 PROJECT RESPONSIBILITIES

6.1 Site-Specific Responsibilities

The following personnel are anticipated to work on this project:

- Mary Lou Rochotte, KEMRON Project Manager
- Field Project Manager
- Field support personnel
- Personnel from Drilling Subcontractor
- Personnel from Surveyor Subcontractor

6.2 General Organization and Responsibilities

The organization and responsibilities for implementing safe on-site activities, and more specifically the requirements contained in this HSP, are described below. The implementation of health and safety at this site will be an integrated effort among the KEMRON Project Manager (PM), the Field Project Manager, the Project Health and Safety Officer (Project HSO), Client Personnel (CP), and Subcontractors.

6.3 Project Manager

The PM is the individual who has the primary responsibility for ensuring the overall health and safety of this project. The PM, therefore, has the primary responsibility for ensuring the implementation of the requirements of this HSP. Some of the PM's specific responsibilities include:

- Assuring that all on-site personnel have received a copy of and read this HSP and have completed the HSP sign-off sheet
- Assuring that all personnel have attended a briefing apprising them of the contents of the HSP and site specific hazards prior to performing work on the Site
- Assuring that sufficient PPE, as required by this HSP, is available on site
- Assuring that all subcontractor personnel submit the documentation of employee participation in a medical monitoring program and training program
- Maintaining a high level of health and safety consciousness among employees at the work site
- Maintaining regular communications with the HSO and CP

6.4 Project Health and Safety Manager

The Project HSM is the individual responsible for the preparation, interpretation and modification of this HSP. Corporate Health and Safety Management will provide clarification and interpretation of regulatory and corporate standards as requested by the Project HSM. Modifications to this HSP, which may result in less stringent precautions, cannot be undertaken by the PM or the on-site HSO without the approval of from Project HSM. Specific duties of the Project HSM include:

- Advising the PM and HSC on matters relating to health and safety on this site
- Recommending appropriate PPE and air monitoring instrumentation to protect personnel

from site hazards

- Performing field audits to monitor the effectiveness of this HSP and to assure compliance with it
- Performing personal exposure monitoring where required and where deemed necessary to determine the adequacy of protective measures and PPE specified by this HSP
- Maintaining contact with PM to regularly evaluate site conditions and new information that might require modifications to the HSP
- Working with the PM to ensure that sufficient PPE is available on site
- A daily safety meeting will be conducted prior to commencement of all daily work in order to apprise personnel of the contents of the HSP and the site hazards

6.5 On-Site Health and Safety Coordinator

The Field Project Manager will serve as the On-Site Health and Safety Coordinator (HSC). The Field Project Manager may designate an alternate, properly trained, On-Site HSC; the On-Site HSC will be a member of the project field team. The HSC is responsible for enforcing the requirements of this HSP once on-site work begins. By design, the HSC has the authority to immediately correct all situations where noncompliance with this HSP is noted and to immediately stop work in cases where an immediate danger is perceived. Some of the HSC's specific responsibilities include:

- Procuring and distributing the PPE needed for this project
- Procuring the air monitoring instrumentation required and performing air monitoring
- Verifying that all PPE and health and safety equipment is in good working order
- Setting up and maintaining the personnel decontamination facility
- Notifying the PM and the HSM of all noncompliance situations and immediate danger situations
- Supervising and monitoring the safety performance of all personnel to ensure that required safety and health procedures are followed and correcting any deficiencies
- Conducting accident/incident remediation and preparing accident/incident remediation reports
- Initiating emergency response procedures

6.6 Field Personnel

All client and subcontractor field personnel are responsible for following the health and safety procedures specified in this HSP and for performing their work in a safe and responsible manner. Some of the specific responsibilities of the field personnel are as follows:

- Obtaining a copy of the HSP and reading it in its entirety prior to the start of on-site work. A sign-off sheet will be attached to the HSP and must be signed prior to initiating any site work.
- Bringing forth any questions or concerns regarding the content of the HSP to the PM or the RHSM prior to the start of work

- Reporting all accidents and incidents to the PM
- Complying with the requests of the appointed HSC

SECTION 7.0 TRAINING/MEDICAL SURVEILLANCE/FIRST AID

7.1 Training

All personnel who will be potentially exposed to safety or health hazards during implementation of Work Plan at the Site, must have completed the training and medical surveillance requirements as specified in the OSHA Hazardous Waste Operations and Emergency Response Standard [29 CFR 1910.120(e) and (f)], and as required by AK Steel health and safety policies.

Therefore, such personnel must have completed the 40 hours of initial training or the eight hours of refresher training within the last year.

Managers or supervisors of personnel performing such activities must have completed the specified eight hours of management training in addition to the initial/refresher training.

Site personnel will have received Hazard Communication training, in compliance with 29 CFR 1910.1200, regarding handling or use of flammable liquids, decontamination solutions, samples, preservatives, or other substances brought on site.

In addition, all personnel who will perform any field-related work at the Site must have completed the required AK Steel safety training, and must fully comply with AK H&S requirements for this site. This includes submission and approval of work permits for confined spaces, hot work, and excavation, and other procedures as required. These requirements are reviewed during the AK Steel contractor safety training prior to Site work.

7.2 Medical Surveillance

Site personnel must also have completed and passed, without restrictions, an annual and/or baseline occupational medical surveillance examination within the last year. Employees potentially wearing respirators must have passed a fit-test for the specific respirators utilized, and must have medical approval to wear a respirator.

Documentation of the above, in the form of a copy of each employee's training certificate(s) and summary letter from the occupational medical surveillance examination, must be provided to the on-site manager, prior to performing activities at the Site.

7.3 First Aid

Site personnel will be trained to provide first aid and CPR. The training will also encompass 29 CFR 1910.1030, Occupational Exposure to Bloodborne Pathogens (BBP), including universal precautions and BBP exposure prevention equipment.

SECTION 8.0 EMERGENCY RESPONSE

All personnel entering the work area will receive orientation training from the Field Project Manager. This training will include the safety rules, the emergency alarm/evacuation system, and a site orientation.

The phone numbers of the police and fire departments, ambulance service, local hospital, and AK and contractor representatives are included in the **emergency reference sheet on the following page**. Muster areas (at the main gates) and directions to the nearest hospital that will provide care for injured employees are also provided on the following page.

Prior to initiating work at the Site, the Field Project Manager will activate emergency response provisions. In the event of an injury or illness requiring more than first aid treatment, other site personnel, who will remain with the person until release, will accompany the injured person to the medical facility or admittance is determined. The escort will relay all appropriate medical information to the PM and the HSM.

All accidents, incidents or near misses, regardless of severity must be reported and a written report filed. If medical attention is required, the injured or ill employee will be decontaminated, to the extent possible, and if appropriate, before leaving the Exclusion Zone for transport to the medical facility.

The Site Supervisor must report the injury or illness to the Project Manager as soon as the injured or ill employee is secured. The Site Supervisor is responsible for investigating the cause of the accident. Work can only resume when the cause of the accident has been identified and corrective actions have been completed. If an accident occurs, the Site Supervisor must complete the Injury/Illness reporting form. The completed forms must be sent to the Project Manager and Corporate Health and Safety within 24 hours of the incident.

Any accident/incident resulting in an OSHA recordable injury or illness, treatment at a hospital or physician's office, property damage or a near miss accident requires that an accident/incident report be completed and submitted to the HSM. **The investigation will be initiated as soon as emergency conditions are under control**. The purpose of this investigation is not to attribute blame but to determine the pertinent facts so that repeat or similar occurrences can be avoided. Site work will not continue after a serious incident until appropriate corrections are made.

Site access will be controlled to provide personal security. Gates will be used to prevent unauthorized personnel from entering. Cell phones will be on hand for any emergency phone calls.

SECTION 9.0 EMERGENCY RESPONSE CONTACTS AND TELEPHONE NUMBERS

AK Steel Middletown Works
Middletown, Ohio

9.1 Local Emergency Contacts

Ambulance Service 911
Police 911or 513-425-7700 – Middletown Police Department
Fire 911 or 513-425-7996 – Middletown Fire Department

Hospital: Atrium Medical Center

Location: 200 Medical Center Drive
Middletown, Ohio 45005

Directions:

1. Start out going north on Yankee Rd toward Oxford State Rd.
2. Stay straight to go onto S University Blvd.
3. Turn slight right onto OH-122 E / Roosevelt Blvd. Continue to follow OH-122 E. OH-122 E is 0.1 miles past 14th Ave.
4. Turn slight left onto N Union Rd. N Union Rd is 0.6 miles past Towne Blvd.
5. Take the 1st right onto Medical Center Dr.
6. Take the 2nd left to stay on Medical Center Dr. Medical Center Dr is 0.1 miles past Campus Loop Rd.

Phone: 513-420-5222

9.2 Contacts

Mary Lou Rochotte, KEMRON Project Manager:	Office (740) 373-4308 (X 1266) (b) (6) Fax (740) 376-2536
Field Team Leader:	To Be Determined
Project Engineer:	To Be Determined
Corporate Health and Safety Manager/Program Manager	To Be Determined

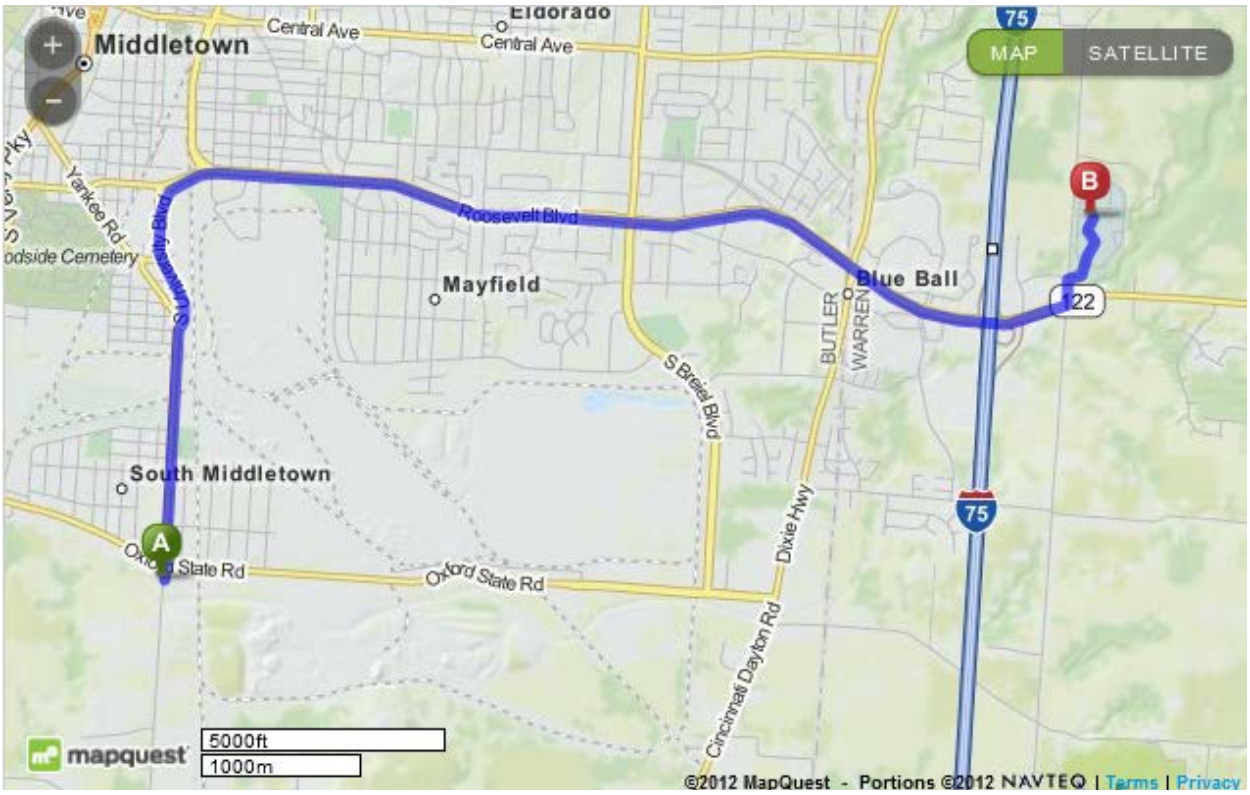
9.3 Client – AK Steel Corporation

Primary Contact: Mr. James Kemp
Office: 513-425-6177
Cell: (b) (6)

Secondary Contact Mr. Pat Gallo
Office: (513) 425-3476
Cell: (b) (6)

Figure 9-1 – Hospital Map

The following map shows the location of the subject property and the location of nearest hospital. Highlight shows route to the hospital.



HEALTH AND SAFETY PLAN
SIGNATURE SHEET

Project: Sampling and Analysis Plan Implementation
AK Steel Corporation
Middleton Works

Location: Middletown, Ohio

I have been provided access to a copy of the Health and Safety Plan prepared for the above-referenced site. I have read and understand its content and I agree that I will abide by its requirements.

Name	Signature/Date

ATTACHMENT H

SWMU 23 Lagoon Closure Historic Files

July 22, 1986

ENGINEERING REPORT #6073

Subject: Closing Coil Paint Ponds
RES-6073 PWR/LMM Coil Paint

Prepared by: L. M. McAdams, Senior Engineer

Introduction:

There are two ponds remaining at the Coil Paint Dept. which were used for wastewater percolation and solids settling. To satisfy EPA requirements, it is necessary to fill in the ponds, cover them with two feet of clay and seed the clay cap with grass.

Recommendation:

Fill in, grade, and install a clay cap on the two Coil Paint ponds. The clay will be excavated from the new sludge disposal area. The estimated project cost is $45,000. Engineering will request an expense appropriation and be in charge of the project.

Discussion:

A. General

The two remaining Coil Paint ponds (a third pond was closed in 1983) were excavated in a sand and gravel area west of the Coil Paint building. The ponds are abandoned and will be closed to meet Ohio Environmental Protection Agency requirements. To minimize percolation of water through the pond area, the ponds will be filled with dirt and slag, graded to drain properly, covered with two feet of compacted clay and seeded with grass. The clay will be excavated from the new sludge disposal area, adjacent to the southeast end of the sludge unloading track.

B. Equipment Description:

Filling and grading of the ponds will be done with a bulldozer, using soil and slag from the area immediately surrounding the ponds (Armco Dwg. #615840). (The third pond, already closed, will not be disturbed).

Excavation of clay from the new sludge area (Armco Dwg. #402865) will be done using a bulldozer and front-end loader. Hauling will be by tandem or Euclid trucks through the Middletown Plant property. A bulldozer and compactor will be used to place the two-foot thick clay cap in six-inch lifts, over the pond area (approx. 230 ft. by 250 ft.).

C. Installation Description:

Work will be done on regular eight hour per day turns, Monday through Friday, using Armco equipment.

D. Engineering Schedule:

Item	Completion Following Approp. Approval.
Final Engineering	2 weeks
Start Installation	3 weeks
Finish Installation	6 weeks

Lelia M. McAdams 7/22/86
Senior Engineer Date

William Daniel 7/22/86
Supt., Coil Paint Date

7-22-86
Manager - Processing & Coat. Date

LMM/js

cc: Supervising Engineers
L. F. Young
G. Grob
C. Batliner
E. Tannreuther

LMM Rec'd 8/25/86

REQUEST FOR APPROPRIATION

DATE July 23, 1986

ARMCO

APPROPRIATION NO. MS9110

GROUP Eastern Steel

CONS'T. ORDER NO.

UNIT _____ LOCATION Midd. Works

ESTIMATE NO. M-6073

BUDGET _____ SHEET NO. _____ ITEM NO. _____

AN APPROPRIATION IS REQUESTED FOR $ 45,000 TO COVER THE FOLLOWING "S" ORDER EXPENDITURE:

DESCRIPTION OF THE PROJECT

Fill in two abandoned wastewater ponds and cover area with a clay cap at the Coil Paint Department.

JUSTIFICATION FOR THE EXPENDITURE

Closing of the ponds is needed to satisfy EPA requirements.

NOTIFICATION OF APPROVAL

RATE OF EXPENDITURE BY QUARTER

3rd Quarter 1986 - $20,000
4th Quarter 1986 - $25,000

SIGNED _____ DATE _____
SUPT. OR FOREMAN
APPROVED [signature] DATE 8-7-86
VEC ASST. GEN'L. SUPT.
PREPARED BY LMM RES# 6073/PWR

ACCTG. CLASS./G.L. CLASS NO. 1983-9012/3195

Asset is ☐ New ☐ Used

Qualifies for ITC ☐ No ☐ Yes _____ %

Qualifies for ETC ☐ No ☐ Yes _____ %

Pollution Control ☐ Air ☐ Water ☐ Solid ☐ Other

Lease Appr. ☐ Fair Market Value ☐ Net Present Value

If Lease, Describe Lease Term Above

[signature] 7/24/86

REQUEST ORIGINATED BY L. M. McAdams DATE

PROJECT UNDER DIRECTION OF Works Engineering

UNIT APPROVALS:

[signature] DATE 8/6/86

[signature] DATE 8/8/86

DIVISION CHIEF FINANCIAL OFFICER DATE

DIVISION PRESIDENT DATE

RELATED REQUESTS: Capital $ _____

Expense $ _____

Retirement $ _____

BUDGET AMOUNT

PREVIOUS APP'NS

THIS REQUEST $45,000

BALANCE

CORPORATE MANAGEMENT APPROVALS:

GROUP/AREA V.P./CORP. STAFF V.P. DATE

V.P.-CORPORATE FINANCE DATE

PRESIDENT DATE

FORM G-1818 REV. 1/81

REQUEST FOR APPROPRIATION

DATE 7-22-86

APPROPRIATION NO. _____

GROUP STEEL

CONS'T. ORDER NO. _____

UNIT EASTERN STEEL LOCATION MIDDLETOWN WORKS

ESTIMATE NO. M-6073

BUDGET EXPENSE SHEET NO. _____ ITEM NO. _____

AN APPROPRIATION IS REQUESTED FOR $ 45,000 TO COVER THE FOLLOWING S ORDER EXPENDITURE:

DESCRIPTION OF THE PROJECT

FILL IN TWO ABANDONED WASTEWATER PONDS, AND COVER AREA WITH A CLAY CAP, AT THE COIL PAINT DEPT.

JUSTIFICATION FOR THE EXPENDITURE

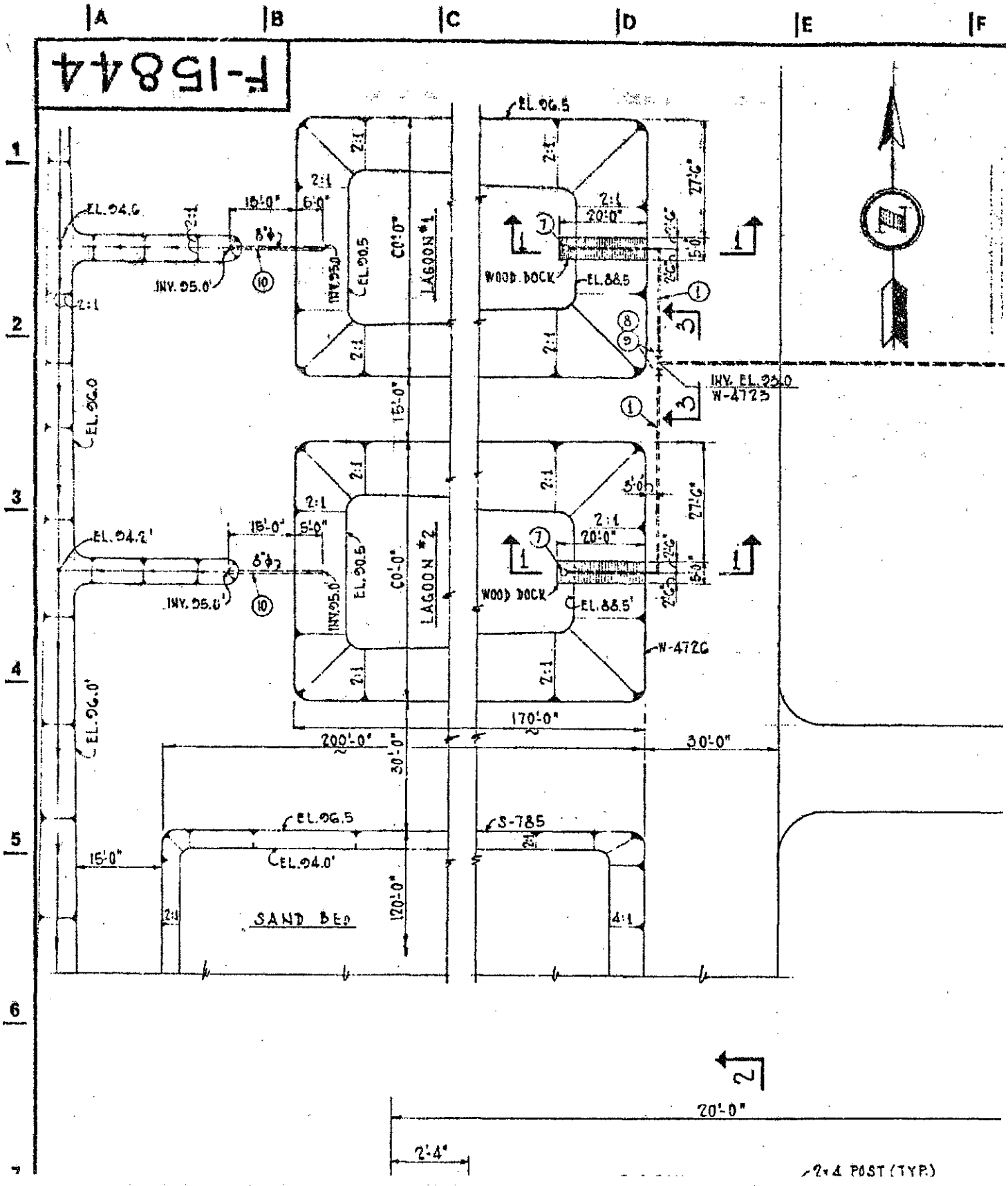
CLOSING OF THE PONDS IS NEEDED TO SATISFY EPA REQUIREMENTS.

RATE OF EXPENDITURE BY QUARTER

3RD QTR. 1986 — #20,000

4TH QTR. 1986 — #25,000

ACCTG. CLASS./G.L. CLASS NO. _____ Asset is ☐ New ☐ Used Qualifies for ITC ☐ No ☐ Yes _____ % Qualifies for ETC ☐ No ☐ Yes _____ % Pollution Control ☐ Air ☐ Water ☐ Solid ☐ Other Lease Appr. ☐ Fair Market Value ☐ Net Present Value If Lease, Describe Lease Term Above	RELATED REQUESTS: Capital $ — Expense $ — Retirement $ —
REQUEST ORIGINATED BY L. M. McADAMS DATE 7-22-86	BUDGET AMOUNT
PROJECT UNDER DIRECTION OF WORKS ENGINEERING	PREVIOUS APP'NS
UNIT APPROVALS:	THIS REQUEST #45,000
DATE	BALANCE
DATE	CORPORATE MANAGEMENT APPROVALS:
DIVISION CHIEF FINANCIAL OFFICER DATE	GROUP/AREA V.P./CORP. STAFF V.P. DATE
DIVISION PRESIDENT DATE	V.P.-CORPORATE FINANCE DATE
	PRESIDENT DATE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EL. 94.6
EL. 94.2'
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EL. 96.0'
INV. 95.0'
INV. 95.0
EL. 90.5
LAGOON #1
LAGOON #2
WOOD DOCK
EL. 88.5
EL. 88.5'
INV. EL. 95.0
W-4723
W-4726
S-785
EL. 94.0'
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60'-0"
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170'-0"
200'-0"
30'-0"
120'-0"
2:1
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