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QUALITY ASSURANCE PROJECT PLAN (QAPP)
REMEDIAL INVESTIGATION/FEASIBILITY STUDY

H.O.D. LANDFILL
ANTIOCH, ILLINOIS

VOLUME 1 OF 2

SEPTEMBER 1992

PREPARED FOR:
WASTE MANAGEMENT OF ILLINOIS, INC.
WESTCHESTER, ILLINOIS

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QUALITY ASSURANCE PROJECT PLAN (QAPP)
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
H.O.D LANDFILL
ANTIOCH, ILLINOIS
(September 1992)

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QUALITY ASSURANCE PROJECT PLAN (QAPP)
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H.O.D. LANDFILL SITE
ANTIOCH, ILLINOIS

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LIST OF ACRONYMS AND ABBREVIATIONS

AOC	Administrative Order on Consent
ARAR	Applicable or Relevant and Appropriate Requirements
ASTM	American Standards for Testing Materials
CDO	Central District Office
CLP	Contract Laboratory Program
CRDL	Contract Required Detection Limits
CRQL	Contract Required Quantitation Limits
CRL	Central Regional Laboratory
DQO	Data Quality Objective
ESI	Expanded Site Inspection
ETC	Environmental Testing and Certification Corporation
FIT	Field Investigation Team
HRS	Hazard Ranking System
HSP	Site Specific Health and Safety Plan
IEPA	Illinois Environmental Protection Agency
LL TAL	Low Level Detection Target Analyte List
LL TCL	Low Level Detection Target Compound List
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NPL	National Priorities List
PA	Preliminary Assessment
PRP	Potentially Responsible Party
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QAS	Quality Assurance Section
QC	Quality Control
RI/FS	Remedial Investigation/Feasibility Study
RMAL	Rocky Mountain Analytical Laboratory
RPD	Relative Percent Difference
RPM	Remedial Project Manager
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SOW	Statement of Work
TAL	Target Analyte List
TDS	Total Dissolved Solids
TCL	Target Compound List
TIC	Tentatively Identified Compound
TOC	Total Organic Carbon
U.S. EPA	United States Environmental Protection Agency, Region V
VOA	Volatile Organic Analysis
Warzyn	Warzyn Inc.
WMII	Waste Management of Illinois, Inc.

INTRODUCTION

The United States Environmental Protection Agency (U.S. EPA) requires that all environmental monitoring and measurement efforts mandated or supported by U.S EPA participate in a centrally managed quality assurance (QA) program.

Any party generating data under this program has the responsibility to implement minimum procedures to assure that precision, accuracy, completeness, and representativeness of its data are known and documented. To ensure the responsibility is met uniformly, each party must prepare a written QA Project Plan (QAPP) covering each project it is to perform.

This QAPP presents the organization, objectives, functional activities, and specific Quality Assurance (QA) and Quality Control (QC) activities associated with the Remedial Investigation/Feasibility Study (RI/FS) for the H.O.D. Landfill Site. This QAPP also describes the specific protocols which will be followed for sampling, sample handling and storage, chain of custody, and laboratory and field analysis.

All QA/QC procedures will be in accordance with applicable professional technical standards, U.S. EPA requirements, government regulations and guidelines, and specific project goals and requirements. This QAPP is prepared by Warzyn Inc. (Warzyn) in accordance with the U.S. EPA QAPP guidance documents:

- U.S. EPA, December 1980, Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80
- U.S. EPA, May 1991, Region V Model Superfund Quality Assurance Project Plan
- U.S. EPA, January 1989, Region V Content Requirements for Quality Assurance Project Plan

SECTION 1
PROJECT DESCRIPTION

1.1 Site History and Background Information

The H.O.D. Landfill Site (Site) is an inactive landfill located in the eastern boundary of the Village of Antioch in Lake County in northeastern Illinois (Township 46 North, Range 10 East, Sections 8 and 9). The Site is bordered on the south and west by Sequoit Creek. The Silver Lake residential subdivision is located east of the Site, and agricultural land, scattered residential areas, and undeveloped land is located to the north. A large wetland area extends south of the Site from Sequoit Creek. Silver Lake is located approximately 200 feet southeast of the Site. A large industrial park area (Sequoit Acres Industrial Park), constructed on former landfill and fill areas, is located west of the Site and borders Sequoit Creek. Refer to Figure 1-1 for the Site Location Map.

The Site consists of a total of 80 acres, of which 51 acres which have been landfilled. Although the landfilled area is visually continuous, it consisted of two separate landfill areas, identified as the "old" and "new" landfills. The "old" landfill, consisting of 24.2 acres situated on the western third of the property, operated between 1963 and 1975. The "new" landfill, consisting of 26.8 acres situated immediately east of the "old" landfill, operated between 1975 and 1984. The landfill accepted both municipal waste and a variety of industrial and special wastes.

A more detailed description of the site history and background are contained in the Work Plan.

1.2 Past Data Collection Activity / Current Status

The Site has been subject to a number of investigations since 1973. A discussion of the investigations is provided in Section 3.1.6 of the Work Plan.

A Preliminary Assessment (PA) was prepared by the E&E Field Investigation Team (FIT) for U.S. EPA in 1983. The FIT conducted a site inspection on July 10, 1984; the Site was given a Hazard Ranking System (HRS) model score of 52.02. At that time, the U.S. EPA proposed the Site be placed on the National Priorities List (NPL).

An Expanded Site Inspection (ESI) was conducted by E&E during the period of 1986 through 1989, resulting in an ESI report submitted to U.S. EPA on September 22 , 1989. In January 1990, the H.O.D. Landfill was rescored under the HRS and received a revised score of 34.68.

The Site was placed on the NPL in February 1990. On August 20, 1990 Waste Management of Illinois, Inc. (WMII) and U.S. EPA entered an Administrative Order on Consent (AOC) to conduct a Potentially Responsible Party (PRP) lead RI/FS at the Site. Warzyn was contracted by WMII to support the PRP lead RI/FS effort in developing the RI/FS Work Plan and other planning documents, including this QAPP.

1.3 Project Objectives and Scope

The purpose of the RI is to gather sufficient information to quantify risk to public health and environment (Baseline Risk Assessment) and to develop and evaluate viable remedial alternatives (Feasibility Study) at the Site. The objectives of the RI are to determine the nature and extent of contamination at the Site in order to support the activities of the FS. The objective of the RI/FS is to develop and evaluate appropriate remedial action alternatives based on the RI/FS data.

The objectives of the data collection are as follows:

- Further define the nature and extent of contamination in previously identified on-site and off-site areas

- Determine the nature and extent of contamination in previously uninvestigated areas

- Collect sufficient data on all contaminated media to support the baseline risk assessment and feasibility study.

All tasks, subtasks and activities are directed toward the accomplishment of these primary objectives. Refer to the Work Plan for a detailed description of the RI tasks, subtasks and activities.

1.4 Sample Network Design and Rationale

The sample network design and rationale for sample locations is described in detail in Section 3 of the Sampling and Analysis Plan (SAP) (refer to Appendix A).

1.5 Parameters to be Tested and Frequency

Sample matrices, analytical parameters, and frequencies of sample collection can be found in Table 1-1. A summary of sample volume, bottle, preservative, and packaging requirements is provided in Table 1-2.

1.6 Intended Data Usage and Data Quality Objectives

Data Quality Objectives (DQOs) are qualitative and quantitative statements which specify the quality of the data required to support decisions made during the RI/FS activities, and are based on the end uses of the data to be collected. As such, different data uses may require different levels of data quality. There are five analytical levels which address various data uses and the QA/QC effort and methods required to achieve the desired level of quality. These levels are:

- Screening (DQO Level I): This provides the lowest data quality but the most rapid results. It is often used for health and safety monitoring at the Site, preliminary comparison to Applicable or Relevant and Appropriate Requirements (ARARs),

initial site characterization to locate areas for subsequent and more accurate analyses, and for engineering screening of alternatives (bench-scale tests). For the H.O.D. Landfill Site, data generated under DQO Level I are for health and safety monitoring. Health and Safety Monitoring activities are described in the Site Specific Health and Safety Plan (HSP) and are not covered in this QAPP.

- Field Analyses (DQO Level II): This provides rapid results and better quality than in Level I. This level may include mobile lab generated data depending on the level of quality control exercised. For the H.O.D. Landfill Site, data generated under DQO Level II are: the determination of methane, oxygen, and carbon dioxide in landfill gas, and pH, conductivity, temperature, dissolved oxygen, and redox potential in water.

- Engineering (DQO Level III): This provides an intermediate level of data quality and is used for site characterization. Engineering analyses may include mobile lab generated data and some analytical lab methods (e.g., laboratory data with quick turnaround for screening, but without full quality control documentation). For the H.O.D. Landfill Site, data generated under DQO Level III are the physical characterization of soils for: grain size, atterberg limits, natural moisture content, density, TOC, and clay mineralogy.

- Confirmational (DQO Level IV): This provides the highest level of data quality and is used for purposes of risk assessment, evaluation of remedial alternatives, and PRP determination. These analyses require full Contract Laboratory Program (CLP) analytical and data validation procedures in accordance with U.S. EPA recognized protocol. For the H.O.D. Landfill Site, data generated under DQO Level IV are the Target Compound List (TCL) organic and Target Analyte List (TAL) inorganic parameters for groundwater, sediment/soil, leachate, and surface water matrices. Low

level TCL organic and low level TAL inorganic data for municipal/private wells will also fall under DQO Level IV.

- Non-Standard (DQO Level V): This refers to analyses by non-standard protocols, for example, when exacting detection limits or analysis of an unusual chemical compound is required. These analyses often require method development or adaptation. The level of quality control is usually similar to DQO Level IV data. For the H.O.D. Landfill Site, data generated under DQO Level V are the volatiles for the landfill gas samples and indicator parameters (alkalinity, chloride, hardness, sulfate, total organic carbon (TOC), total dissolved solids (TDS), nitrate nitrogen, nitrite nitrogen, and ammonia nitrogen) for groundwater and leachate.

A summary of data generating activities, intended data uses and associated DQOs for the H.O.D. Landfill Site are presented in Table 1-3.

1.7 Project Schedule

A schedule of RI/FS activities for the H.O.D. Landfill Site is summarized in Figure 1-2.

SECTION 2

PROJECT ORGANIZATION AND RESPONSIBILITY

Warzyn will be conducting all phases of the RI/FS. Warzyn will perform the field investigation, prepare the RI report, and perform the subsequent feasibility study. Project management will also be provided by Warzyn, under the direction of WMII. The various quality assurance and management responsibilities of key project personnel are defined below. Refer to Figure 2-1 for the project organizational chart.

2.1 Overall Responsibility

U.S. EPA Remedial Project Manager

The U.S. EPA Remedial Project Manager (RPM) is Fred Micke. The RPM has the overall responsibility for all phases of the RI/FS.

WMII Site Project Manager

The Site Project Manager is March Smith. The Site Project Manager is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. The Site Project Manager's primary function is to ensure that technical, financial, and scheduling objectives are achieved successfully and will provide the major point of contact and control for matters concerning the project.

Warzyn Project Manager

The Warzyn Project Manager is Gary Parker. The Warzyn Project Manager has the overall responsibility for ensuring that the project meets U.S. EPA objectives and Warzyn's quality standards. In addition, he is responsible for technical quality control and project oversight, and will provide the Site Project Manager with access to corporate management.

The WMII Site Project Manager and Warzyn Project Manager will:

- Define project objectives and develop a detailed work plan schedule
- Establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task
- Acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule constraints
- Orient all field leaders and support staff concerning the project's special considerations
- Monitor and direct the field leaders
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness
- Review and analyze overall task performance with respect to planned requirements and authorizations
- Approve all external reports (deliverables) before their submission to U.S. EPA
- Ultimately be responsible for the preparation and quality of interim and final reports
- Represent the project team at meetings and public hearings

Warzyn Remedial Investigation Leader

The Warzyn RI Leader is Al Schmidt. The RI Leader is a support to the Warzyn Project Manager. He is responsible for leading and coordinating the day-to-day activities of the various resource specialists under his supervision. The RI Leader is a highly experienced environmental professional and will report directly to the Warzyn Project Manager. Specific responsibilities

include:

- Provision of day-to-day coordination with the Project Managers on technical issues in specific areas of expertise
- Development and implementation of field-related work plans, assurance of schedule compliance, and adherence to management-developed study requirements
- Coordination and management of field staff including sampling, drilling, and other investigative work
- Implementation of QC for technical data provided by the field staff including field measurement data
- Adherence to established project schedule
- Review and approval of text and graphics required for field team efforts
- Coordination and oversight of technical efforts of subcontractors assisting the field team
- Identification of problems at the field team level, discussion of resolutions with the Project Managers, and provision of communication between team and upper management
- Participation in the preparation of the final RI report

Technical Staff

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

Warzyn Quality Assurance Officer

The Warzyn Quality Assurance Officer (QAO) is Kevin Domack. The QAO will remain independent of direct job involvement and day-to-day operations, and has direct access to corporate executive staff as necessary to resolve any QA dispute. He is responsible for auditing the implementation of the QA program in conformance with the demands of specific investigations, Warzyn's policies, and U.S. EPA requirements. Specific functions and duties include:

- Provide QA audit on various phases of the field operations
- Review and approval of QA plans and procedures
- Provide QA technical assistance to project staff

U.S. EPA Quality Assurance Officer

The U.S. EPA QAO has the responsibility to review and approve the QAPP. The U.S. EPA QAO is Valerie Jones.

2.2 Specialized Responsibilities

2.2.1 Monitoring and Sampling Operations and QC

- Principal Engineering Firm - Warzyn, Chicago, Illinois
- Drilling - To be determined through bidding process
- Sampling, Monitoring and Survey - Warzyn, Chicago, Illinois
- On Site Day-to-day Field Activities - RI Leader, Warzyn, Chicago, Illinois
- Quality Control - QAO, Warzyn, Chicago, Illinois

2.2.2 Laboratory Analysis

- Analysis of groundwater, surface water, sediment/soil, and leachate samples for TCL organics and TAL inorganics as specified in Table 1-1:

  Environmental Testing and Certification Corporation (ETC)
  284 Raritan Center Parkway
  Edison, New Jersey 08818

- Analysis of sediment/soil for TOC as specified in Table 1-1:

  ETC
  284 Raritan Center Parkway
  Edison, New Jersey 08818

- Analysis of groundwater and leachate for indicator parameters (alkalinity, chloride, hardness, sulfate, TOC, TDS, nitrate nitrogen, nitrite nitrogen, and ammonia nitrogen) as specified in Table 1-1:

  Enseco/Rocky Mountain Analytical Laboratory (Enseco/RMAL)
  4955 Yarrow Street
  Arvada, Colorado 80002

- Analysis of municipal/private well samples for low level TCL organics and low level TAL inorganics as specified in Table 1-1:

  Enseco/RMAL
  4955 Yarrow Street
  Arvada, Colorado 80002

- Analysis of landfill gas samples for volatile organics as specified in Table 1-1:

  Enseco - Air Toxics Laboratory
  9537 Telstar Avenue, Suite 118
  El Monte, California 91731

- Analysis of sediment/soil samples for physical characteristics (grain size, natural moisture content, atterberg limits, permeability, and density) as specified in Table 1-1:

  Warzyn Soils Laboratory
  505 Science Drive, Suite C
  Madison, Wisconsin 53711

- Analysis of soil samples for clay mineralogy using x-ray diffraction as specified in Table 1-1:

  Core Laboratories Inc.
  1875 Monetary Lane
  Carrollton, Texas 75006

2.2.3 Laboratory Data and QC

Laboratory Data

- Analytical protocol specified - Warzyn, Chicago, Illinois

- Review of analytical protocol - ETC, Edison, New Jersey

- Review of analytical procedures - U.S. EPA Region V Quality Assurance Section (QAS) and Central Regional Laboratory (CRL), Chicago, Illinois

- Internal QA/QC - Laboratory QAO, ETC, Edison, New Jersey

- Final data review and validation - Chemist, Warzyn, Chicago, Illinois

- Review of tentatively identified compounds and assessment of need for confirmation - Chemist, Warzyn, Chicago, Illinois

2.2.4 Performance and Systems Audits

Field Operations

- Internal Audits - QAO, Warzyn, Chicago, Illinois

- External Audits - U.S. EPA Region V CRL and Central District Office (CDO), Chicago, Illinois

Analytical Laboratories

- Internal Audits - Laboratory QAO, ETC, Edison, New Jersey

- External Audits - U.S. EPA Region V CRL, Chicago, Illinois

Final Evidence File

- Final Evidence File Audits - QAO, Warzyn, Chicago, Illinois

SECTION 3

QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results which are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instruments calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field equipment, and corrective action are described in other sections of this QAPP. The purpose of this section is to address the specific objectives for accuracy, precision, completeness, representativeness, and comparability.

3.1 Level of Quality Control Effort

Field blank, trip blank, duplicate, and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling program. Field and trip blanks consisting of deionized water, will be submitted to the analytical laboratories to provide the means to assess the quality of the data resulting from the field sampling program. Field blank samples are analyzed to check for procedural contamination at the Site which may cause sample contamination. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. Matrix spikes are performed in duplicate for organic analyses, and are hereinafter referred to as matrix spike/matrix spike duplicate (MS/MSD) samples. MS/MSD samples are designated/collected for organic analyses only.

The general level of the QC effort will be one field duplicate and one field blank for every 10 or fewer investigative samples. One volatile organic analysis (VOA) trip blank consisting of deionized, ultra pure water, will be included along with each shipment of aqueous VOA samples.

MS/MSD samples are not investigative samples. Soil MS/MSD samples require no extra volume for VOAs or extractable organics. However, aqueous MS/MSD samples must be collected at triple the volume for VOAs and double the volume for extractable organics. One MS/MSD sample will be collected/designated for every 20 or fewer investigative samples per sample matrix (i.e., groundwater, soil). The number of field duplicate and field blank samples to be collected for this Site are listed in Table 1-1. Sampling procedures are specified in the SAP (refer to Appendix A).

Soil/sediment, landfill gas, groundwater, surface water, leachate, municipal well, and private well samples will be sent to ETC, Enseco/RMAL, Enseco - Air Toxics Laboratory, Warzyn, and Core for analysis. Parameter lists and required quantitation levels are summarized in Tables 3-1, 3-2, 3-3, 3-4, 3-5, and 3-6.

The level of laboratory QC effort for testing of TAL inorganics and TCL organics is specified in the current CLP Statements of Work (SOWs): ILM02.0 for inorganics (including low level inorganics), OLM01.8 for organics, and OLC01.0 for low level organics. The levels of laboratory QC effort for indicator parameters, field measurements, physical characteristics, and landfill gas volatiles are specified in Table 3-7.

3.2 Accuracy, Precision and Sensitivity of Analyses

The fundamental QA objective with respect to accuracy, precision, and sensitivity of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols.

TCL Organics/TAL Inorganics

Laboratory analysis of TCL organics and TAL inorganics will follow the current CLP SOWs (OLM01.8 for organics, ILM02.0 for inorganics). The accuracy and precision, and sensitivity requirements are summarized within the CLP SOWs.

Low Level TCL Organics/TAL Inorganics

The laboratory analysis of low level TCL organics for private and municipal well samples will follow the current CLP SOW OLC01.0. The accuracy and precision, and sensitivity requirements for the low level TCL organics are summarized within the CLP SOW. The laboratory analysis of low level TAL inorganics for private and municipal well samples will follow the SOPs provided in Appendix B. The accuracy and precision requirements for the low level TAL inorganics are the same as those in the CLP SOW ILM02.0. The sensitivity requirements for these analyses are summarized in Table 3-4.

Indicators

SOPs for the indicator parameters (alkalinity, chloride, hardness, sulfate, TOC, TDS, nitrate nitrogen, nitrite nitrogen, and ammonia nitrogen) are provided in Appendix B. The accuracy and precision requirements for these analyses are summarized in Table 3-7. The sensitivity requirements for these analyses are summarized in Table 3-6.

Physical Characteristics

Physical characteristics of grain size, natural moisture content, atterberg limits, density, TOC, and clay mineralogy will follow the methods provided in Appendix B. The accuracy, precision, and sensitivity requirements for these analyses are summarized in Table 3-7.

Landfill Gas Volatiles

The SOP for the analysis of landfill gas for volatiles is provided in Appendix B. The accuracy and precision requirements are summarized in Table 3-7. The sensitivity requirements are summarized in Table 3-5.

Field Measurements

SOPs for the field equipment to measure pH, conductivity, temperature, dissolved oxygen, redox potential, methane, oxygen, and carbon dioxide are provided in Appendix C. The accuracy and precision requirements of these analyses are summarized in Table 3-7. The sensitivity requirements of these analyses are summarized in Table 3-6.

3.3 Completeness, Representativeness and Comparability

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that the participating laboratories will provide data meeting QC acceptance criteria for 95 percent or more for all samples tested using the SOWs and SOPs referenced in Section 3.2. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness (\%)} = A/B \times 100$$

where, A = number of valid data
B = number of samples collected for each parameter analyzed

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling network was designed to provide data representative of Site conditions. During development of this network, consideration was given to past waste disposal practices, existing analytical data,

physical setting and processes, and constraints inherent to the Superfund program. The rationale of the sampling network is discussed in detail in the SAP. Representativeness will be satisfied by insuring that the SAP is followed, proper sampling technique are used, proper analytical procedure are followed, and holding times of the samples are not exceeded in the laboratory. Representativeness will be assessed by the analysis of field duplicate samples.

Comparability expresses the confidence with which one data set can be compared with another. The extent to which existing and planned analytical data will be comparable, depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data, as documented in the QAPP, are expected to provide comparable data. These new analytical data, however, may not be directly comparable to existing data because of differences in procedures and QA objectives.

SECTION 4
SAMPLING PROCEDURES

4.1 Sampling Procedures

Sampling procedures are described in the SAP which is contained in Appendix A of this document.

4.2 Bottle Requirements

The contaminant-free sample containers (bottles) used for analyzing TCL organics, TAL inorganics, and indicator parameters for the H.O.D Landfill Site will be prepared according to the procedures specified in U.S. EPA's Specifications and Guidance for Obtaining Contaminant-Free Sample Containers, April 1990. It will assure that the bottles used for the sampling activity do not contain contaminants exceeding the level specified in the above-mentioned document. Specifications for the bottles will be verified by checking the supplier's certified statement and analytical results for each bottle lot, and will be documented on a continuing basis. This data will be maintained in the project evidence file, and will be available, if requested, for EPA review.

In addition, the data for field blanks and trip blanks will be monitored for contamination, and corrective actions will be taken as soon as a problem is identified. This will be accomplished either by discontinuing the use of a specific bottle lot, contacting the bottle supplier(s) for retesting a representative bottle from a suspect lot, resampling the suspected samples, validating the data taking into account that the contaminants could be introduced by the laboratory (i.e., common lab solvents, sample handling artifacts, etc.) or could be a bottle QC problem, so as to make an educated determination of whether the bottles and hence the data are still usable, etc., whichever is appropriate.

SECTION 5

SAMPLE CUSTODY AND DOCUMENTATION

It is U.S. EPA policy to follow the U.S. EPA sample custody, or chain of custody protocols described in "NEIC Policies and Procedures", EPA-330/9-78-DDI-R, Revised June 1985. This custody is in three parts: sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if they:

- Are in your possession
- Are in your view, after being in your possession
- Are in your possession and you place them in a secured location
- Are in a designated secure area

5.1 Field Specific Custody Procedures

The sample packaging and shipment procedures summarized below will assure that the samples will arrive at the laboratory with the chain of custody intact. The protocol for sample designations are included in the SAP, Appendix A of this QAPP.

5.1.1 Initiation of Chain of Custody Field Procedures

The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched to the laboratory. As few people as possible should handle the samples. All bottles will be tagged with sample numbers and locations. Sample tags are to be completed for each sample using waterproof ink unless prohibited by weather conditions. For

example, a logbook notation would explain that a pencil was used to fill out the sample tag because the ballpoint pen would not function in freezing weather.

The RPM will review all field activities to determine whether proper custody procedures were followed during the field work and decide if additional samples are required.

5.1.2 Field Logbooks/Documentation

Field logbooks will provide the means of recording data collecting activities performed. As such, entries will be described in as much detail as possible so that persons going to the site could re-construct a particular situation without reliance on memory.

Field logbooks will be bound, field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each logbook will be identified by the project-specific document number.

The title page of each logbook will contain the following:

- Person to whom the logbook is assigned
- Logbook number
- Project name
- Project start date
- Project end date

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the site, field sampling or investigation team personnel, and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. All entries will be made in ink, and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark, initialed, and dated by the person making the crossout. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the station, which includes compass and distance measurements, shall be recorded. The number of the photographs taken of the station, if any, will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in the SAP, Appendix A of this QAPP. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. A sample identification number will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

5.1.3 Transfer of Custody and Shipment Procedures

Samples are accompanied by a properly completed chain of custody form. The sample numbers and locations will be listed on the chain of custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of the samples from the sampler to another person to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.

Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be locked and secured with strapping tape and custody seals for shipment to the laboratory. The preferred procedure includes the use of a custody seal attached to the front right

and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.

Whenever samples are split with another source or government agency, a separate sample custody record is prepared for those samples, and marked to indicate with whom the samples are being split with. The person relinquishing the samples to the facility or agency should request the representatives signature acknowledging the sample receipt. If the representative is unavailable or refuses, the person relinquishing the samples should note this in the "received by" space of the custody form.

All shipments will be accompanied by the chain of custody record identifying the contents. The original record will accompany the shipment, and the pink and yellow copies will be retained by the sampler for returning to the sampling office.

If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on the custody forms as long as the custody forms are sealed inside the sample cooler, and the custody seals remain intact. The person shipping the samples should note the carrier name and airbill number on the chain of custody record.

5.2 Laboratory Chain of Custody Procedures

Laboratory custody procedures for the sample receiving and log-in; sample storage; tracking during sample preparation and analysis; and storage of data are described in the individual laboratory custody SOPs provided in Appendix D.

5.3 Final Evidence File Custody Procedure

The final evidence file for the H.O.D. Landfill RI/FS will be located at and maintained by Warzyn. The content of the evidence file will include all relevant records, reports, correspondence, logs, field logbooks, laboratory sample preparation and analysis logbooks/documentation, analytical data packages, pictures, subcontractor reports, chain of custody records/forms, data review reports, etc. The evidence file will be under custody of the file custodian in a locked, secured area.

SECTION 6

CALIBRATION PROCEDURES AND FREQUENCY

This section describes procedures for maintaining the accuracy of all the instruments and measuring equipment which are used for conducting field tests and laboratory analyses. These instruments and equipment should be calibrated prior to each use or scheduled, periodic basis.

6.1 Field Instruments/Equipment

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 to certify that it is in operating condition. This includes checking the manufacturer's operating manual and the instruction and the instructions for each instrument to ensure that all maintenance requirements are being observed. Field notes from previous sampling trips will be reviewed so that the notation on any prior equipment problems are not overlooked, and all necessary repairs to equipment have been carried out.

Calibration procedures and frequencies for field instruments are governed by the specific field SOPs provided in Appendix C. Field measurements include pH, conductivity, temperature, dissolved oxygen, redox potential, methane, oxygen, and carbon dioxide.

6.2 Laboratory Instruments

Procedures for the calibration of laboratory instruments must be established and maintained to ensure that equipment is functioning properly and that data collected are accurate and reliable. Requirements include step-by-step calibration procedures, frequency of re-calibration, equipment

maintenance logs, instrument accuracy criteria, corrective action procedures and equipment limitations (e.g., working ranges), and are described, in detail, in the SOPs provided in Appendix B, and the SOWs for organics and inorganics.

SECTION 7

ANALYTICAL PROCEDURES

Groundwater, private/municipal well, surface water, soil/sediment, landfill gas, and leachate samples collected during field sampling activities for the H.O.D. Landfill Site RI will be analyzed by ETC, Enseco/RMAL, Enseco - Air Toxics Laboratory, Warzyn, and Core (see Table 1-1). Refer to Table 7-1 for a summary of analytical method references used for analysis.

7.1 Field Screening Analytical Protocols

The procedures for the field measurement of pH, conductivity, temperature, dissolved oxygen, redox potential, methane, oxygen and carbon dioxide are described in the SOPs provided in Appendix C.

7.2 Laboratory Analysis

Samples (groundwater, surface water, soil/sediment, and leachate samples) for CLP TCL organics and CLP TAL inorganics will be analyzed by ETC according to the analytical procedures set forth in the current CLP SOWs OLM01.8 for organics analysis, and ILM02.0 for inorganic analysis. Indicator parameters analyzed by Enseco/RMAL will follow the SOPs in Appendix B.

Low level TCL organic analysis of municipal/private wells by Enseco/RMAL will utilize the current low level CLP SOW OLC01.0. Low level TAL inorganic analysis for municipal/private wells analyzed by Enseco/RMAL will follow the SOPs set forth in Appendix B.

Landfill gas samples for volatiles will be analyzed by Enseco - Air Toxics Laboratory according to the SOP set forth in Appendix B.

Analysis of soil/sediments for physical characteristics will be performed by Warzyn (TOC for soil/sediment analyzed by ETC and clay mineralogy will be performed by Core Laboratories) using the methods provided in Appendix B.

SECTION 8

INTERNAL QUALITY CONTROL CHECK

8.1 Field Sample Collection

The assessment of field sampling precision and accuracy will be made through the collection of field duplicates and field blanks in accordance with the procedures described in the SAP (Appendix A of this QAPP). Refer to Table 1-1 for a summary of sample numbers and required field QC samples.

8.2 Field Measurements

QC procedures for field measurements of pH, conductivity, temperature, dissolved oxygen, redox potential, methane, oxygen, and carbon dioxide are limited to checking the reproducibility of the measurements by obtaining multiple readings on a single sample or standard, and by calibrating the instruments. Refer to Table 3-7 for a summary of QC requirements.

8.3 Laboratory Analysis

The laboratories used for the analysis of samples for the H.O.D. Landfill Site (ETC, Enseco/RMAL, Enseco - Air Toxics Laboratory, Warzyn and Core Laboratories) have written QA/QC programs which provides rules and guidelines to ensure the reliability and validity of work conducted at the laboratory. Compliance with the QA/QC program is coordinated and monitored by the laboratory QAOs, which are independent of the operating departments. Laboratory procedures used are documented in writing and are provided or referenced in this QAPP. Internal quality control checks are an integral part of the analytical methods, and are discussed in detail within the analytical procedures. The overall objectives of the internal quality control checks are to verify the established precision, accuracy and integrity of the methodology and to support the technical validity of the data. Where appropriate, internal quality control checks for analyses other than those following the CLP SOWs will include method blanks,

preparation/reagent blanks, calibration check samples, laboratory duplicates, matrix spikes and continuing calibration standards.

The required quality control frequency and performance criteria for TCL organics and TAL inorganics are summarized in the current CLP SOWs OLM01.8 and ILM02.0, respectively. The required QC frequency and performance criteria for low level organics are summarized in the current CLP SOW OLC01.0.

The required quality control frequency and performance criteria for the indicator parameters, landfill gas volatiles, physical characteristics, and low level TAL inorganics are summarized in Table 3-7.

The performing laboratories will document, in each data package provided, that both initial and ongoing instrument and analytical QC functions have been met. Any samples analyzed in non-conformance with the QC criteria set forth will be reanalyzed by the laboratory. It is expected that sufficient sample volume will be collected for re-analyses.

SECTION 9

DATA REDUCTION, VALIDATION AND REPORTING

9.1 Field Measurements and Sample Collection

Raw data from field measurements and sample collection activities will be appropriately recorded in the field logbook. Data will be reviewed to ensure procedures were followed and QC requirements were met, however, no formal data validation effort will be performed. If the data are to be used in the project reports, they will be reduced onto data summary tables.

9.2 Laboratory Services

9.2.1 Data Reduction

Each laboratory is responsible for identification, quantification, data reporting, and data deliverables for the analyses performed. Data reduction of TCL organic, low level TCL organic, and landfill gas volatile data will follow the requirements set forth in the CLP SOWs (OLM01.8 and OLC01.0). Data reduction of TAL inorganic, low level TAL inorganic, and indicators data will follow the requirements set forth in the CLP SOW ILM02.0. Deliverables will include raw data, summaries of calibration standards, duplicates, spikes, blanks, and performance evaluation samples.

9.2.2 Data Validation

Organic data (including low level TCL organics and landfill gas volatiles) generated under a DQO level of IV or V will be validated by Warzyn using Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses, February 1988.

Inorganic data (including low level TAL inorganics) generated under a DQO level of IV will be validated by Warzyn using Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses, July 1988.

Indicator data generated under a DQO level of V will be validated by Warzyn using the Data Validation SOP provided in Appendix E.

Physical characteristic data (DQO level III) will be reviewed to ensure procedures were followed and QC requirements were met, however, no formal data validation effort will be performed.

Refer to Table 1-2 for a summary of data generating activities, intended data uses, and associated DQOs for the H.O.D. Landfill Site.

9.2.3 Data Reporting

Analytical data generated for the H.O.D. Landfill Site RI/FS will be computerized in a format organized to facilitate data review and evaluation. The computerized data set will include the data qualifiers provided by the performing laboratory in accordance with the CLP SOWs, as well as qualifiers added by the data reviewer in accordance with the data validation procedures noted in Section 9.2.2 of this QAPP.

The laboratory-provided qualifiers will include such items as:

- Non-detects
- Concentration below required detection limit
- Estimated concentration due to poor QC data
- Concentration of chemical also found in the laboratory blank

The data review qualifiers will indicate whether the data are:

- Usable as a quantitative concentration
- Usable with caution as an estimated concentration
- Unusable due to out-of-control QC results

A summary of the validated data will be incorporated into the RI report.

SECTION 10

PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the SAP and QAPP. The audits of field and laboratory activities include two separate independent parts: internal and external audits.

10.1 Field Audits

10.1.1 Internal Audits

Internal audits of field activities (sampling and measurements) will be conducted by the Warzyn QAO and/or RI Leader. The purpose of the field audit will be to evaluate and document adherence to procedures described in the QAPP and SAP. The audit will include review of field activities, sample documentation, chain of custody forms, field logbooks, and sampling and decontamination activities. Follow-up audits will be conducted to correct deficiencies, and to verify that QA procedures are maintained throughout the investigation.

10.1.2 External Audits

External audits conducted will be the responsibility of the U.S. EPA CRL and/or CDO.

10.2 Laboratory Audits

10.2.1 Internal Audits

The purpose of the internal laboratory audit is to evaluate and document adherence to analytical procedures described in this QAPP. Internal audits of the participating laboratories are the responsibility of the individual laboratory QAO. System audits will include examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain of custody procedure, sample preparation and analysis, instrument operating records, etc., and will be

performed annually. Performance audits consisting of blind QC samples prepared and submitted to the laboratory for analysis, will be performed quarterly. Results of these blind QC samples will be reviewed by the laboratory QAO.

10.2.2 External Audits

The U.S. EPA CRL will audit performing laboratories and provide recommendations for approval of the laboratory for the requested analyses to the U.S. EPA RPM. The audit may consist of a review of analytical and chain of custody procedures, evaluation of performance samples, and may also include an on-site audit of each participating laboratory.

SECTION 11
PREVENTATIVE MAINTENANCE

11.1 Field Instruments/Equipment

Field equipment used for this project include: thermometers, pH meter, conductivity meter, dissolved oxygen meter, redox meter, gas-tech meter, and sampling and filtering equipment. Specific preventative maintenance procedures to be followed for field equipment are those recommended by the manufacturer.

Field instruments will be checked and calibrated before they are transported to the field. These instruments will be checked and calibrated daily before use. Calibration checks will be performed as noted in Table 3-8 and will be recorded in the field logbook.

Critical spare parts such as electrodes, batteries, and pH probes will be kept on-site to minimize instrument down time. Backup instruments and equipment will be available within on-day shipment to avoid delays in the field schedule.

11.2 Laboratory Instruments

Preventative maintenance procedures for laboratory instrumentation and equipment for TCL organics (including low level TCL organics and landfill gas volatiles) are referenced in the current CLP SOWs OLM01.8 and OLC01.0. Preventative maintenance procedures for laboratory instrumentation and equipment for TAL inorganics (including low level TAL inorganics) are referenced in the current CLP SOW ILM02.0.

Preventative maintenance of laboratory instruments associated with the indicator parameters will be as directed with manufacturer's specifications, instrument operating procedures, and analytical methods. Maintenance is carried out on a regular, scheduled basis, and is documented in the instrument service logbook for each instrument. Emergency repair or scheduled manufacturer's maintenance is provided by an on-site technician or maintenance contract with the factory representatives.

SECTION 12

SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

12.1 Field Measurements

Field data will be assessed by the Site QAO. The Site QAO will review the field results for compliance with the established QC criteria that are specified in the QAPP and SAP. Accuracy of field measurements will be assessed using daily instrument calibration, calibration check, and blank data. Precision will be assessed on the basis of reproducibility by taking multiple readings of a single sample. Data completeness will be calculated as follows:

$$\text{Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100$$

12.2 Laboratory Data

12.2.1 Precision

Precision of laboratory analysis will be assessed by comparing the analytical results between MS/MSD for organic analysis, and laboratory duplicate analyses for inorganic and indicator analysis. The relative percent difference (RPD) will be calculated for each pair of duplicate analysis results as follows:

$$\text{RPD} = \frac{|S - D|}{(S + D)/2} \times 100$$

where, S = first sample value (original or MS value)
D = Second sample value (duplicate or MSD value)

12.2.2 Accuracy

Accuracy of laboratory results will be assessed based on the established QC criteria that are described in Section 3 of this QAPP, using the analytical results of method blanks, matrix spike samples, and field blanks. The percent recovery (%R) will be calculated as follows:

$$\%R = \frac{A - B}{C} \times 100$$

where,
- A = The analyte concentration determined experimentally from the spiked sample;
- B = The background level determined by a separate analysis of the unspiked sample; and
- C = The amount of the spike added.

12.2.3 Completeness

The data completeness of laboratory analysis results will be assessed for compliance with the amount of data required for decision making. Completeness is calculated by the following formula:

$$\text{Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100$$

12.2.4 Sensitivity

The achievement of method detection limits depend on instrument sensitivity and matrix effects. It is important to monitor the instrument sensitivity to ensure the data quality through constant instrument performance. The instrument sensitivity will be monitored through the analysis of method blanks, calibration check samples, and laboratory control samples.

SECTION 13
CORRECTIVE ACTION

Corrective actions may be required for two classes of problems: analytical and equipment problems, and noncompliance problems. Analytical and equipment problems may occur during sampling and sample handling, sample preparation, laboratory instrumental analysis, and data review.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the RPM. If the problem is analytical in nature, information on these problems will be promptly communicated to the U.S. EPA QAS. Implementation of corrective action will be confirmed in writing through the same channels.

Any nonconformance with the established quality control procedures in the QAPP or SAP will be identified and corrected in accordance with the QAPP. The U.S. EPA RPM, or his/her designee will issue a nonconformance report for each nonconformance condition.

Corrective actions will be implemented and documented in the field record book. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by stop-work order by the RPM.

13.1 Sample Collection/Field Measurements

Technical staff and project personnel will be responsible for reporting all suspected technical or QA nonconformances or suspected deficiencies of any activity or issued document by reporting the situation to the Warzyn RI Leader, or his/her designee. This manager will be responsible for

assessing the suspected problems in consultation with the Warzyn Project Manager on making a decision based on the potential for the situation to impact the quality of the data. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the manager.

The manager will be responsible for ensuring that corrective action for nonconformances are initiated by:

- Evaluating all reported nonconformances
- Controlling additional work on nonconforming items
- Determining disposition or action to be taken
- Maintaining a log of nonconformances
- Reviewing nonconformance reports and corrective actions taken
- Ensuring nonconformance reports are included in the final site documentation in the project files

If appropriate, the Warzyn Project Manager will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

Corrective action for field measurements may include:

- Repeat the measurement to check the error
- Check for all proper adjustments for ambient conditions such as temperature
- Check the batteries
- Re-calibration
- Check the calibration
- Replace the instrument or measurement devices

- Stop work (if necessary)

The Warzyn RI Leader, or his/her designee, is responsible for all site activities. In this role, the Warzyn RI Leader, at times is required to adjust the site programs to accommodate the site-specific needs. When it becomes necessary to modify a program, the Warzyn RI Leader notifies the Warzyn Project Manager of the anticipated change and implements the necessary changes after obtaining the approval of the Project Manager. The change in the program will be documented and signed by the appropriate personnel.

The Warzyn RI Leader for the H.O.D. Landfill Site is responsible for the controlling, tracking, and implementation of the identified changes. Reports on all changes will be distributed to all affected parties, including the U.S. EPA RPM, the Warzyn Project Manager, and the WMII Project Manager.

13.2 Laboratory Analysis

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken is dependent on the analysis and the event. Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy
- Blanks contain target analytes above acceptable levels
- Undesirable trends are detected in spike recoveries or RPD between duplicates
- There are unusual changes in detection limits
- Deficiencies are detected by the QA Department during internal or external audits , or from the results of performance evaluation samples
- Inquiries concerning data quality are received

Corrective action procedures are often handled at the bench level, by the analyst who reviews the sample preparation procedure for possible errors and checks the instrument calibration, spike and calibration mixes, instrument sensitivity, etc. If the problem persists, or cannot be identified, the matter is referred to the laboratory supervisor, manager, or QAO for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA Department.

SECTION 14

QUALITY ASSURANCE REPORTS TO MANAGEMENT

Reports will be submitted to the U.S. EPA and Illinois Environmental Protection Agency (IEPA) as described in Section 4.4.7, "Task 7: Reports" of the Work Plan. Reports will consist of monthly progress reports, technical memoranda, and the draft RI report.

Monthly progress reports submitted to the U.S. EPA and IEPA will include:

- A summary of the validated sampling and testing results
- A description of activities completed during the past month, as well as actions which are scheduled for the next month
- A summary of target and actual completion dates for each activity
- Changes in key personnel
- Problems encountered and how they were resolved
- Anticipated problems and recommended solutions

The results of specific RI activities such as the Risk Assessment, and Site Characterization will be submitted to the U.S. EPA and IEPA in the form of Draft Technical Memoranda. Technical Memoranda will include the following:

- Physical and Source Characterization Results
- Contaminant and Migration Pathways Characterization Results
- Baseline Risk Assessment

A draft RI Report summarizing the RI activities will be submitted to the U.S. EPA. The report will characterize the Site and summarize the data collected and conclusions. The RI report will not be considered final until Site characterization activities are complete for the supporting remedial alternatives screening activities and a letter of approval is issued by the U.S. EPA RPM.

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Table 1-1
Sample Type and Estimated Sample Numbers
H.O.D. Landfill RI/FS

Sample[1] Matrix	Lab[2]	No. of Samples	Field Duplicates	Field[3] Blanks	MS/MSD[4]	Total No. Samples	Test[5,6] Parameters	Field Parameters
PHYSICAL INVESTIGATION								
Soil borings	Warzyn Soils	16	2	-	-	18	Grain Size	
SOURCE CHARACTERIZATION								
Soils (clay cap)	Warzyn Soils	10	1	-	-	11	Grain Size, Atterberg Limits, Natural Moisture Content, and Density	
	Core	10	1	-	-	11	Clay Mineralogy	
Sediments/Surface Soils	ETC	5	1	-	2	6	TCL Organics	
	ETC	5	1	-	-	6	TAL Inorganics	
	ETC	5	1	-	-	6	TOC	
	Warzyn Soils	5	1	-	-	6	Grain Size, Natural Moisture Content, and Atterberg Limits	
Leachate	ETC	5	1	1	1	7	TCL Organics	pH, Conductivity,
	ETC	5	1	1	-	7	TAL Inorganics	Temperature, Redox,
	Enseco/RMAL	5	1	1	-	7	Indicators	and Dissolved Oxygen
Landfill Gas	Enseco - Air Toxics	5	1	1	-	7	VOCs	Methane, Oxygen, and Carbon Dioxide
Landfill Soil Borings	Warzyn Soils	10	1	-	-	11	Grain Size	
DEFINE NATURE AND EXTENT OF CONTAMINATION								
Groundwater - Upper	ETC	12	2	2	2	16	TCL Organics	pH, Conductivity,
and Lower Aquifers	ETC	12	2	2	-	16	TAL Inorganics	Temperature, Redox,
	Enseco/RMAL	12	2	2	-	16	Indicators	and Dissolved Oxygen
Municipal/Private Wells	Enseco/RMAL	8	1	1	1	10	LL TCL Organics	pH, Conductivity,
	Enseco/RMAL	8	1	1	-	10	LL TAL Inorganics	Temperature
Surface Water	ETC	3	1	1	1	5	TCL Organics	pH, Conductivity,
	ETC	3	1	1	-	5	TAL Inorganics	Temperature, Redox, and Dissolved Oxygen

Table 1-1
(continued)

Notes:

1. Samples will be considered low concentration, and will be packaged and shipped accordingly.

2. ETC
284 Raritan Center Parkway
Edison, NJ 08818

Warzyn Soils Laboratory
505 Science Drive, Suite C
University Research Park
Madison, WI 53711

Core Laboratories
1875 Monetary Lane
Carrollton, TX 75006

Enseco/RMAL
4955 Yarrow Street
Arvada, CO 80002

Enseco - Air Toxics Laboratory
9537 Telstar Avenue
Suite 118
El Monte, CA 91731

3. A trip blank for VOC analysis will be included with each cooler shipped for aqueous (leachate, groundwater, surface water, municipal well, and private well) samples. Trip blanks are not included in the total number of samples. One trip blank (pre-cleaned SUMMA passivated canister) is required for the sampling of landfill gas vents for volatiles.

4. EXTRA VOLUME REQUIREMENT: Extra volume is required for the MS/MSD quality control requirement (triple volume for VOCs, double volume for SVOCs and Pest/PCBs) for aqueous samples. MS/MSDs are required for soil samples, however do not require that additional volume be collected in the field. Inorganics and general water quality indicator parameters require duplicate and spike analyses, however, do not require additional sample volume to meet the specified QC. MS/MSD samples are not included in the total number of samples.

5. Refer to Tables 3-1 and 3-2 for the TCL organic and TAL inorganic parameter lists and required detection limits. Indicator parameters consist of chloride, sulfate, alkalinity, hardness, nitrate-N, nitrite-N, ammonia-N, TOC, and TDS. See Table 3-5 landfill gas VOC list and detection limits and Table 3-6 for indicator, field measurement, and physical characteristic lists and associated detection limits.

6. Groundwater samples for metals analysis will be field filtered through a 0.45 micron filter prior to the addition of preservatives.

7. LL = Low level detection limits are required. Refer to Tables 3-3 and 3-4 for the low level TCL organic and TAL inorganic parameter lists and required detection limits. Municipal/Private Wells will be sampled by the Lake County Health Department.

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Table 1-2
Sample Quantities, Containers, Preservatives and Packaging Requirements
H.O.D. Landfill RI/FS

Analysis	Bottles and Jars	Preservation	Holding Time[1]	Volume of Samples	Shipping	Normal Packaging[2]
Low Concentration (Organics)						
Groundwater, Surface Water, and Municipal/Private Wells						
TCL Semi-volatiles	Two 1-Liter amber glass bottles	Iced to 4°C.	7 days to extraction, analysis within 40 days after extraction.	Fill bottle to neck	Shipped daily by overnight carrier	Vermiculite
TCL Pesticides/PCBs	Two 1-Liter amber glass bottles	Iced to 4°C.	7 days to extraction, analysis within 40 days after extraction.	Fill bottle to neck	Shipped daily by overnight carrier	Vermiculite
TCL Volatiles	Three 40-mL volatile organic analysis (VOA) vials (Four for municipal/private well samples).	1:1 HCL (2 drops/ vial), iced to 4°C.	14 days	Fill completely no headspace	Shipped daily by overnight carrier	Vermiculite
Leachate						
TCL Semi-volatiles	Two 1-Liter amber glass bottles	Iced to 4°C.	7 days to extraction, analysis within 40 days after extraction.	Fill bottle to neck	Shipped daily by overnight carrier	Vermiculite
TCL Pesticides/PCBs	Two 1-Liter amber glass bottles	Iced to 4°C.	7 days to extraction, analysis within 40 days after extraction.	Fill bottle to neck	Shipped daily by overnight carrier	Vermiculite
TCL Volatiles	Three 40-mL VOA vials	Iced to 4°C.	7 days	Fill completely no headspace	Shipped daily by overnight carrier	Vermiculite

Table 1-2
(continued)

Analysis	Bottles and Jars	Preservation	Holding Time[1]	Volume of Samples	Shipping	Normal Packaging[2]
Soil and Sediment						
TCL Semivolatiles	One 8-oz wide mouth glass jar	Iced to 4°C.	14 days to extraction, analysis within 40 days after extraction.	Fill 3/4 full	Shipped daily by overnight carrier	Vermiculite
TCL Pesticides/PCBs	One 8-oz wide mouth glass jar	Iced to 4°C.	14 days to extraction, analysis within 40 days after extraction.	Fill 3/4 full	Shipped daily by overnight carrier	Vermiculite
TCL Volatiles	Two 4-oz wide mouth glass jars	Iced to 4°C.	14 days	Fill completely no headspace	Shipped daily by overnight carrier	Vermiculite
Landfill Gas						
Volatiles	One 6-liter SUMMA passivated canister	Iced to 4°C.	Not established	Fill as described in procedure	Shipped daily by overnight carrier	Vermiculite
Low Concentration (Inorganics)						
Groundwater						
Metals	One 1-liter high density polyethylene bottle	Field filter through 0.45 um filter. HNO_3 to pH<2. Iced to 4 C.	180 days (28 days for mercury)	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
Cyanide	One 1-liter high density polyethylene bottle	Add NaOH to pH>12. Iced to 4°C.	14 days	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
Private/Municipal Wells						
Metals	One 1-liter high density polyethylene bottle	HNO_3 to pH<2. Iced to 4°C.	180 days (28 days for mercury)	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
Cyanide	One 1-liter high density polyethylene bottle	Add NaOH to pH>12. Iced to 4°C.	14 days	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite

Table 1-2
(continued)

Analysis	Bottles and Jars	Preservation	Holding Time[1]	Volume of Samples	Shipping	Normal Packaging[2]
Surface Water and Leachate						
Metals	One 1-liter high density polyethylene bottle	HNO_3 to pH<2. Iced to 4°C.	180 days (28 days for mercury)	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
Cyanide	One 1-liter high density polyethylene bottle	Add NaOH to pH>12. Iced to 4°C.	14 days	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
Soil and Sediment						
Metals and Cyanide	One 8-oz wide mouth glass jar	Iced to 4°C.	180 days (28 days for mercury, 14 days for cyanide)	Fill 3/4 full	Shipped daily by overnight carrier	Vermiculite (Med in cans/ vermiculite)
Water Quality Indicator Parameters						
Groundwater and Leachate						
Nitrate-N, Ammonia, and TOC	One 1-liter high density polyethylene bottle	H_2SO_4 to pH<2. Iced to 4°C.	28 days	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
Hardness	One 500-ml high density polyethylene bottle	HNO_3 to pH<2. Iced to 4°C.	180 days	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
Alkalinity, Chloride, Sulfate, and Nitrite-N	One 1-liter high density polyethylene bottle	Iced to 4°C.	28 days (14 days alkalinity, 48 hours nitrite)	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
TDS	One 500-mL polyethylene bottle	Field filter through 0.45 um filter. Iced to 4°C	7 days	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite

Table 1-2
(continued)

Analysis	Bottles and Jars	Preservation	Holding Time[1]	Volume of Samples	Shipping	Normal Packaging[2]
Physical Characteristics						
Soil						
Grain size, moisture content, density, and atterberg limits	Four 8-oz wide mouth glass jars	NONE	Not established	Fill 3/4 full	Ship by carrier	Vermiculite
Clay Mineralogy	Two 8-oz wide mouth glass jars	NONE	Not established	Fill 3/4 full	Ship by carrier	Vermiculite
TOC	One 4-oz wide mouth glass jar	Iced to 4°C.	28 days	Fill 3/4 full	Ship by carrier	Vermiculite

Notes:
1. These are technical holding times, which are started from the day of sample collection.
2. The packing material should completely cushion the sample bottles - botttom, sides and top.

[Wautlet.mass11]60953 QAPP Table 1-2
CAW/caw/JAH

Table 1-3
Summary of Data Generating Activities and Associated Quality Objectives
H.O.D. Landfill RI/FS

Activity	Description	Intended Data Usages	Parameters	Data Quality Objective	Anticipated No. of Investigative Samples
PHYSICAL INVESTIGATION					
Hydrogeologic Evaluation	Eight borings taken in areas specified in the Work Plan.	Confirm glacial stratigraphy.	Grain Size	Level III data	16
SOURCE CHARACTERIZATION					
Landfill Cap Evaluation	Collect samples of the clay cap.	Evaluate the effectiveness of the existing cap. Landfill cap evaluation will provide data used to perform percolation analysis.	Grain Size, Atterberg Limits, Natural Moisture Content, Density, and Clay Mineralogy	Level III data	10
On-site Surface Soil and Sediment Sampling	Collection of on-site surface soil/sediments in likely soil/sediment deposition areas.	Contaminant characterization.	TCL, TAL Grain Size, Atterberg Limits, TOC, and Natural Moisture Content	Level IV data Level III data	5
Leachate Sampling	Collection of leachate samples.	Chemically characterize the leachate composition.	TCL, TAL Indicators[1] pH, Conductivity, Temperature, Dissolved Oxygen, and Redox (field)	Level IV data Level V data Level II data	5 5 5
Landfill Gas Probe Installation	Installation of multi-stage landfill gas probes in the same borehole as each new leachate piezometer.	Assess methane gas production.	Methane, Oxygen, and Carbon Dioxide (field)	Level II data	up to 39 measurements per round
Landfill Gas Sampling	Collection of landfill gas from leachate piezometers and/or gas well flares.	Chemically characterize landfill gas, and assess the potential for contamination of air.	VOCs Methane, Oxygen, and Carbon Dioxide (field)	Level V data Level II data	5 5

Table 1-3
(continued)

Activity	Description	Intended Data Usages	Parameters	Data Quality Objective	Anticipated No. of Investigative Samples
Landfill Borings	Drill approximately five landfill borings along the southern perimeter of the "old" landfill.	Determine the subsurface conditions and evaluate the feasibility of constructing a barrier along the perimeter of the landfill to contain leachate.	Grain Size	Level III data	10
DEFINE NATURE AND EXTENT OF CONTAMINATION					
Groundwater Monitoring	Obtain water level measurements. Conduct in-situ hydraulic conductivity tests. Install new wells, and sample and analyze the existing and new monitoring wells.	Evaluate potential groundwater flow paths, confirm hydraulic conductivity results with previous data, assess groundwater contamination migration pathway, and characterize groundwater quality.	TCL, TAL Indicators[1] pH, Conductivity, Temperature, Dissolved Oxygen, and Redox (field)	Level IV data Level V data Level II data	16 16 16
Municipal/Private Well Monitoring	Sampling private and municipal wells in the vicinity of the Site. Sampling to be performed by Lake County Health Department.	Characterize the water quality.	Low level TCL and TAL pH, Conductivity, and Temperature	Level IV data Level II data	8 8
Hydrologic Evaluation/Surface Water Sampling	Measure the surface water levels, flow, and inspect the Sequoit Creek banks.	Evaluate the potential for surface water contamination and characterize the surface water quality.	TCL, TAL pH, Conductivity, Temperature, Dissolved Oxygen, and Redox (field)	Level IV data Level II data	3 3

Notes:
1. Indicator parameters consist of: chloride, sulfate, alkalinity, hardness, nitrate-N, nitrite-N, ammonia, TOC, and TDS.
2. Grain Size = Sieve plus Hydrometer Analyses

[Wautlet.mass11]60953 QAPP Table 1-3
CAW/caw/JAH
60953 revised 5/11/92

Table 3-1
Target Compound List
and Contract Required Quantitation Limits - Organics

	Compound	Quantitation Limits[1] Water (ug/l)	Low Soil (ug/kg)	Medium Soil (ug/kg)
	Volatiles			
1.	Chloromethane	10	10	1200
2.	Bromomethane	10	10	1200
3.	Vinyl chloride	10	10	1200
4.	Chloroethane	10	10	1200
5.	Methylene chloride	10	10	1200
6.	Acetone	10	10	1200
7.	Carbon disulfide	10	10	1200
8.	1,1-Dichloroethene	10	10	1200
9.	1,1-Dichloroethane	10	10	1200
10.	1,2-Dichloroethene (total)	10	10	1200
11.	Chloroform	10	10	1200
12.	1,2-Dichloroethane	10	10	1200
13.	2-Butanone	10	10	1200
14.	1,1,1-Trichloroethane	10	10	1200
15.	Carbon tetrachloride	10	10	1200
16.	Bromodichloromethane	10	10	1200
17.	1,2-Dichloropropane	10	10	1200
18.	cis-1,3-Dichloropropene	10	10	1200
19.	Trichloroethene	10	10	1200
20.	Dibromochloromethane	10	10	1200
21.	1,1,2-Trichloroethane	10	10	1200
22.	Benzene	10	10	1200
23.	trans-1,3-Dichloropropene	10	10	1200
24.	Bromoform	10	10	1200
25.	4-Methyl-2-pentanone	10	10	1200
26.	2-Hexanone	10	10	1200
27.	Tetrachloroethene	10	10	1200
28.	Toluene	10	10	1200
29.	1,1,2,2-Tetrachloroethane	10	10	1200
30.	Chlorobenzene	10	10	1200
31.	Ethylbenzene	10	10	1200
32.	Styrene	10	10	1200
33.	Xylenes (total)	10	10	1200

Table 3-1
(continued)

	Compound	Quantitation Limits[1] Water (ug/l)	Low Soil (ug/kg)	Medium Soil (ug/kg)
	Semi-volatiles			
34.	Phenol	10	330	10000
35.	bis(2-Chloroethyl) ether	10	330	10000
36.	2-Chlorophenol	10	330	10000
37.	1,3-Dichlorobenzene	10	330	10000
38.	1,4-Dichlorobenzene	10	330	10000
39.	1,2-Dichlorobeznene	10	330	10000
40.	2-Methylphenol	10	330	10000
41.	2,2'-oxybis-(1-Chloropropane)	10	330	10000
42.	4-Methylphenol	10	330	10000
43.	n-Nitroso-di-n-dipropylamine	10	330	10000
44.	Hexachloroethane	10	330	10000
45.	Nitrobenzene	10	330	10000
46.	Isophorone	10	330	10000
47.	2-Nitrophenol	10	330	10000
48.	2,4-Dimethylphenol	10	330	10000
49.	bis(2-Chloroethoxy) methane	10	330	10000
50.	2,4-Dichlorophenol	10	330	10000
51.	1,2,4-Trichlorobenzene	10	330	10000
52.	Naphthalene	10	330	10000
53.	4-Chloroaniline	10	330	10000
54.	Hexachlorobutadiene	10	330	10000
55.	4-Chloro-3-methylphenol	10	330	10000
56.	2-Methylnaphthalene	10	330	10000
57.	Hexachlorocyclopentadiene	10	330	10000
58.	2,4,6-Trichlorophenol	10	330	10000
59.	2,4,5-Trichlorophenol	25	800	25000
60.	2-Chloronaphthalene	10	330	10000
61.	2-Nitroaniline	25	800	25000
62.	Dimethylphthalate	10	330	10000
63.	Acenaphthylene	10	330	10000
64.	2,6-Dinitrotoluene	10	330	10000
65.	3-Nitroaniline	25	800	25000
66.	Acenaphthene	10	330	10000
67.	2,4-Dinitrophenol	25	800	25000
68.	4-Nitrophenol	25	800	25000

Table 3-1
(continued)

	Compound	Quantitation Limits[1] Water (ug/l)	Low Soil (ug/kg)	Medium Soil (ug/kg)
69.	Dibenzofuran	10	330	10000
70.	2,4-Dinitrotoluene	10	330	10000
71.	Diethylphthalate	10	330	10000
72.	4-Chlorophenyl-phenyl ether	10	330	10000
73.	Fluorene	10	330	10000
74.	4-Nitroaniline	25	800	25000
75.	4,6-Dinitro-2-methylphenol	25	800	25000
76.	n-Nitrosodiphenylamine	10	330	10000
77.	4-Bromophenyl-phenyl ether	10	330	10000
78.	Hexachlorobenzene	10	330	10000
79.	Pentachlorophenol	25	800	25000
80.	Phenanthrene	10	330	10000
81.	Anthracene	10	330	10000
82.	Carbazole	10	330	10000
83.	Di-n-butylphthalate	10	330	10000
84.	Fluoranthene	10	330	10000
85.	Pyrene	10	330	10000
86.	Butylbenzylphthalate	10	330	10000
87.	3,3'-Dichlorobenzidine	10	330	10000
88.	Benzo(a)anthracene	10	330	10000
89.	Chrysene	10	330	10000
90.	bis(2-Ethylhexyl)phthalate	10	330	10000
91.	Di-n-ocylphthalate	10	330	10000
92.	Benzo(b)fluoranthene	10	330	10000
93.	Benzo(k)fluoranthene	10	330	10000
94.	Benzo(a)pyrene	10	330	10000
95.	Indeno(1,2,3-cd)pyrene	10	330	10000
96.	Dibenzo(a,h)anthracene	10	330	10000
97.	Benzo(g,h,i)perylene	10	330	10000

Table 3-1
(continued)

	Compound	Quantitation Limits[1] Water (ug/l)	Low Soil (ug/kg)	Medium Soil (ug/kg)
	Pesticides/PCBs			
98.	alpha-BHC	0.05	1.7	1.7
99.	beta-BHC	0.05	1.7	1.7
100.	delta-BHC	0.05	1.7	1.7
101.	gamma-BHC (Lindane)	0.05	1.7	1.7
102.	Heptachlor	0.05	1.7	1.7
103.	Aldrin	0.05	1.7	1.7
104.	Heptachlor epoxide	0.05	1.7	1.7
105.	Endosulfan I	0.05	1.7	1.7
106.	Dieldrin	0.10	3.3	3.3
107.	4,4'-DDE	0.10	3.3	3.3
108.	Endrin	0.10	3.3	3.3
109.	Endosulfan II	0.10	3.3	3.3
110.	4,4'-DDD	0.10	3.3	3.3
111.	Endosulfan sulfate	0.10	3.3	3.3
112.	4,4'-DDT	0.10	3.3	3.3
113.	Methoxychlor	0.50	17	17
114.	Endrin ketone	0.10	3.3	3.3
115.	Endrin aldehyde	0.10	3.3	3.3
116.	alpha-Chlordane	0.05	1.7	1.7
117.	gamma-Chlordane	0.05	1.7	1.7
118.	Toxaphene	5.0	170	170
119.	Aroclor-1016	1.0	33	33
120.	Aroclor-1221	2.0	67	67
121.	Aroclor-1232	1.0	33	33
122.	Aroclor-1242	1.0	33	33
123.	Aroclor-1248	1.0	33	33
124.	Aroclor-1254	1.0	33	33
125.	Aroclor-1260	1.0	33	33

Notes:

1. Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on a dry weight basis as required by the method, will be higher.

[Wautlet.mass11]60953 QAPP Table 3-1
CAW/caw/

Table 3-2
Target Analyte List and
Contract Required Detection Limits - Inorganics

	Analyte	Contract Required [1,2,3] Detection Limit (ug/L)
1.	Aluminum	200
2.	Antimony	60
3.	Arsenic	10
4.	Barium	200
5.	Beryllium	5
6.	Cadmium	5
7.	Calcium	5000
8.	Chromium	10
9.	Cobalt	50
10.	Copper	25
11.	Iron	100
12.	Lead	3
13.	Magnesium	5000
14.	Manganese	15
15.	Mercury	0.2
16.	Nickel	40
17.	Potassium	5000
18.	Selenium	5
19.	Silver	10
20.	Sodium	5000
21.	Thallium	10
22.	Vanadium	50
23.	Zinc	20
24.	Cyanide	10

Notes:

1. Subject to the restrictions specified in the first page of Part G, Section IV of Exhibit D (Alternate Methods - Catastrophic Failure) any analytical method specified in SOW ILM02.0, Exhibit D, may be utilized as long as the documented instrument or method detection limits meet the Contract Required Detection Limit (CRDL) requirements. Higher detection limits may only be used in the following circumstance:

   If the sample concentration exceeds five times the detection limit of the instrument or method in use, the values

Table 3-2
(continued)

may be reported even though the instrument or method detection limit may not equal the CRDL. This is illustrated in the example below:

For lead:
Method in use = ICP
Instrument Detection Limit (IDL) = 40
Sample concentration = 200
CRDL = 3

The value of 200 may be reported even though the instrument detection limit is greater than the CRDL. The instrument or method detection limit must be documented as described in SOW ILM02.0, Exhibit E.

2. The CRDL are the instrument detection limits obtained in pure water that must be met using the procedure in Exhibit E. The detection limits for samples may be considerably higher depending on the sample matrix. Soil/sediment detection limits are approximately 200 times the CRDLs noted for water, and may vary as the soil/sediment results are reported on a dry weight basis. Soil/sediment results are reported in mg/kg.

3. This table is extracted from the Inorganic SOW ILM02.0 (Exhibit C).

[Wautlet.mass11]60953 QAPP Table 3-2
CAW/caw/JAH

Table 3-3
Target Compound List
and Contract Required Quantitation Limits
Low Concentration Water Organics

	Compound	Quantitation Limits[1] Water (ug/L)
	Volatiles	
1.	Chloromethane	1
2.	Bromomethane	1
3.	Vinyl chloride	1
4.	Chloroethane	1
5.	Methylene chloride	2
6.	Acetone	5
7.	Carbon disulfide	1
8.	1,1-Dichloroethene	1
9.	1,1-Dichloroethane	1
10.	cis-1,2-Dichloroethene	1
11.	trans-1,2-Dichloroethene	1
12.	Chloroform	1
13.	1,2-Dichloroethane	1
14.	2-Butanone	5
15.	Bromochloromethane	1
16.	1,1,1-Trichloroethane	1
17.	Carbon tetrachloride	1
18.	Bromodichloromethane	1
19.	1,2-Dichloropropane	1
20.	cis-1,3-Dichloropropene	1
21.	Trichloroethene	1
22.	Dibromochloromethane	1
23.	1,1,2-Trichloroethane	1
24.	Benzene	1
25.	trans-1,3-Dichloropropene	1
26.	Bromoform	1
27.	4-Methyl-2-pentanone	5
28.	2-Hexanone	5
29.	Tetrachloroethene	1
30.	1,1,2,2-Tetrachloroethane	1
31.	1,2-Dibromomethane	1
32.	Toluene	1
33.	Chlorobenzene	1
34.	Ethylbenzene	1
35.	Styrene	1

Table 3-3
(continued)

	Compound	Quantitation Limits[1] Water (ug/L)
36.	Xylenes (total)	1
37.	1,3-Dichlorobenzene	1
38.	1,4-Dichlorobenzene	1
39.	1,2-Dichlorobenzene	1
40.	1,2-Dibromo-3-chloropropane	1
	Semi-volatiles	
41.	Phenol	5
42.	bis(2-Chloroethyl) ether	5
43.	2-Chlorophenol	5
44.	2-Methylphenol	5
45.	2,2'-oxybis-(1-Chloropropane)	5
46.	4-Methylphenol	5
47.	n-Nitroso-di-n-dipropylamine	5
48.	Hexachloroethane	5
49.	Nitrobenzene	5
50.	Isophorone	5
51.	2-Nitrophenol	5
52.	2,4-Dimethylphenol	5
53.	bis(2-Chloroethoxy) methane	5
54.	2,4-Dichlorophenol	5
55.	1,2,4-Trichlorobenzene	5
56.	Naphthalene	5
57.	4-Chloroaniline	5
58.	Hexachlorobutadiene	5
59.	4-Chloro-3-methylphenol	5
60.	2-Methylnaphthalene	5
61.	Hexachlorocyclopentadiene	5
62.	2,4,6-Trichlorophenol	5
63.	2,4,5-Trichlorophenol	20
64.	2-Chloronaphthalene	5
65.	2-Nitroaniline	20
66.	Dimethylphthalate	5
67.	Acenaphthylene	5
68.	2,6-Dinitrotoluene	5
69.	3-Nitroaniline	20
70.	Acenaphthene	5

Table 3-3
(continued)

	Compound	Quantitation Limits[1] Water (ug/L)
71.	2,4-Dinitrophenol	20
72.	4-Nitrophenol	20
73.	Dibenzofuran	5
74.	2,4-Dinitrotoluene	5
75.	Diethylphthalate	5
76.	4-Chlorophenyl-phenyl ether	5
77.	Fluorene	5
78.	4-Nitroaniline	20
79.	4,6-Dinitro-2-methylphenol	20
80.	n-Nitrosodiphenylamine	5
81.	4-Bromophenyl-phenyl ether	5
82.	Hexachlorobenzene	5
83.	Pentachlorophenol	20
84.	Phenanthrene	5
85.	Anthracene	5
86.	Di-n-butylphthalate	5
87.	Fluoranthene	5
88.	Pyrene	5
89.	Butylbenzylphthalate	5
90.	3,3'-Dichlorobenzidine	5
91.	Benzo(a)anthracene	5
92.	Chrysene	5
93.	bis(2-Ethylhexyl)phthalate	5
94.	Di-n-ocylphthalate	5
95.	Benzo(b)fluoranthene	5
96.	Benzo(k)fluoranthene	5
97.	Benzo(a)pyrene	5
98.	Indeno(1,2,3-cd)pyrene	5
99.	Dibenzo(a,h)anthracene	5
100.	Benzo(g,h,i)perylene	5
	Pesticides/PCBs	
101.	alpha-BHC	0.01
102.	beta-BHC	0.01
103.	delta-BHC	0.01
104.	gamma-BHC (Lindane)	0.01
105.	Heptachlor	0.01

Table 3-3
(continued)

	Compound	Quantitation Limits[1] Water (ug/L)
106.	Aldrin	0.01
107.	Heptachlor epoxide	0.01
108.	Endosulfan I	0.01
109.	Dieldrin	0.02
110.	4,4'-DDE	0.02
111.	Endrin	0.02
112.	Endosulfan II	0.02
113.	4,4'-DDD	0.02
114.	Endosulfan sulfate	0.02
115.	4,4'-DDT	0.02
116.	Methoxychlor	0.10
117.	Endrin ketone	0.02
118.	Endrin aldehyde	0.02
119.	alpha-Chlordane	0.01
120.	gamma-Chlordane	0.01
121.	Toxaphene	1.0
122.	Aroclor-1016	0.20
123.	Aroclor-1221	0.20
124.	Aroclor-1232	0.40
125.	Aroclor-1242	0.20
126.	Aroclor-1248	0.20
127.	Aroclor-1254	0.20
128.	Aroclor-1260	0.20

Notes:
1. This table was extracted from the low level detection SOW OLC01.0.

[Wautlet.mass11]60953 QAPP Table 3-3
CAW/caw/JAH

Table 3-4
Target Analyte List and
Required Detection Limits
Low Concentration Water Inorganics

	Analyte	Required Detection Limit (ug/L)
1.	Aluminum	10
2.	Antimony	5
3.	Arsenic	0.5
4.	Barium	200
5.	Beryllium	0.5
6.	Cadmium	5
7.	Calcium	1000
8.	Chromium	10
9.	Cobalt	50
10.	Copper	25
11.	Iron	100
12.	Lead	2
13.	Magnesium	1000
14.	Manganese	15
15.	Mercury	0.2
16.	Nickel	40
17.	Potassium	1000
18.	Selenium	5
19.	Silver	10
20.	Sodium	1000
21.	Thallium	10
22.	Vanadium	50
23.	Zinc	20
24.	Cyanide	10

Table 3-5
Landfill Gas Volatile Organic List and
Required Detection Limits

Volatile Compound	Reporting Limit ppb (vol/vol)
Dichlorodifluoromethane	2
Chloromethane	2.5
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	2
Vinyl Chloride	2.5
Bromomethane	3
Chloroethane	5
Trichlorofluoromethane	1
cis-1,2-Dichloroethene	2
Carbon Disulfide	10
1,1,2-Trichloro-1,2,2-Trifluoroethane	2
Acetone	10
Methylene Chloride	4
trans-1,2-Dichloroethene	4
Hexane	8
1,1-Dichloroethane	2.5
Vinyl Acetate	2.5
1,1-Dichloroethene	2
2-Butanone	3
Chloroform	2
1,1,1-Trichloroethane	2
Carbon Tetrachloride	2
Benzene	3
1,2-Dichloroethane	2
Trichloroethene	2.5
1,2-Dichloropropane	8
1,4-Dioxane	7
Bromodichloromethane	2
cis-1,3-Dichloropropene	3
4-Methyl-2-Pentanone	3
Toluene	3
trans-1,3-Dichloropropene	3
1,1,2-Trichloroethane	3
Tetrachloroethene	3
2-Hexanone	5
Dibromochloromethane	3
1,2-Dibromomethane	2
Chlorobenzene	2.5
Ethylbenzene	2.5
Total Xylenes	5
Styrene	7

Table 3-5
(continued)

Volatile Compound	Reporting Limit ppb (vol/vol)
Bromoform	2
1,1,2,2-Tetrachloroethane	4
Benzyl Chloride	2
4-Ethyl Toluene	4
1,3,5-Trimethylbenzene	2.5
1,2,4-Trimethylbenzene	3
1,3-Dichlorobenzene	3
1,4-Dichlorobenzene	4
1,2-Dichlorobenzene	5
1,2,4-Trichlorobenzene	7
Hexachlorobutadiene	5

[Wautlet.mass11]60953 QAPP Table 3-5
CAW/caw/JAH

Table 3-6
Indicators, Field Measurements, and Physical Characteristics
Parameter Lists and Required Detection Limits

Indicator Parameter	Required Detection Limit (mg/L)
Alkalinity	5
Chloride	3
Hardness	5
Sulfate	5
Total Organic Carbon	0.1
Total Dissolved Solids	10
Nitrate Nitrogen	0.10
Nitrite Nitrogen	0.01
Ammonia Nitrogen	0.10

Field Measurement Parameter	Required Detection Limit
pH (s.u.)	to nearest 0.05 pH unit
Conductivity @25 Deg. C (umhos/cm)	10
Temperature (Deg. C)	to nearest 0.5 degree
Redox Potential (mV)	to nearest 1 mV
Dissolved Oxygen (mg/L)	1
Oxygen (%)	1
Methane (%)	1
Carbon Dioxide (%)	1

Physical Characteristic Parameter	Required Detection Limit
Grain Size	-
Atterberg Limits	-
Natural Moisture Content (%)	0.1
Density	-
Total Organic Carbon (mg/kg)	100
Clay Mineralogy by X-ray Diffraction (weight percent of fraction analyzed)	1

Table 3-7
Summary of Quality Control Requirements
H.O.D. Landfill Site RI/FS

Parameter	Audit[1]	Frequency[2]	Quality Control[3] Criteria
TCL Organics	Requirements per OLM01.8 (or most current)		
TAL Inorganics	Requirements per ILM02.0 (or most current)		
Low Level TCL Organics	Requirements per OLC01.0 (or most current)		
Low Level TAL Inorganics	Requirements per ILM02.0 (or most current)		
Alkalinity, Chloride, Hardness, Sulfate, Nitrate Nitrogen, Nitrite Nitrogen, and TOC			
	ICB/CCB	After calibration, every 10 samples, and at the end of the run.	< DL
	ICV/CCV	After calibration, every 10 samples, and at the end of the run.	90 - 110 % Recovery
	EPA QC Reference Standard	1 per set	80 - 120 % Recovery
	Duplicate	1 per 10 samples	10% RPD (± 2xDL if sample concentration is <5 x DL)
	Matrix Spike	1 per 10 samples	85 - 115 % Recovery
Total Dissolved Solids			
	Lab Blank	1 per set	< DL
	EPA QC Reference Standard	1 per set	80 - 120 % Recovery
	Duplicate	1 per 10 samples	10% RPD (± 2xDL if sample concentration is <5 x DL)

Table 3-7
(continued)

Parameter	Audit[1]	Frequency[2]	Quality Control[3] Criteria
Ammonia Nitrogen			
	ICB/CCB	After calibration, every 10 samples, and at the end of the run.	< DL
	ICV/CCV	After calibration, every 10 samples, and at the end of the run.	90 - 110 % Recovery
	PB	1 per set	< DL
	EPA QC Reference Standard	1 per set	80 - 120 % Recovery
	Duplicate	1 per 10 samples	10% RPD (± 2xDL if sample concentration is <5 x DL)
	Matrix Spike	1 per 10 samples	85 - 115 % Recovery
Grain Size, Natural Moisture Content, Atterberg Limits, and Density			
	Lab Duplicate	1 per 10 samples	40% RPD or <2% by weight
pH (Field)			
	Check Standard	1 per 10 samples	± 0.05 pH unit of buffer selection
	Duplicate	1 per 10 samples	± 0.2 pH unit
Specific Conductance (Field)			
	Check Standard	1 per 10 samples	± 5% of standard
	Duplicate	1 per 10 samples	15% RPD (± 2xDL if sample concentration is <5 x DL)
Dissolved Oxygen (Field)			
	Duplicate	1 per 10 samples	20% RPD
Redox Potential (Field)			
	Check Standard	1 per 10 samples	± 10 MV of true value
	Duplicate	1 per 10 samples	20% RPD

Table 3-7
(continued)

Parameter	Audit[1]	Frequency[2]	Quality Control[3] Criteria
Methane, Oxygen and Carbon Dioxide (Field)			
	Duplicate	1 per 10 samples	20% RPD

Notes:

1. ICB/CCB = Initial and Continuing Calibration Blank, ICV/CCV = Initial and Continuing Calibration Check Standard, and PB = Preparation Blank. The preparation blank will be carried through the entire procedure including any digestion/distillation steps. Instruments are calibrated daily. Reanalysis and/or recalibration is triggered when quality control criteria are not met.

2. Frequencies apply to each individual matrix.

3. DL = Detection Limit and RPD = Relative Percent Difference. Refer to Tables 3-1, 3-2, 3-3, 3-4, 3-5, and 3-6 for required detection limits for each analyte.

[wautlet.mass11]60953 QAPP Table 3-7
CAW/caw/JAH

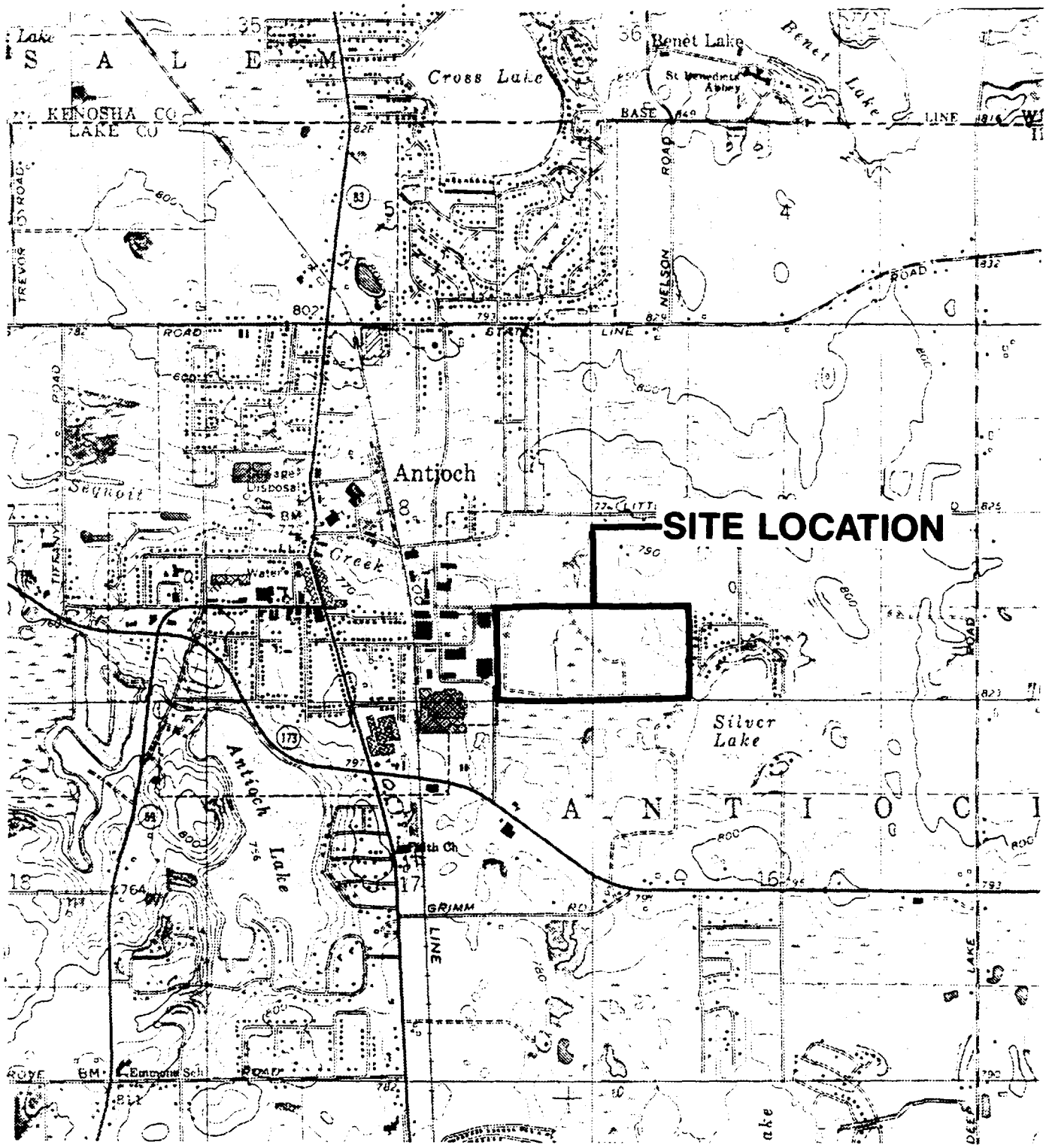
Table 7-1
Summary of Analytical Methods
H.O.D. Landfill RI/FS

Parameter	SOP[1]	Method[2]
TCL Volatiles	SOW OLM01.8	EPA 624
TCL Semi-volatiles	SOW OLM01.8	EPA 625
TCL Pesticides/PCBs	SOW OLM01.8	EPA 608
TCL Volatiles (Low level detection)	SOW OLC01.0	EPA 524.2
TCL Semi-volatiles (Low level detection)	SOW 0LC01.0	EPA 625
TCL Pesticides/PCBs (Low level detection)	SOW 0LC01.0	EPA 608
Landfill Gas Volatiles	CRL-LM-7001	EPA Compendium Method TO-14
TAL Inorganics	SOW ILM02.0	EPA 200.7 (ICP) EPA 200 (AA) EPA 245 (Mercury) EPA 335 (Cyanide)
TAL Inorganics (Low level detection)	LE-RMA-2000 (ICP), LM-RMA-2033 (AA), and SOW ILM02.0	EPA 200.7 (ICP) EPA 200 (AA) EPA 245 (Mercury) EPA 335 (Cyanide)
Alkalinity	LM-RMA-1071	EPA 120.1
Hardness	LM-RMA-1090	EPA 130.2
Ammonia Nitrogen	LM-RMA-1114	EPA 350.1
Total Dissolved Solids	LM-RMA-1060	EPA 160.1
Chloride	LM-RMA-1025	EPA 300.0
Sulfate	LM-RMA-1025	EPA 300.0
Nitrate Nitrogen	LM-RMA-1025	EPA 300.0
Nitrite Nitrogen	LM-RMA-1041	EPA 354.1
Total Organic Carbon (water)	LM-RMA-1066	EPA 415.1
Total Organic Carbon (soil)	ED522902	EPA 415.1 and SW846 9060

Table 7-1
(continued)

Field Measurements		
pH	pH	EPA 150.1
Specific conductance	CONDYSI	EPA 120.1
Temperature	TEMPFC	EPA 170.1
Redox potential	REDOX	Bechman pH Meter Instruction Manual
Dissolved Oxygen	YSIDO	YSI Meter Instruction Manual
Methane and Oxygen	GASTECHTOR	Gastech Model 1939 Instruction Manual
Carbon Dioxide	-	Sensidyne Instruction Manual
Physical Characteristics		
Grain Size Analysis	-	ASTM D422, D1140
Atterberg Limits	-	ASTM D4318, D427
Natural Moisture Content	-	ASTM D2216
Density	-	ASTM D854
Clay Mineralogy	-	Refer to SOP in Appendix B

[wautlet.mass11]60953 QAPP Table 7-1
CAW/caw/JAH



QUALITY CONTROL

	INITIALS	DATE		INITIALS	DATE
Drafting Standards	DLF	10-22-90	PM	GEP	10-22-90
Lead Professional	AJS		Division	DWH	
Section	RM		Other		

north

0 2000 4000

SCALE IN FEET

NOTES

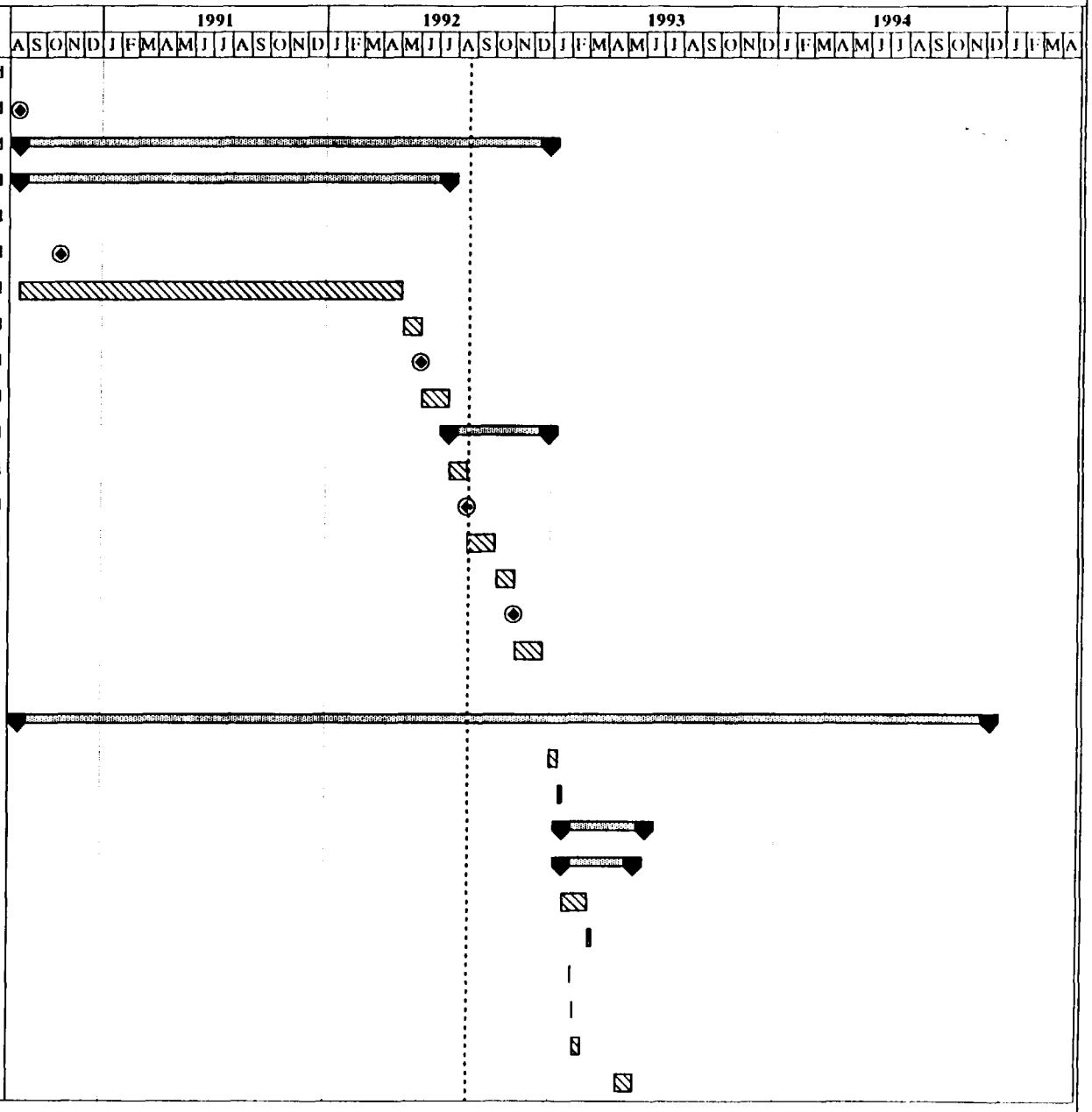
1. SITE LOCATION MAP DEVELOPED FROM THE ANTIOCH, ILLINOIS 7 1/2 MINUTE U.S.G.S. TOPOGRAPHIC QUADRANGLE MAP, DATED 1960, PHOTOREVISED 1972.

FIGURE 1-1

WARZYN	SITE LOCATION MAP	Drawn HLH	Checked RJM	App'd. AJS
	PRELIMINARY SITE EVALUATION REPORT H.O.D. LANDFILL, RI/FS ANTIOCH, ILLINOIS	Revisions		Date 10/24/90 60953 A1

FIGURE 1-2
H.O.D. LANDFILL RI/FS
ANTIOCH, ILLINOIS

ID	Name	Duration
1	HOD LANDFILL RI/FS	0ed
2	Admin. Order by Consent Signed	0d
3	PLANNING DOCUMENTS	862.38ed
4	TASK 1: Work Plan	699ed
5	Prepare Draft Work Plans (WPs)	1d
6	Draft WPs (PSER/TS) to EPA	0d
7	EPA Review/Comment	45ed
8	Revise PSER/TS	30ed
9	Final PSER/TS to EPA	0d
10	EPA Review/Approve PSER/TS	45ed
11	TASK 2: Project Plans	162.38ed
12	Prepare draft Project Plans (PPs)	30ed
13	Draft PPs to EPA	0d
14	EPA Review/Comment on PPs	45ed
15	Revise PPs	30ed
16	Final PPs to EPA	0d
17	EPA Review/Approve PPs	45ed
18	Meeting w/ WMII & EPA	1d
19	REMEDIAL INVESTIGATION	1575ed
20	Mobilization to Site	10d
21	Prepare Staging Area	1w
22	TASK 3: Site Investigation (Phase I)	134.38ed
23	Physical Characterization	116.38ed
24	Hydrogeologic Eval.	30d
25	Hydrologic Evaluation	5d
26	Soil/Sediment Evaluation	2d
27	Air Evaluation	1d
28	Human Population Evaluation	2w
29	Ecological Evaluation	4w



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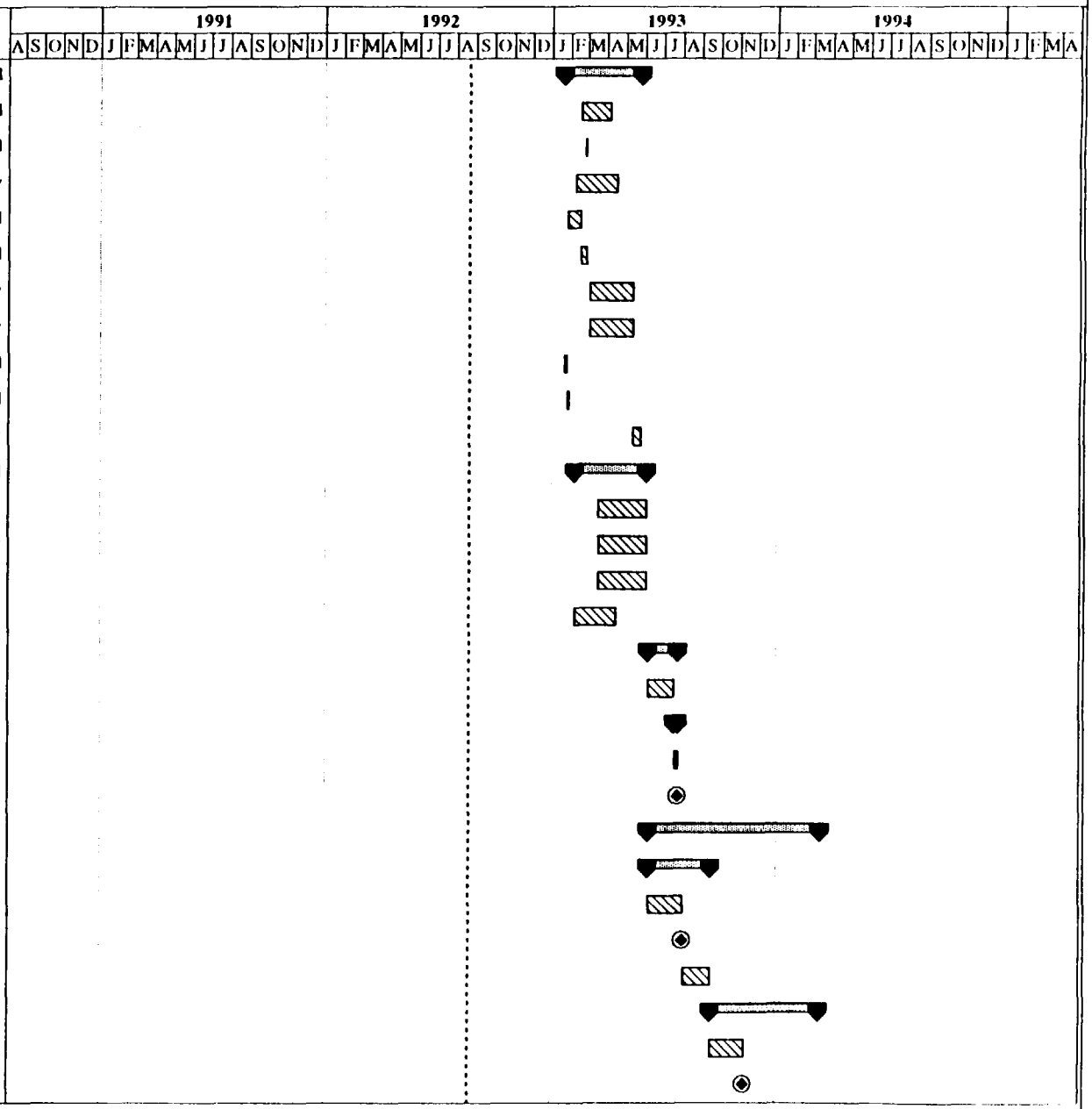
Scheduled Activity Milestone Summary

d = work days ed = elapsed calendar days w = work weeks

FIGURE 1-2
H.O.D. LANDFILL RI/FS
ANTIOCH, ILLINOIS

ID	Name	Duration
30	Source Characterization	125.38ed
31	Cap Evaluation	34d
32	Leach Collect Sys. Eval	2d
33	On-Site Soil/Sed. Sampling/Analysis	10w
34	Leachate Piez./Gas Probes	16d
35	Downhole Gamma Logging	8d
36	Landfill Gas Sampling/Analysis	10w
37	Leachate Sampling/Analysis	10w
38	Landfill Borings	3d
39	Construct Perimeter Borings	3d
40	Off-Site Source Evaluation	2w
41	Migration Path./Contaminant Char.	116.38ed
42	Groundwater Sampling/Analysis	11w
43	Private/Muni. Well Sampling/Analysis	11w
44	Surface Water Sampling/Analysis	11w
45	Soil/Sediment Sampling/Analysis	10w
46	TASK 4: Site Investigation Analysis	49.38ed
47	Perform Site Invest. Analysis	6w
48	Tech Memo #1	6.38ed
49	Prepare Memo	1w
50	Tech Memo #1 to EPA	0d
51	TASK 5: Baseline Risk Assessment	278ed
52	Tech Memo #2 (BRA - TWP)	102ed
53	Prepare BRA -TWP	40d
54	BRA - TWP to EPA	0d
55	EPA Review / Comment	45ed
56	Baseline Risk Assessment	176ed
57	Prepare Draft BRA Report	8w
58	Draft BRA Report to EPA	0d



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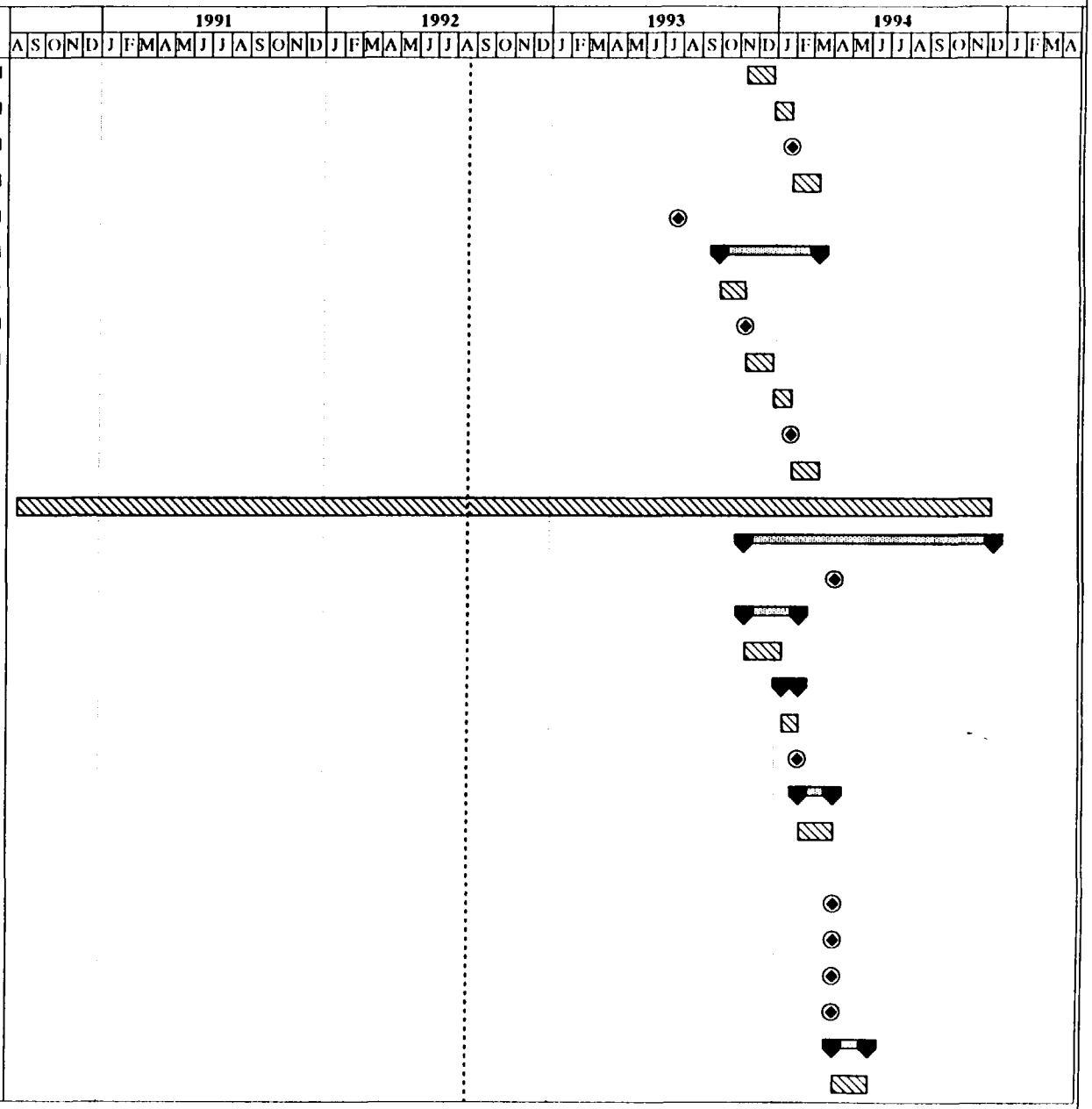
Scheduled Activity Milestone Summary

d = work days ed = elapsed calendar days w = work weeks

FIGURE 1-2
H.O.D. LANDFILL RI/FS
ANTIOCH, ILLINOIS

ID	Name	Duration
59	EPA Review/Comment	45ed
60	Revise BRA Report	30ed
61	Final BRA Report to EPA	0d
62	EPA Review/Approve BRA	45ed
63	TASK 6: Treatability Studies	0d
64	TASK 7: RI Report	162ed
65	Prepare Draft RI Report	6w
66	Draft RI Report to EPA	0d
67	EPA Review / Comment	45ed
68	Revise RI Report	30ed
69	Final RI Report to EPA	0d
70	EPA Review/Approve RI Report	45ed
71	TASK 8: Community Relations	1575ed
72	FEASIBILITY STUDY	400ed
73	TASK 9: Interim Actions	0d
74	TASK 10: Devlp Rem Act Alt	88.38ed
75	Develop Remedial Alternatives	8w
76	Tech Memo #3 (Altern. Array Doc.)	27.38ed
77	Prepare Memo	4w
78	Tech Memo #3 to EPA	0d
79	TASK 11: Screening Alternatives	55.38ed
80	Screen Alternatives	8w
81	TASK 12: Treat. & Supplemental RI	0ed
82	Prepare SI Work Plan	0d
83	EPA Review/Approve SI WP	0d
84	Conduct Supplemental Investgation	0d
85	Conduct Treatibility Study	0d
86	TASK 13: Detailed Analysis of Alt.	57.38ed
87	Conduct Detailed Analysis	8w



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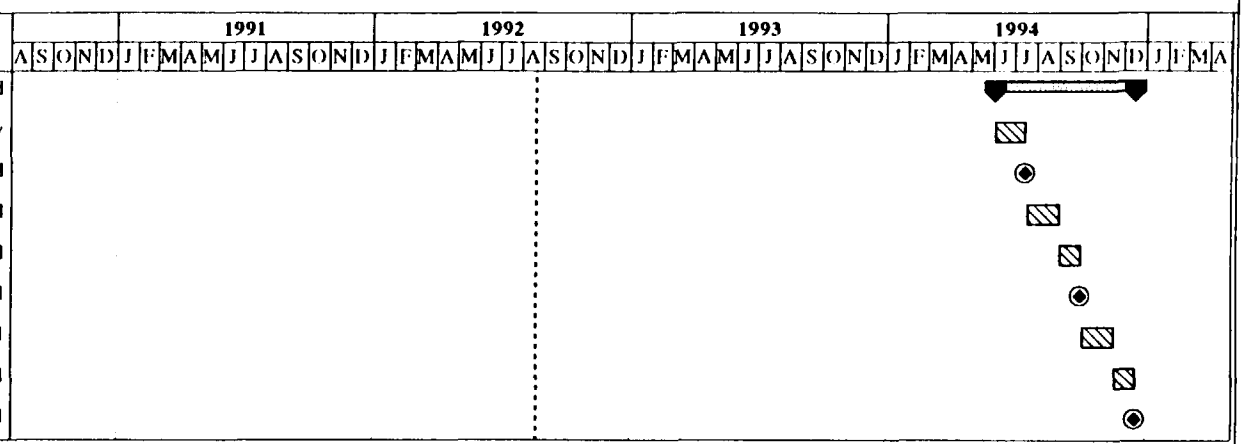
Scheduled Activity Milestone Summary

d = work days ed = elapsed calendar days w = work weeks

FIGURE 1-2
H.O.D. LANDFILL RI/FS
ANTIOCH, ILLINOIS

ID	Name	Duration
88	TASK 14: FS Reports	197ed
89	Prepare Draft FS Report	6w
90	Draft FS Report to EPA	0d
91	EPA Review/Comment	45ed
92	Revise FS Report	30ed
93	Final Draft FS Report to EPA	0d
94	EPA Review FS Report	45ed
95	Revise FS Report	30ed
96	EPA Approval	0d



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Scheduled Activity Milestone Summary

d = work days ed = elapsed calendar days w = work weeks

WARZYN

Project Organization
H.O.D. Landfill RI/FS

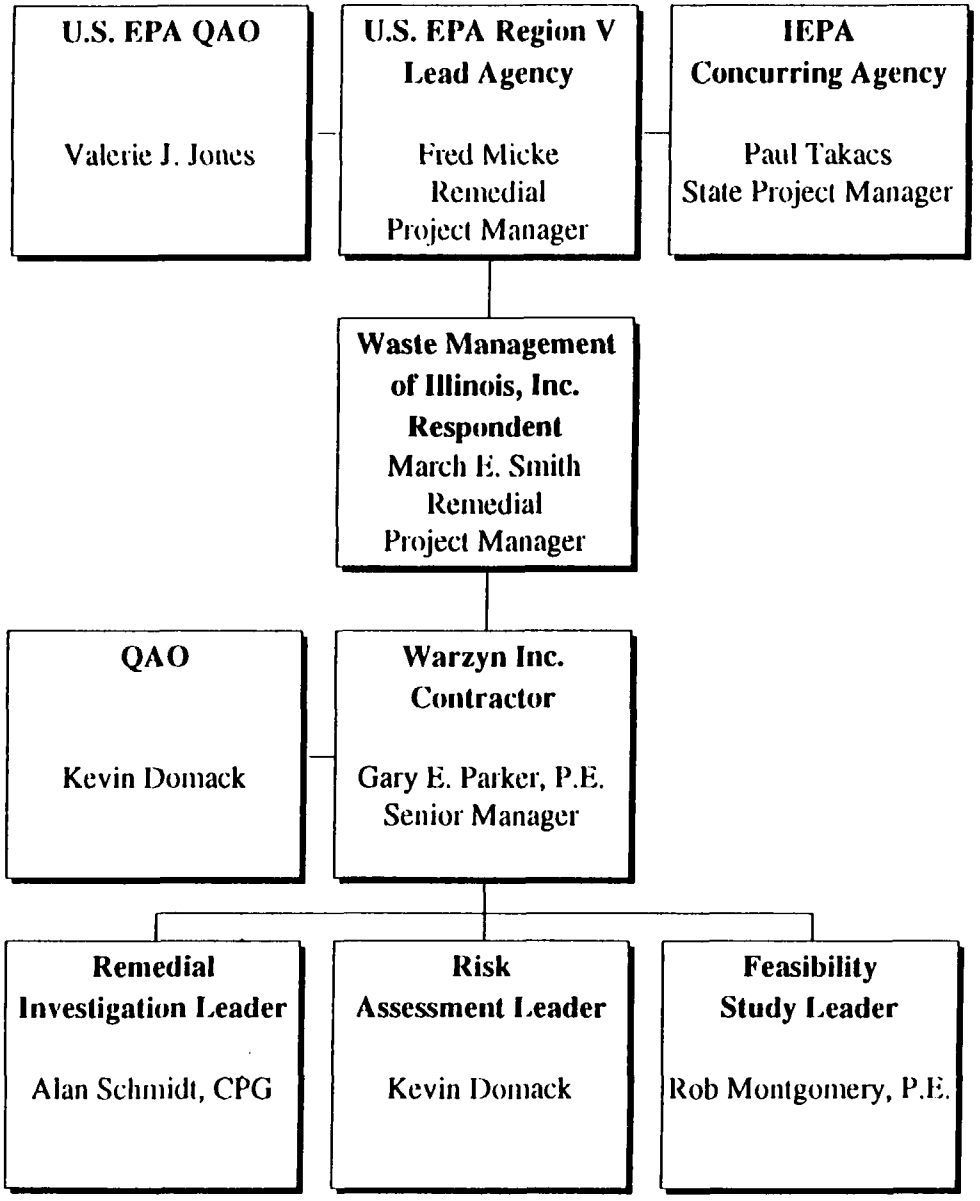

FIGURE 2-1

APPENDIX A

SAMPLING AND ANALYSIS PLAN
HOD LANDFILL
ANTIOCH, ILLINOIS

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1

OBJECTIVES

The primary objective of the activities described in the Sampling and Analysis Plan (SAP) is to obtain representative data to be used for the Remedial Investigation/Feasibility Study (RI/FS) Analysis. Sampling activities described in this plan will be performed to complete the site investigation activities of the RI. Documentation procedures are described in the Data Management Plan.

The RI will consist of physical characterization, source characterization, and contaminant characterization. Data use objectives of the physical characterization include:

- Confirmation of the site stratigraphy, hydrogeology, hydrology, and ecology

Data use objectives of the source characterization site investigation include:

- Location and extent of contaminant source areas
- Investigation of chemical and physical characteristics

Data use objectives for the contaminant characterization include:

- Determination of potential migration pathways
- Evaluation of the magnitude and extent of groundwater contamination
- Evaluation of soil, surface water, sediment and air contamination

2

SCOPE

This Sampling and Analysis Plan (SAP) describes the procedures and practices (Section 5) to be used in obtaining site investigation data for use in the RI/FS. This plan also includes a description of sampling locations and number of samples (Section 3), the sample designation system (Section 4), decontamination and investigative waste disposal procedures (Section 6), and sample handling (Section 7) and sample documentation (Section 8) methods to be employed. The field activities, discussed in this report, include:

- Physical characterization
  - soil borings/soil sampling

- Source characterization
  - landfill cap evaluation
  - leachate collection system effectiveness (no sampling involved)
  - on-site surficial soil and sediment sampling
  - leachate piezometer installation (no sampling involved)
  - landfill gas probe installation
  - downhole gamma logging (no sampling involved)
  - leachate sampling
  - landfill gas sampling
  - landfill borings

- Contaminant characterization
  - groundwater sampling
  - private and municipal well sampling
  - surface water sampling

3

SAMPLING LOCATIONS AND NUMBER OF SAMPLES

3.1 PHYSICAL CHARACTERIZATION

The physical investigation will consist of a hydrogeologic evaluation designed to evaluate glacial stratigraphy around the perimeter of the Site. The evaluation will be accomplished by drilling eight soil borings around the perimeter of the Site through native unconsolidated deposits. The locations of the borings are specified in Table 8 and Drawing 60953-F10, both included in Volume 1 of 3 of the Work Plan.

Up to 16 soil samples, and 2 field duplicates will be analyzed for grain size.

3.2 SOURCE CHARACTERIZATION

Source characterization sampling and analysis activities will include landfill cap evaluation, on-site surface soil and sediment sampling, leachate sampling, landfill gas probe installation and landfill gas sampling, and landfill borings.

3.2.1 Landfill Cap Evaluation

Approximately 10 test pits will be performed to aid in evaluating the effectiveness of the existing cap to minimize infiltration of precipitation. Ten soil samples and one field duplicate will be collected and analyzed for grain size, Atterberg limits, natural moisture content, in-place density, and clay mineralogy using x-ray diffraction.

Test pits will be excavated using a backhoe. Field observations including density and nature of cover vegetation, root penetration, and evidence of inhomogeneities

in the cap will be recorded in a field notebook. Test pit profiles will be recorded on test pit logs.

3.2.2 On-Site Surface Soil and Sediment Sampling

On-site surficial soil and sediments will be collected in likely deposition areas in order to characterize contaminants. One sample from each of five areas of deposition (determined during site inspections) will be collected and analyzed for TCL organics, TAL inorganics, total organic carbon (TOC), grain size, natural moisture content, and Atterberg limits. One duplicate sample will also be collected and analyzed for the same parameters.

Two soil samples will also be collected as matrix spike/matrix spike duplicate (MS/MSD) samples.

3.2.3 Landfill Gas Probe Installation

In order to assess methane gas production within the landfill, multi-stage landfill gas probes will be installed in the same boreholes as each new leachate piezometer. A total of thirteen new multi-stage gas probes will be installed. In addition, five perimeter gas probes will be installed on-site outside of the landfill area. Landfill gas monitoring will consist of monitoring percent methane, oxygen, carbon dioxide, and VOCs.

3.2.4 Leachate Sampling

In order to chemically characterize the leachate composition, samples will be collected from the following five locations: Manhole MHE and leachate piezometers LP1, LP6, LP8 and LP11. One duplicate sample, one field blank, and one MS/MSD will also be collected and analyzed.

Samples collected will be analyzed in the laboratory for TCL organics, TAL inorganics, chloride, sulfate, alkalinity, total hardness, nitrate nitrogen, nitrite nitrogen, ammonia nitrogen, TOC, and total dissolved solids (TDS). Field analysis will be conducted on each sample to determine field pH, field specific conductance, temperature, redox, and dissolved oxygen.

3.2.5 Landfill Gas Sampling

Approximately five landfill gas samples will be collected from landfill gas probes associated with leachate piezometers and/or gas well flares. Landfill gas samples will be collected at the following leachate piezometers: LP1, LP6, LP7, LP8, and LP11. Samples will be analyzed in the field for methane, oxygen, and carbon dioxide. Five investigative samples, one field blank and one duplicate collected will be submitted for VOC analysis.

3.2.6 Landfill Borings

In order to determine subsurface conditions and evaluate the feasibility of constructing a leachate barrier along the perimeter of the "old" landfill, approximately five soil borings will be drilled along the southern perimeter of the "old" landfill. Approximately ten soil samples (two from each boring) and one field duplicate will be submitted for grain size analysis.

3.3 CONTAMINANT CHARACTERIZATION

Contaminant characterization activities will include groundwater monitoring (water level measurements, in-situ hydraulic conductivity tests, new well installation, and sampling of new and existing wells), municipal and private well monitoring, and a hydrogeologic evaluation/surface water sampling.

3.3.1 Groundwater Monitoring

Eight monitoring wells will be installed at eight different locations around the site.

Initially, water level measurements will be made at all monitoring wells. Single well in-situ hydraulic conductivity tests will be conducted at selected existing and new monitoring wells (see Work Plan Section 4.4.2). Prior to new monitoring well installation, existing monitoring wells will be inspected to confirm that each is functional and capable of being sampled.

Phase 1 groundwater sampling will consist of sampling the following wells and analyzing for TCL/TAL parameters, alkalinity, chloride, hardness, sulfate, TOC, total dissolved solids (TDS), nitrate nitrogen, nitrite nitrogen, and ammonia nitrogen:

- US1S and 1D
- US4S and 4D
- US6S, 6I and 6D
- G11S and 11D
- W5S
- US3S, 3I, and 3D
- W4S
- W6S
- W7D

3.3.2 Municipal/Private Well Monitoring

Municipal wells 3 and 5 and up to six private groundwater supply wells will be sampled and analyzed for for TCL organics and TAL inorganics. One field duplicate and field blank will be collected for each analysis. An MS/MSD sample will also be collected and analyzed for TCL organics. Private well sampling will be coordinated with the Lake County Health Department. Field parameters will consist of pH, conductivity and temperature.

3.3.3 Surface Water Sampling

Three surface water samples will be collected for analysis of TCL organics and TAL inorganics. One field duplicate and field blank will be collected for each analysis. An MS/MSD sample will also be collected for TCL organic analysis. Field parameters will consist of pH, conductivity, temperature, redox, and dissolved oxygen.

During sampling, surface water levels and flow rates will be measured. A physical inspection of the Sequoit Creek banks will also be conducted.

3.4 QUALITY ASSURANCE SAMPLING

Quality control samples will collected during sampling activities associated with the physical characterization of soils, and the chemical characterization of sediments/surface soils, leachate, landfill gas, groundwater and surface water. Quality control samples will consist of sample duplicates, field blanks and matrix spike/matrix spike duplicate (MS/MSD) samples as described below.

3.4.1 Field Blanks

For sediment and soil samples, no field blanks will be collected due to the unavailability of suitable blank material. For water and leachate samples, one field blank will be prepared for each type and container size. Field blanks will be prepared according to the following schedule for each sampling activity:

- One field blank for every 10 or fewer samples collected; and
- For each sampling period, a minimum of one blank for each group of parameters per sample matrix.

The field blank samples will be prepared using deionized water stored in polyethylene containers. The water will be routed through the appropriate sampling equipment following decontamination. Landfill gas field blanks will be prepared by collecting a sample of ambient air after decontamination of the sampling system.

3.4.2 Matrix Spike and Matrix Spike Duplicates (MS/MSD)

For water and leachate samples, one sample per group of 20 or fewer samples collected for VOA and extracted organics analysis during each sampling activity will be selected for MS/MSD analysis. MS/MSD samples will not be collected for inorganic analysis.

For soil samples, two MS/MSD samples will be collected for organic analysis.

3.4.3 Sample Duplicate

One duplicate sample will be collected for each increment of 10 or fewer samples collected for each matrix during each sampling period. A duplicate sample will consist of a sample obtained from the same sampling device as the original sample.

3.4.4 Trip Blanks (TB)

A trip blank for VOC analysis will be included in each sample shipment containing water matrix samples for VOCs. The trip blank will consist of two 40-ml VOC vials filled with deionized water with a Milli-Q cleanup. It will be prepared in the laboratory or office, transported to the field and shipped with the other samples to the designated laboratory without being opened. It will be packaged using standard procedures as for the other sample bottles.

4

SAMPLE DESIGNATION

A sample numbering system will be used to identify each investigative and quality control sample. Each sample identifier will include the project identifier code, sample type and location code, and a sampling event code. The sampler will maintain a log book containing the sample identification listings.

4.1. PROJECT IDENTIFIER CODE

A two-letter designation will be implemented to identify the sampling site. The project identifier will be "HD" to signify the HOD Landfill Site in Antioch, Illinois.

4.2. SAMPLE TYPE AND LOCATION CODE

Each sample collected will be identified by a two-letter code corresponding to the sample type. Sample type codes to be used for the subtasks covered in this sampling plan include:

- GW- groundwater sample from monitoring well
- SS - split spoon or soil boring sample
- SD - sediment sample
- SW - surface water sample
- SU - surface soil sample
- PW - groundwater from a private residential well or municipal well
- SC - soil sample collected from test pits of the clay cap
- LC - leachate sample
- LG - landfill gas

Other letter designators may be added for sample activities of later subtasks.

The location code will follow the sample type code. The location code consists of a two-to-five digit numeric or alpha-numeric code that indicates the sample location. Surface water, sediment, field blanks, and air samples will use a consecutive numbering system starting at 01, assigned in the field.

4.3. SAMPLING ROUND CODE/DUPLICATE CODE

A two-digit numerical code will be used to designate additional location information. For soil samples, the Round code will represent the depth of the sample in feet below the ground surface. Duplicate samples will be designated by the Round code preceded by a 9. Matrix spike and matrix spike duplicate samples are collected as additional sample volume at selected locations.

Although identified as such on chain-of-custody records, specific sampling codes will not be provided for matrix spike or matrix spike duplicate samples.

4.4 EXAMPLES OF SAMPLE NUMBERS

Examples of sample number codes are as follows:

- HD-SSW5S-15 = HOD Landfill, split spoon sample from monitoring well W5S at a depth of 15 feet.
- HD-SCTP7-3 = HOD Landfill, soil sample of clay cap collected from at Test Pit 7 a depth of 3 feet.

5

GENERAL SAMPLING EQUIPMENT AND PROCEDURES

5.1 SOIL BORINGS AND SOIL SAMPLING

5.1.1 Objective

The objective of this activity is to physically characterize the subsurface soils and to confirm the local glacial stratigraphy.

5.1.2 Personnel and Responsibilities

A drill crew of two individuals will perform the borings for soil samples. A geologist will supervise their efforts, collect soil samples and function as site safety officer.

5.1.3 Methods

Borings will be drilled using 4.25-inch inner diameter (ID) x 8.5-inch outer diameter (OD) or 6.25-inch ID x 10.25-inch OD hollow stem augers. Soil samples will be collected with split spoon samplers, visually classified in the field by a geologist and placed into the appropriate sampling jars. Soil samples will be collected at 2.5 foot intervals. Selected soil samples will be analyzed in the laboratory for grain size. Two samples will be collected from each boring for analysis.

Soil samples will be selected for analysis based on visual observations, sample depth and stratigraphy.

5.2 LANDFILL CAP EVALUATION

5.2.1 Objective

The objective of this activity is to evaluate the effectiveness of the existing cap.

5.2.2 Personnel and Responsibilities

Test pits will be excavated by a crew of two individuals, one individual will operate the backhoe under the supervision of a geologist. The geologist will observe the excavation and record observations and function as the site safety officer.

5.2.3 Methods

At each test pit location, topsoil will be carefully scraped away and placed to one side. Cap materials will then be excavated and placed separately from the covered materials. The on-site geologist will observe and describe and photodocument the excavation and monitor air quality with a photoionization detector (PID). Excavations will be terminated at approximately 5 to 6 feet or when the on-site geologist and U.S. EPA oversight professionals agree that the cap profile has been adequately evaluated.

Decontamination will include steam cleaning the backhoe bucket before the initial excavation during each visit and between subsequent excavations to minimize carry-over contamination.

5.3 SURFICIAL SOIL AND SEDIMENT SAMPLING

5.3.1 Objective

The objective of this activity is to chemically characterize site surficial soils/sediments.

5.3.2 Personnel and Responsibilities

A crew of two individuals will collect samples.

5.3.3 Methods

Samples will be collected using a stainless steel trowel and will be transferred directly to the appropriate sample jars. Samples will be analyzed in the laboratory for U.S. EPA CLP TCL and TAL parameters, and physical parameters (pH, TOC, grain size, natural moisture content, and Atterberg limits).

5.4 LANDFILL GAS MONITORING

5.4.1 Objective

The primary objective of this activity is to monitor the presence of landfill gas (LG) within and outside the landfill and, if found, conduct analysis to determine percent methane, oxygen, and carbon dioxide, and the presence and concentrations of volatile organic compounds (VOCs).

5.4.2 Personnel and Responsibilities

A geologist, hydrogeologist, or field engineer, and a technician, will be responsible for obtaining landfill gas samples.

5.4.3 Methods

Samples of landfill gas will be collected for VOC analysis in the following manner:

- Samples will be collected in canisters provided by the analytical laboratory. Each canister is under vacuum, and has a valve opening.
- One end of tygon tubing will be attached to the canister; the other end will be attached to the gas probe that has been purged suing a personal sampling pump.
- The valve connecting the canister to the tubing will be opened for two minutes, allowing the sample to collect in the canister.
- The valve will then be closed, and the tygon tubing disconnected.
- The canister will be stored in a cooler for shipment to the laboratory.

Percent methane, oxygen, and carbon dioxide will be measured in the field using a GasTech or similar monitoring instrument.

5.5 LEACHATE SAMPLING

5.5.1 Objective

The primary objective of this activity is to chemically characterize leachate.

5.5.2 Personnel and Responsibilities

A team of two individuals will be responsible for the sampling.

5.5.3 Methods

Each well will be sampled using a stainless steel bailer attached to stainless steel cable. The sampling equipment will be cleaned between wells with a Liquinox or other non-phosphate detergent solution and will be rinsed with deionized water.

Decontamination will include steam cleaning or high pressure hot water washing the drilling equipment and tools between field boreholes and detergent washing and water rinsing the split-spoon samplers after each collected sample. Decontamination fluids and borehole cuttings will be containerized and stored in a secure area pending results of groundwater and soil analyses. If borehole cuttings, purge water, decontamination water are found to be contaminated, these materials will be disposed of appropriately after consultation with EPA.

5.6 MONITORING WELL INSTALLATION AND ASSOCIATED TESTING

5.6.1 Objective

The objective of this activity is to determine groundwater flow direction and physical characteristics of the geologic media in the site area.

5.6.2 Personnel and Responsibilities

Groundwater monitoring wells will be installed and soil borings performed by a drill crew of two individuals each. Monitoring well installation and soil borings will be supervised by a geologist, who will collect soil samples and function as the site safety officer. Water levels will be measured by a technician. Hydraulic conductivity tests will be performed by a team of two individuals.

5.6.3 Methods

Monitoring Well Installation - Each boring will be drilled using 4.25-inch ID or 6.25-inch ID hollow stem augers from ground surface to the base of the boring. Soil samples will be collected at ground surface and at 2.5 ft intervals to the base of the boring. Split-spoon samples will be analyzed for grain size and Atterberg limits (if appropriate). The purpose of these analyses is to confirm field identification of soils and to assist in assessing subsurface soils as a contaminant migration pathway. Soil samples will be field screened with a PID to assess potential hazards to the field personnel.

Final soil boring logs will be prepared based on field observations, soil testing, and laboratory sample classification. Soil samples and rock cores will be retained by Warzyn for the respondents until termination of the RI/FS. Procedures for sample handling are discussed in the QAPP.

Monitoring wells will be constructed of 2-inch ID Schedule 40 PVC pipe. Water table wells will be constructed with 10-ft long, PVC screens placed so the screen is likely to intercept the water table. Piezometers will be constructed with 5 ft long PVC screens.

The annular space between the well and the edge of the borehole will be backfilled with clean silica sand to approximately 2 ft above the top of the well screen. At water table wells, the remainder of the annulus will be backfilled with granular bentonite. At piezometers, a fine silica sand or bentonite pellets will be placed above the filter pack and bentonite slurry placed above the fine silica sand or bentonite pellets. The remainder of the annular space will be backfilled with a sodium based bentonite slurry. Where more than 5 ft of slurry needs to be placed, a tremie pipe will be used. A locking steel protective casing will be installed at the surface of each well.

A PID will be used to monitor air quality for site safety purposes and to screen soils for VOCs. Drill cuttings from the boreholes will be containerized if PID readings exceed 5 ppm. Water used during the drilling operations and well development will be containerized pending results of groundwater sample analysis.

To minimize potential inadvertent contamination by drilling equipment and/or inter-borehole contamination, steam cleaning will be used. The drill rig and drilling tools will be steamed cleaned before mobilization onto the site. The drilling tools coming into contact with site soils will be steam-cleaned between each boring. Water from the steam cleaning will not be collected.

The split-spoon sampler will be cleaned in a liquinox or non-phosphate detergent solution between samples. Water from split-spoon cleaning will not be collected. Well pipe, screen and protective casings will be steam cleaned or washed with a high pressure hot water washer before installation. Water from steam cleaning will not be collected.

New monitoring wells will be developed by alternatively surging and purging each wells using a bailer for a minimum of 30 min. After the surge and purge cycles are completed, the well will be pumped until 10 well volumes are removed or until the well produces sediment free water. Purged water will not be collected.

Water Level Measurements - Water levels in existing and new monitoring wells will be measured. An electronic water level indicator will be lowered into the observation wells until the water level is reached. Depths will be recorded to the nearest 0.1 ft.

In-Situ Hydraulic Conductivity Tests - Hydraulic conductivity will be measured by drawdown testing in selected shallow water table wells and by air pressure in deeper piezometers. Hydraulic conductivity testing will be conducted after groundwater quality sampling. The methods to be used are as follows:

- Measure water level with a tape and sounding device or electronic water level indicator.
- Place the pressure transducer into the well and allow approximately three minutes for the probe to equilibrate to the water temperature and pressure.
- Install the well head device to seal the well head (for piezometers only).
- Enter the reference water level into the data logger and check the water level using the pressure transducer until water level reading is stable.
- After a stabilized water level reading is obtained from the pressure transducer, the well is pressurized with sufficient air pressure to displace 10 ft of water (0.4 PSI/ft of water) (for piezometers only).
- Air pressure is maintained until the water level reading from the transducer is constant (for piezometers only).
- The air pressure is then instantaneously released while running the pressure transducer recorder in the log sampling mode (for piezometers only).
- At water table wells a single bailer full is removed to reduce the water level, while running the pressure transducer recorder in the log sampling mode.
- The test results are immediately printed out to obtain a hard copy.
- Data are transferred to a micro-computer.

5.7 GROUNDWATER QUALITY SAMPLING

5.7.1 Objectives

The objective of groundwater sampling is to determine the nature, magnitude and extent of groundwater contamination.

5.7.2 Personnel and Responsibilities

A crew of two technicians will collect samples.

5.7.3 Methods

New and selected existing monitoring wells will be sampled during the investigation. Sampling will not be conducted for a minimum of two weeks after well development. Several private wells and municipal wells may also be sampled during the investigation. Groundwater sampling will proceed from wells expected to have the lowest contaminant concentrations (based upon observations during drilling and existing groundwater quality data), to the wells suspected of having the highest contaminant concentrations. Each sampled well will be purged immediately prior to sampling using a submersible sampling pump, stainless steel bailer attached to stainless steel cable, or bladder pump. The volume of water removed from the well will be measured so that a minimum of three well volumes are removed. Groundwater samples will be collected immediately after well purging has been completed. Samples to be analyzed for VOCs will be collected first to minimize volatilization.

The sampling equipment and water level measurement tape will be cleaned between wells with a Liquinox or another non-phosphate detergent solution and rinsed with deionized water.

Groundwater sample blanks will be collected by pouring deionized water from the sampling device into the sample bottles. Matrix spike/matrix spike duplicate samples will be collected using the same device used for groundwater sample collection. Duplicate samples for volatile organics analysis will be obtained by alternately filling VOC vials for the primary and duplicate sample. The remainder of the sample for inorganic analysis, will be split between the principle sample and the duplicate sample.

5.8 SURFACE WATER CHARACTERIZATION

5.8.1 Objective

The objective of the surface water investigation is to chemically characterize surface water.

5.8.2 Personnel and Responsibilities

A team of two technicians will collect surface water samples and provide their own site safety monitoring.

5.8.3 Methods

VOC samples will be collected first to minimize volatilization. Samples for metal analysis will be unfiltered. Surface water samples will be collected using stainless steel dippers as sampling equipment. Sampling equipment will be decontaminated using Liquinox or another non-phosphate detergent and rinsed with deionized water.

6

DECONTAMINATION AND INVESTIGATIVE WASTE DISPOSAL PROCEDURES

6.1 DECONTAMINATION PROCEDURES

Procedures to be followed to decontaminate equipment and personnel are described in the Site Health and Safety Plan. The procedures are summarized below.

Site Personnel Decontaminate Procedure:

- Dispose of outer latex booties
- Wash boots in Liquinox bootwash
- Clean outer gloves in Liquinox wash solution (discard if too soiled to clean thoroughly)
- Dispose of polycoated tyvek suits
- Dispose of surgical gloves
- Wash hands in hand wash
- Wash face and neck in face wash
- Clean and sanitize face mask

Site personnel will perform the above mentioned decontamination procedure at a specified decontamination area prior to leaving the site.

Discarded clothing and other articles will be collected in double-lined, heavy duty garbage bags.

Equipment and vehicle decontamination procedure:

- Decontamination will be performed prior to site entry
- Decontamination will be performed on-site
- Gross contamination will be removed with a brush and Liquinox solution
- Steam cleaning or hot water high pressure washing will follow

The drilling equipment and the backhoe will be steam cleaned or high pressure hot water washed, at a designated decontamination area, between boring/excavation locations and prior to exiting the site. The equipment and vehicle decontamination area will be constructed above grade adjacent to an existing on-site manhole. The catchment will be lined and will drain to the existing manhole. Collected waste water will be immediately pumped from the manhole into the temporary leachate tanker and disposed of with the extracted leachate. The equipment decontamination area will be located away from the designated personnel decontamination area.

Decontamination will include steam-cleaning or hot water high pressure washing the drilling equipment, backhoe and tools between boreholes and test pits, and detergent washing and water rinsing the split spoon samplers after each collected sample. The drill rig and equipment may also require scrubbing of accessible parts with a detergent/water solution. Well materials will also be steam-cleaned or hot water high pressure washed and wrapped in plastic until installed. The bailer cable, trowels, spatulas, stainless steel bucket and water level measurement tape will be cleaned with Liquinox or another non-phosphate detergent solution, and rinsed with deionized water.

Equipment remaining on-site overnight will be decontaminated at the end of the day, if not done earlier in the day.

6.2 INVESTIGATIVE WASTE DISPOSAL PROCEDURES

Performance of the remedial investigation will generate liquid and solid investigative wastes. The disposition of these wastes is described below.

6.2.1. Monitoring Well Fluids

Waste liquids derived from the development or purging for sampling of monitoring wells will be collected and disposed with leachate.

6.2.2. Soils

Impacted soil will be generated through drill cuttings in the performance of soil borings. These soils will be segregated on the basis of whether impacts are indicated by PID field screening. Soils are considered impacted if PID readings are greater than 10 ppm. Non-impacted soil (<10 ppm) will be left in the area near the soil boring. Impacted soils (>10 ppm) will be stored on-site in a covered roll-off container until a final site remedy is selected. When the final remedy is implemented, the impacted soils may be incorporated with the waste under the final remedy, or handled as a solid waste if they pass the TCLP test.

7

SAMPLE HANDLING AND ANALYSIS

7.1 PARAMETERS

Samples collected for chemical analysis will be analyzed by a laboratory approved by the U.S. EPA Region V Contract Program Management Section (CPMS). Chemical parameters for which groundwater and surface water may be analyzed are summarized below:

- U.S. EPA CLP TCL organics
- U.S. EPA CLP TAL inorganics
- Field pH
- Field Specific Conductance
- Chloride
- Sulfate
- Alkalinity
- Nitrate - Nitrogen
- Nitrite Nitrogen
- Ammonia - Nitrogen
- Total Organic Carbon
- Total Dissolved Solids

Measurement of pH, specific conductance and temperature, dissolved oxygen and redox will be performed in the field. The groundwater samples to be analyzed for inorganics will be field filtered with 0.45 micron filters. Surface water samples will not be field filtered. Surficial soil and sediment samples will be analyzed for U.S. EPA TCL and TAL parameters and grain size, Atterberg limits, TOC, and natural moisture content. Soil samples from monitoring well installation borings will be analyzed for grain size and Atterberg limits (if appropriate).

Soil samples from the landfill cap evaluation will be analyzed for grain size, Atterberg limits, natural moisture content, density, and clay mineralogy. Leachate

samples will be analyzed for the same parameters as groundwater. Landfill gas samples will be analyzed for VOCs, methane, oxygen, and carbon dioxide.

7.2 SAMPLE PRESERVATION

Samples will be collected and preserved in a manner appropriate for the analyses they receive (see Table 1-2 of the QAPP). The portion of groundwater samples requiring field filtering prior to analysis (see Table 1-2 of the QAPP) will be filtered using a pressure filtration device, through a 0.45 micron filter, as soon as possible after collection. Filtered portions of the samples will be preserved, as appropriate, immediately after filtration. Sample fractions will be preserved before shipment according to the procedures shown in Table 1-2 of the QAPP. Preservatives added to the samples will be prepared using reagent grade chemicals. Table 1-2 of the QAPP should be consulted for details regarding sample packaging and shipping.

8

SAMPLE DOCUMENTATION

Field sampling activities will be documented using a bound notebook/logbook. Information recorded in the field notebook will include date of sampling, sampler, weather conditions, observations and methods of preservation. Additional data pertaining to sampling may also be included in the logbook.

Samples will be collected under chain-of-custody procedures. Standard forms including sample labels, sample tags, chain-of-custody forms, and custody seals used for sample tracking will be maintained (see attachments). A brief description of sample documents follow:

A. Chain of Custody Form

1. One Form per shipping container (cooler).

2. Carrier service does not need to sign form, if custody seals remain intact.

3. Use for all samples

B. Chain of Custody Seals

1. Two seals per shipping container to secure the lid and provide evidence that samples have not been tampered with.

2. Cover seals with clear tape.

3. Record seal numbers on Chain of Custody Form.

4. Use for all samples.

C. Sample Tags

1. Each sample container must have a sample tag affixed to it.

2. Sample tag numbers are recorded on the Chain of Custody Forms.

3. Use for all samples.

D. Sample Identification Record Form will:

1. Provide means of recording crucial sample shipping and tracking information.

2. Contain information such as:

- Sample number
- Sample matrix
- Sample location code
- Sample round
- Chain of custody number
- Lab code
- Date sampled
- Date shipped
- Airbill number
- Sampling tag number

Paperwork accompanying the samples being shipped to the laboratory will be sealed in a plastic bag that is taped to the inside of the cooler lid. Copies of the chain-of-custody forms, and other paperwork (if possible), will be retained with the field files.

Two sample seals will be placed on opposite sides of the lid and extending down the sides of the cooler. The lid will be securely taped shut prior to shipment.

APPENDIX B

ANALYTICAL SERVICES STANDARD OPERATING PROCEDURES (SOPs)

APPENDIX B-1

ALKALINITY (ENSECO)

pH, Specific Conductance, and Alkalinity
(Automatic Titrator)

SOP No.:	Revision No.:	Effective Date:
LM-RMA-1071	1.0	August 8, 1990

Supersedes: Original

1. Scope and Application

1.1 Analytes

This method is to be used for the determination of pH, Specific Conductance, and Alkalinity. The different forms of alkalinity (total, bicarbonate, carbonate, and hydroxide) can also be calculated.

1.2 Applicable Matrices

This method is applicable to drinking, surface and saline waters, domestic and industrial wastes.

1.3 Reporting Limits

The Enseco reporting limits are:
pH -- Detection limit not applicable
Specific conductance -- 1 umho/cm
Alkalinity -- 5 mg/L as Calcium Carbonate

1.4 Dynamic Range

This method is applicable to all ranges likely to be encountered. As a practical matter, samples with alkalinities greater than about 1200 mg/L as Calcium Carbonate require a smaller than normal volume or stronger titrant in order to keep the titrant volume to a reasonable amount.

Prepared by:	Date:
Lindsay Breyer/Roxanne Sullivan	5/24/88
Management Approval:	Date:
[signature]	8/6/90
QA Officer Approval:	Date:
[signature]	8/6/90

1.5 Analysis Time

The approximate analytical time is five minutes per sample. Samples with high alkalinity or undissolved solids may require longer titration times.

2. Method Summary

2.1 Samples are analyzed on an automatic titrator.

2.2 The Specific Conductance is measured first by application of a voltage across the conductivity cell in series with a known reference resistance. The ratio of voltages across the cell and the known resistance is measured which allows the resistance of the cell to be calculated. This is then converted to the conductance of the cell. By calibrating against a solution of known conductance, the cell constant is determined and also the specific conductance of the sample in the cell. A thermocouple measures the temperature of the sample and electronically corrects the measurement to 25°C.

2.3 The pH is then determined electrometrically with a glass electrode in combination with a reference electrode. The special glass used in the electrode develops a voltage across it that depends on the pH of the solution being analyzed. The voltage is measured and converted to pH by calibration against buffers of known pH.

2.4 Alkalinity is determined by titrating the sample with a standardized acid to specified endpoints (pH 8.3 and 4.5). Alkalinity is calculated from the volume of acid required to reach the endpoints and is traditionally reported as Calcium Carbonate.

3. Comments

3.1 Interferences

3.1.1 Sodium can interfere with pH determinations at pH's higher than 10. This is controlled by using a "low sodium error" electrode.

3.1.2 Oil and particulate matter in the samples may coat the pH electrode and conductivity cell causing erroneous readings. This will also affect the alkalinity results.

3.1.3 Temperature variations will affect both pH and conductance measurements. The conductance measurements are corrected for temperature automatically. The effect on pH is controlled by performing the measurements at the same (i.e. room) temperature. All samples must be within 2°C.

3.1.4 Salts of weak organic and inorganic acids will contribute to alkalinity. If the alkalinity is intended to be a measure of carbonate and biocarbonate only, the presence of these substances will cause high results.

3.1.5 The alkalinity is titrated to pH 4.5. This endpoint may not be appropriate for all samples.

3.1.6 Specific conductance is extremely sensitive to contamination. The titration head, electrodes, and all glassware must be thoroughly rinsed with deionized water to remove all traces of salts.

3.1.7 Samples not in equilibrium with the atmosphere may exhibit changes in pH and in the distribution of the various forms of alkalinity when exposed to the atmosphere. The sample containers should be filled completely and kept closed until just prior to the analysis. The analysis should be performed as soon as possible.

3.1.8 The pH electrode may exhibit slow or noisy response with high purity waters due to the lack of ionic strength.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco-RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your supervisor or safety officer.

4.2 Normal laboratory safety precautions should be followed. There are no special concerns with this method.

5. Sample Collection, Preservation, and Holding Times

5.1 Samples are to be collected in plastic bottles and refrigerated to 4°C.

5.2 Samples for alkalinity must not be filtered, diluted, concentrated, or altered in any way.

5.3 If the conductance measurement cannot be performed within 24 hours of collection, the sample should be filtered.

5.4 The holding time for these parameters will be considered to be 24 hours since all are analyzed at once. The official holding times are:

pH -- 24 hours
Conductance -- 28 days
Alkalinity -- 14 days

6. Apparatus

6.1 Radiometer Automatic Titration System, consisting of autosampler, burette, pH meter and electrodes, conductance meter and cell, temperature probe, stirrer, printer, etc.

6.2 Plastic beakers to fit autosampler.
These must be thoroughly rinsed to remove all traces of salt.

6.3 Oxford pipette calibrated to 10.0 mL, and disposable tips.

6.4 Miscellaneous laboratory apparatus and glassware.

7. Reagents and Standards

7.1 pH Buffers, 4.0, 7.0, and 10.0
Obtained commercially

7.2 0.01 M Potassium Chloride (Conductance Standard)

Keep a beaker full of KCl in a 105°C oven for routine use. Dissolve 0.7456 g in deionized water and dilute to 1 liter. This solution has a conductance of 1417 umho/cm at 25°C. Prepare monthly.

7.3 0.0500 N Sodium Carbonate

Dry anhydrous sodium carbonate at 250°C for four hours and cool in a desiccator. Dissolve 2.6497 g in deionized water and dilute to 1 liter.

7.4 0.1 N Sulfuric Acid (Strong Alkalinity Titrant)

Very carefully add 3.0 mL concentrated sulfuric acid to approximately 500 mL deionized water and dilute to 1 liter. This solution must be standardized using the procedure given in section 8. Record standardization data and obtain a verification number from the Standards Prep Department.

7.5 0.02 N Sulfuric Acid (Alkalinity Titrant)

Very carefully add 3.0 mL concentrated sulfuric acid to 5 liters of deionized water. Standardize using the procedure given in section 8. Record standardization data and obtain a verification number from the Standards Prep Department.

7.6 Saturated NaCl

Dissolve 35.7 g of NaCl in 100 mL of cold DI.

8. Procedure

8.1 Preparation

8.1.1 Thoroughly homogenize samples before taking an aliquot for analysis. Allow aliquots to equilibrate to room temperature before analyzing.

8.2 Instrument Set-up and Calibration

8.1.2 Standardization of Alkalinity Titrant

Accurately measure 0.05 N Sodium Carbonate into a 250 mL beaker. Use 40 mL if 0.1 N Sulfuric Acid is being standardized or 15 mL for 0.02 N Sulfuric Acid. Add deionized water to bring the total volume to 100 mL.

Titrate with the appropriate strength acid to a pH of about 5. Lift electrode and rinse into beaker. Cover with a watch glass and boil solution gently for 3 - 5 minutes. Cool to room temperature, rinse watch glass into beaker, and continue the titration to the pH inflection point. Calculate the acid normality using the formula in section 10.

Perform all titrations for standardization in duplicate.

8.2.2 Calibration for pH

Be sure that the reference electrode has been filled with saturated potassium chloride and that all electrodes and the titration head are clean and dry (only use Kim wipes to dry the electrodes).

Pour approximately 20 mL pH 7 buffer into a beaker and lower the electrodes into it. Place the titrator in pH mode and press the CAL button when the reading has stabilized. Step through the calibration routine by pressing the CAL button. When Buffer 2 is displayed, rinse the electrodes and immerse in pH 4 buffer. Allow the reading to stabilize and continue with the calibration routine. At each step, make sure that the displayed parameter is correct (e.g. buffer value, temperature, etc.). If you need to change an entry, press the SHIFT button and enter the correct value. When Buffer 3 is displayed, press the pH button to exit the calibration routine (this part of the calibration routine is for a different application not used here). Rinse the electrodes.

8.2.3 Calibration for Conductance

Be sure that the electrodes and titration head are clean and dry (only use Kim wipes to dry the electrodes).

Pipette 20 mL Conductance Standard (0.01 M potassium chloride) into a clean beaker and immerse the electrodes. Verify that the reference temperature is set to 25°C and

the temperature compensation is 2% per degree C. Then adjust the cell constant to give a reading of 1.42 mS/cm. Rinse the electrodes when done. Since the accuracy of this measurement is very dependent upon electrode position it might be advised to calibrate on the first two cups of an autotitrator run. The position of the arms and electrodes are consistent during a run. Calibration should be done daily.

8.3 Analysis

8.3.1 Obtain samples from sample receiving. Check each sample for special instructions. Make copies to put into your data package. Fill out the benchsheet.

8.3.2 Turn on the power to the burette and autosampler. Be sure the titrant reservoir is full. The 0.02 N Sulfuric Acid should be used. If new titrant has been prepared, rinse out the reservoir with it, refill, and flush out the burette and titrant delivery line. Be sure that there are no bubbles in the line.

8.3.3 Set the autosampler to 2 rinses and a delay of 0.4 minutes. Fill two beakers with deionized water (20 - 25 mL) and place them in positions 19 and 20. These are the rinse beakers.

8.3.4 Set the titrator to EP mode and be sure that Method 1 is displayed.

8.3.5 Pipette 20 mL of each sample into a clean beaker and place on the turntable. A 10 mL Oxford pipette may be used to dispense two 10 mL aliquots of sample, if it has been calibrated previously to 10 mL. Record the sample ID and position on the titrator. Allow sample aliquots to equilibrate to room temperature.

8.3.6 Press START NEW CYCLE on the autosampler and the analysis will commence. Results will be automatically printed out and the sampler will stop when all samples have been titrated. At this point, additional samples can be placed on the titrator and analyzed starting at step 8.3.4. Be sure to keep the titrant reservoir filled and replace the water in the rinse beakers periodically.

8.3.7 The titrator has been programmed to deliver a maximum volume of 25 mL titrant. Samples requiring more than this should be reanalyzed using a smaller aliquot or strong titrant (0.1 N Sulfuric Acid). See your supervisor for the corrections to be made in the program for titrator.

8.3.8 Samples with a conductance greater than 50,000 umho/cm need to be run on the high curve. See Appendix 1.0.

8.4 Conclusion

8.4.1 Clean up the work area.

8.4.2 Return samples to Sample Receiving.

8.4.3 Calculate the results according to the calculation section below.

8.4.4 Complete the data package making sure it is filled out properly and all the results are highlighted. Be sure to include the tape and copies of the special instructions. Submit the data package to your supervisor for approval.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 Deionized water and tap water are to be analyzed at least once daily. The conductance of these is to be recorded in the notebook provided for that purpose.

9.1.2 Check a 10.0 pH standard with each new calibration.

9.1.3 One blank and DCS samples must be analyzed in every batch of 20 or less samples. The blank is deionized water.

9.1.4 A blank and standard check are to be performed after every 10 or less readings and at the end of the run. Use the DCS for standard checks. The blank is deionized water.

9.1.5 Duplicates may be required as project specific QC.

9.1.6 Spikes for total alkalinity may be required for project specific QC. Spikes are made by adding 1.0 mL 0.05 N Sodium Carbonate to 20 mL sample. The spiking concentration is 2500 x Normality of Sodium Carbonate.

It is not possible to spike pH, conductance, or the individual forms of alkalinity.

9.2 Acceptance Criteria

9.2.1 Deionized water should have a conductance of 2 umho/cm or less. The alkalinity should be less than 5 mg/L. The pH is usually 5 - 6.

9.2.2 There are no acceptance criteria for tap water. Unusual readings should be reported to your supervisor.

9.2.3 pH 10.0 standard should read within ± 2% of 10.0.

9.2.4 Control limits for accuracy and precision (RPD) on DCS samples are:

Parameter	Accuracy	Precision
pH	98 - 102%	5%
Conductance	95 - 105%	5%
Alkalinity	90 - 110%	10%

9.2.5 Standard check should be within 10% of the expected value.

9.2.6 Blanks should be less than the 2x reporting limits for conductance and alkalinity pH blanks should be around 5 to 6.

9.2.7 There are no acceptance criteria for project specific QC.

9.3 Corrective Action Required

9.3.1 Follow the corrective actions outlined in the current Enseco QAPP manual.

9.3.2 If the deionized water has a conductance more than 2 umho/cm, repeat the reading. If it is still high, the deionized water system may need maintenance. Notify your supervisor.

9.3.3 If the pH 10.0 standards is not within limits, recalibrate and check the standard again. If it is still out of limits, see your supervisor.

9.3.4 If the control limits for the DCS samples are exceeded, the problem must be corrected and the samples reanalyzed. Check the calibration procedures and thoroughly clean the electrodes, titration head, and beakers. The most common problem is contamination, which causes high conductance readings.

10. Calculations

10.1 Standardization of Alkalinity Titrant

NACID = NBASE X VBASE / VACID

NACID = Normality of titrant
NBASE = Normality of Sodium Carbonate
VBASE = Volume of Sodium Carbonate titrated, mL
VACID = Volume of titrant needed for titration, mL

10.2 pH values are recorded directly from the titrator printout.

10.3 Conductance values are taken as-is from the print out. If they are printed out in mS/cm, they must be multiplied by 1000. The units uS/cm are equivalent to umho/cm.

10.4 Three values for alkalinity will be printed if the pH is greater than 8.3. Record the values at pH 8.3 and 4.5 on the bench sheet. The third value is the difference and is ignored. Note that some results will be printed in scientific notation; be careful to check this when recording results.

If the pH is less than 8.3, only one value will be printed (at pH 4.5). Record this value on the bench sheet. P alkalinity is ND on these samples.

Calculate the individual forms of alkalinity:

T = Total Alkalinity = Alkalinity at pH 4.5
P = P Alkalinity = Alkalinity at pH 8.3

	Hydroxide Alkalinity	Carbonate Alkalinity	Bicarbonate Alkalinity
P = ND	ND	ND	T
P < T/2	ND	2 x P	T - 2xP
P = T/2	ND	T	ND
P > T/2	2xP - T	2(T - P)	ND
P = T	T	ND	ND

Record the values for Total, Bicarbonate, Carbonate, and Hydroxide Alkalinity.

11. Reporting Requirements

11.1 Units

11.1.1 pH has units of "units".

11.1.2 Conductance is reported as umho/cm at 25°C.

11.1.3 All forms of alkalinity are reported in mg/L as Calcium Carbonate.

11.2 Limits

11.2.1 All values of pH are reported as measured.

11.2.2 Conductance results less than 1 umho/cm are reported as ND.

11.2.3 Alkalinity results less than 5 mg/L are reported as ND.

11.2.4 All reporting limits must be multiplied by any prep or analytical dilution.

11.3 Significant Figures

11.3.1 pH is reported to one decimal place.

11.3.2 Significant figures:

Result	Significant Figure
<10	2
10	3
>10	3

11.4 LDMS data entry

11.4.1 The usual standards for data entry apply.

11.5 Anomalies

Document any non-standard procedures or anomalies.

Some common anomalies are:

1. Missed holding time.
2. Wrong bottle type used.

12. Review Requirements

12.1 All data packages must be complete. They must be properly signed and dated. See attached example.

12.2 Confirm that special instructions were appropriately observed.

12.3 Check the calculations. Make sure all dilution factors were properly applied.

12.4 Check the reporting limits. Remember that the reporting limits must be multiplied by any dilution factor.

12.5 Make sure all anomalies are documented.

12.6 TDS/COND ratio should fall between 0.55 and 0.81.

13. References

13.1 Source Method

13.1.1 pH is EPA Method 150.1.

13.1.2 Conductance is EPA Method 120.1.

13.1.3 Alkalinity is EPA Method 310.1.

13.2 Related Documents

13.2.1 Additional information was obtained from Standard Method #403 for the alkalinity procedure.

13.2.2 Enseco QAPP.

13.2.3 M-EQA-0002 Internal QC Checks--Laboratory Performance QC.

13.2.4 LM-RMA-1003 Alkalinity (Manual Titration)

13.2.5 LM-RMA-1047 pH (soils and wastes)

13.3 Deviations from Source Methods and Rationale

13.3.1 Successive aliquots of sample are not analyzed for pH as in the reference methods.

13.3.2 Temperature is not reported with the pH measurement. All measurements are made at room temperature (approximately 22°C).

13.3.3 Alkalinity titrations are not performed at 25°C as specified in the reference method.

13.3.4 Method 310.1 contains no provisions for determining the different forms of alkalinity. This part of the method was obtained from Standards Methods for the Examination of Water and Wastewater, 14th Edition.

13.3.5 A fixed endpoint of 4.5 is used for all alkalinity titrations.

13.4 Updates to SOP (Original to Version 1.0)

1. Format updated.
2. Significant figures changed.
3. Appendix 1.0 added for high conductance curve.
4. pH 10.0 standard will be checked with each calibration curve.

Appendix 1.0 High Conductivity Curve

1. Calibrate on saturated NaCl. The cell constant should be adjusted so the conductivity of the NaCl reads 251,000 umho/cm.

2. There is no QC.

3. Run your samples. Run DI rinses between each sample and have a double rinse cycle.

pH, CONDUCTANCE, ALKALINITY

ANALYST RS
DATE/LOT 06 AUG 90 R
SPIKE —

CONTROL (8812 / _____): pH 9.0 / _____
COND 1130 / _____
TITRANT: 0.025 N ALK 200 / _____

BKR #	SAMPLE #	VOL ml	pH	COND µS	ALKALINITIES, mg/L @8.3	@4.5	Hydr	Carb	Bicarb	Total
1	TAP		5.7	1.26	—	4.073	ND	ND	ND	ND
2	DI		5.4	150.	—	3.548	ND	ND	ND	ND
3	pH 10		10.01							
4	Blank		5.6	1.8	—	3.548	ND	ND	ND	ND
5	DCS		8.97	1134	125.6	204.5	46.7	157.8	ND	204
6	DCS		8.98	1135	128.6	209.8	47.4	162.4	ND	210
7	ABCD-01		8.9		125.6	204.5	46.7	157.8	ND	204
8	02		7.6	1520	—	49.79	ND	ND	49.8	49.8
9	ABCDI-01		4.5							
10	-02		6.2	7.0						
11	Blank		5.4	1.5	—	3.421	ND	ND	ND	ND
12	Standard		8.97	1135	125.6	204.5	46.7	157.8	ND	204

APPENDIX B-2

HARDNESS (ENSECO)

STANDARD
OPERATING
PROCEDURE

Subject or Title: Hardness (Titration)		Page 1 of 6
SOP No.: LM-RMA-1090	Revision No.: Original	Effective Date: November 7, 1988
Supersedes: None		

1. Scope and Application
   1.1 Analytes
   This method is applicable to the determination of hardness as Calcium Carbonate.

   1.2 The detection limit is 5 mg/L.

   1.3 Applicable Matrices
   This method is applicable to the analysis of drinking, surface, and saline waters, domestic and industrial wastes.

   1.4 Dynamic Range
   The range extends to about 1000 mg/L as Calcium Carbonate. This can be extended by dilution of the sample.

   1.5 Approximate analytical time is 5 minutes per sample.

2. Summary of Method
   Calcium and magnesium are titrated with EDTA. The endpoint is detected with Eriochrome Black T, which is red in the presence of calcium and magnesium and blue when these metals are complexed with EDTA.

3. Comments
   3.1 Interferences
   3.1.1 High concentrations of other metals may interfere by causing fading or indistinct endpoints.

   3.1.2 Some metals other than calcium and magnesium will titrate as hardness.

Prepared by:	Date:	11/1/88
Management Approval:	Date:	11/1/88
QA Officer Approval:	Date:	11/1/88

3.1.3 Some interferences can be controlled by the addition of various inhibitors:

Interfering Substance	Maximum Allowable Concentration (mg/L)[a] Inhibitor I	Inhibitor II	Inhibitor III
Aluminum	20	20	20
Barium	b	b	b
Cadmium	b	20	b
Cobalt	>20	0.3	0[c]
Copper	>30	20	0.3
Iron	>30	5	20
Lead	b	20	b
Manganese (II)	b	1	1
Nickel	>20	0.3	0[c]
Strontium	b	b	b
Zinc	b	200	b
Polyphosphate		10	

a Based on 25 ml sample diluted to 50 ml
b Titrates as hardness
c Inhibitor fails if substance is present

3.2 Helpful Hints
An ICP scan is helpful in determining the potential interferences present.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco/RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your supervisor or safety officer.

4.2 Wear gloves and apron when handling concentrated acids, bases and solvents. Transport only in approved carriers. Avoid breathing fumes and vapors; handle in a fume hood. Neutralize and clean up any spills immediately. In case of skin contact, flush affected area with water for at least 15 minutes. Notify your supervisor or safety officer of any spills or exposures.

4.3 Wear gloves, apron, and face shield when performing digestions or distillations. Digestions and distillations are to be performed in a fume hood.

4.4 Sodium Cyanide (Inhibitor I) is extremely toxic. Wear gloves and handle with extreme caution. Use sparingly and do not allow it to come into contact with acids. Dispose of wastes in a suitable waste container; do not pour it down a sink.

4.5 Ammonium Hydroxide should be handled only in a hood.

5. Samples Collection and Preservation

5.1 Samples are to be collected in glass or plastic bottles and acidified with Nitric Acid to pH < 2. Samples requiring dissolved hardness are to be filtered before adding Nitric Acid.

5.2 The holding time is 6 months.

6. Apparatus

6.1 Burette, pipettes, volumetric flasks, beakers, and other standard laboratory glassware.

6.2 Hot plate and other digestion equipment. See Total Metals prep SOP.

7. Reagents and Standards

7.1 Buffer Solution
Dissolve 16.9 g ammonium chloride in 143 ml concentrated ammonium hydroxide in a 250 ml volumetric flask. Add 1.25 g magnesium EDTA and dilute to volume with deionized water. Store in a tightly stoppered plastic bottle. Prepare fresh monthly or when addition of 1 or 2 ml to a sample fails to produce a pH of 10.0 $\pm$ 0.1 at the endpoint of the titration.

7.2 Inhibitor I
Sodium Cyanide powder.

7.3 Inhibitor II
Dissolve 5.0 g sodium sulfide 9-hydrate in 100 ml deionized water. This solution deteriorates rapidly through air oxidation.

7.4 Inhibitor III
Dissolve 4.5 g hydroxylamine hydrochloride in 100 ml 95% ethanol or isopropanol.

7.5 Calmagite Indicator Powder

7.6 Hydrochloric Acid, 50%
Carefully add 500 ml concentrated hydrochloric acid to 500 ml deionized water with constant mixing.

7.7 Ammonium Hydroxide, 3 N
Dilute 210 ml concentrated ammonium hydroxide to 1000 ml with deionized water.

7.8 Ammonium Hydroxide, 1 N
Dilute 70 ml concentrated ammonium hydroxide to 1000 ml with deionized water.

7.9 Methyl Red Indicator Solution
Dissolve 0.10 g methyl red in deionized water and dilute to 100 ml.

7.10 Standard Calcium Solution, 0.02 N
Place 1.000 g anhydrous calcium carbonate, primary standard grade, in a 500 ml Erlenmeyer flask. A little at a time, add 50% hydrochloric acid until all the calcium carbonate has dissolved. Add 200 ml deionized water and boil for a few minutes to drive off carbon dioxide, then cool. Add a few drops of methyl red indicator and adjust to an intermediate orange color by adding 3 N ammonium hydroxide or 50% hydrochloric acid as necessary. Transfer to a 1000 ml volumetric flask and dilute to volume with deionized water.

7.11 EDTA Titrant, 0.02 N
Dissolve 3.723 g disodium EDTA dihydrate in deionized water and dilute to 1000 ml. Standardize versus standard calcium solution. Store in a polyethylene bottle and check standardization periodically.

8. Procedure

8.1 Standardization of EDTA Titrant

8.1.1 Place 50 ml deionized water in a 125 ml Erlenmeyer flask.

8.1.2 Add 10.0 ml Standard Calcium Solution and mix.

8.1.3 Add 1 ml buffer solution and mix.

8.1.4 Add a small scoop of Calmagite indicator powder and mix.

8.1.5 Titrate slowly with continuous stirring until the last red color disappears. The end point color is blue. Complete the titration within 5 minutes of adding the buffer solution.

8.1.6 Calculate the Normality of the EDTA solution:

N EDTA = 0.2 / ml EDTA

8.2 Sample Preparation

8.2.1 For drinking waters, surface waters, saline waters, and dilutions there of, no preparation is required.

8.2.2 Wastewaters and highly polluted waters should be digested according to the SOP for preparation of Total Metals.

8.3 Analysis of Samples

8.3.1 Use 25 ml sample, or select a volume of sample which will require less than 15 ml EDTA titrant. The titration must be completed within 5 minutes of the addition of the buffer.

8.3.2 Place the sample aliquot in a 125 ml Erlenmeyer flask and dilute to a total volume of 25 ml with deionized water.

8.3.3 Neutralize with 1 N ammonium hydroxide and dilute to 50 ml with deionized water.

8.3.4 Add 2 ml of buffer solution.

8.3.5 If interferences are known or suspected, add one of the inhibitors at this point. Choose the inhibitor by knowledge of the interferences present and consultation to the table above. Use 250 mg Inhibitor I, 1 ml Inhibitor II, or 1 mL Inhibitor III.

8.3.6 Add a small scoop of Calmagite Indicator and titrate with continuous stirring until the endpoint is detected. If the endpoint is indistinct, retitrate and add Inhibitor at step 8.3.5.

8.3.7 If more than 15 ml titrant is required, retitrate a smaller aliquot of sample.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A blank (deionized water) is required with every batch of 20 or less samples.

9.1.2 Two LCS samples are required with each batch of 20 or less samples. Obtain from ERA, EPA, or other reliable source.

9.1.4 Duplicates may be required for project specific QC.

9.1.5 Spikes may be required for project specific QC. Add 5 ml of the standard calcium solution to 25 ml sample and analyze. The spiking concentration is 200 mg/L as $CaCO_3$.

9.2 Acceptance Criteria

9.2.1 Blanks must be less than the detection limit.

9.2.2 LCS recovery must be 90 - 110%.

9.2.3 The RPD for LCS samples must be less than 10 %.

9.2.4 There are no acceptance criteria for project specific QC.

9.3 Corrective Action Required

Follow the corrective actions outlined in the current Enseco QA Plan.

10. Calculations

Calculate hardness from:

HARDNESS = (VOLEDTA x NEDTA x 50000) / VSAMPLE

HARDNESS = Hardness, mg/L as $CaCO_3$
VOLEDTA = Volume EDTA required for titration, ml
NEDTA = Normality of EDTA Titrant
VSAMPLE = Volume of sample titrated, ml

11. Reporting

11.1 Reporting units are mg/L as $CaCO_3$

11.2 Samples less than the detection limit are reported as N.D.

11.3 Significant Figures

Range	Sig. Figures
5 - 10	1
10 - 99	2
100+	3

11.4 LDMS Data Entry

11.4.1 The usual standards for data entry apply.

12. References

12.1 Method source: EPA Method 130.2

12.2 Deviations from source method and rationale

None

APPENDIX B-3

AMMONIA NITROGEN (ENSECO)

Subject or Title: Nitrogen, Ammonia (Technicon AAII)		Page 1 of 9
SOP No.: LM-RMA-1114	Revision No.: Original	Effective Date: January 23, 1991
Supersedes: N/A		

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1. Scope and Application

1.1 Analytes

This method is applicable to the determination of ammonia as nitrogen.

1.2 Applicable Matrices

1.2.1 This method is applicable to the analysis of surface and saline waters, and domestic and industrial wastes. NPDES samples must be distilled before analysis. (See SOP LM-RMA-1085).

Prepared by: Jennifer Franzen	Date: January 23, 1991
Management Approval:	Date: 1/24/91
QA Officer Approval:	Date: 1/24/91

1.2.2 This method is also applicable to the analysis of soils and wastes following a DI leach (SOP LM-RMA-1074).

1.2.3 Dilutions may be made for high concentrations.

1.3 Reporting Limits

The Enseco reporting limit for ammonia is 0.10 mg/L for waters and 0.50 mg/kg for soils and wastes.

1.4 Dynamic Range

The method covers the range from 0.1 mg/L to 3.0 mg/L in waters, soils and wastes. The range may be extended by making dilutions for higher concentrations.

1.5 Analysis Time

The approximate analysis time is 2 hours for a batch of 20 samples.

2. Method Summary

Samples are analyzed on the Technicon Autoanalyzer II which performs the automated colorimetric analysis.

Ammonia is analyzed using the Berthelot Reaction, forming a blue compound with sodium phenoxide and sodium hypochlorite. The color is intensified by the addition of sodium nitroprusside. EDTA prevents the precipitation of calcium and magnesium hydroxides.

3. Comments

This method is extremely sensitive to changes in pH.

3.1 Interferences

3.1.1 Calcium and magnesium may precipitate and clog the ammonia channel. EDTA is used to prevent this. The EDTA may not be effective on salt water samples.

3.1.2 Color and turbidity in the samples will interfere. They may be removed by filtration. Samples with color interferences must be diluted until no interference is detected.

3.1.3 The samples, standards, and rinse water must all contain the same concentration of acid.

3.2 Helpful Hints

3.2.1 Samples, standards, and rinse water work best if the concentration of the acid is 0.2% sulfuric acid.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco-RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your supervisor or safety officer.

4.2 Wear gloves and apron when handling concentrated acids, bases and solvents. Transport only in approved carriers. Avoid breathing fumes and vapors; handle in a fume hood. Neutralize and clean up any spills immediately. In case of skin contact, flush affected area with water for at least 15 minutes. Notify your supervisor or safety officer of any spills or exposures.

4.3 No solvents of any kind or in any amount are to be disposed of in the sinks. Dispose of all solvents in suitable waste containers.

4.4 Wear gloves and apron when performing extractions. Extractions are to be performed in a fume hood.

4.5 Wear gloves, apron, and face shield when performing digestions or distillations. Digestions and distillations are to be performed in a fume hood.

4.6 Samples and other solutions containing high concentrations of toxic materials must not be flushed down the sinks, but are to be disposed of in suitable waste containers.

4.7 All samples are to be considered hazardous. Wear gloves and handle in a fume hood as much as possible.

4.8 Phenol is highly toxic and can be absorbed through the skin or by inhalation of the vapor. Wear gloves and handle in the fume hood.

5. Sample Collection, Preservation and Holding Times

5.1 Samples are to be collected in glass bottles whenever possible.

5.2 Samples are to be preserved by adding sulfuric acid (2 mL per 1000 mL sample) and refrigerated at 4°C.

5.3 The holding time for ammonia in water is 28 days. There is no holding time for soils or wastes.

6. Apparatus

6.1 The technicon AAII equipped with the proportioning pump, autosampler, and colorimeter with 660-nm filters and 50-mm flow cell.

6.2 Miscellaneous laboratory apparatus and glassware.

7. Reagents and Standards

7.1 ASTM Type II Water (Milli-Q or equivalent).

7.2 Sulfuric acid 0.2%

Slowly add 4 mL of concentrated sulfuric to 2 liters of ASTM type water. This will be used to make standards and any dilutions that may be required.

7.3 Sodium phenolate

Add 20.75 g of phenol (crystals) to 100 mL of distilled water in a 250 mL volumetric. Do this in the hood. Allow the phenol to dissolve some then add 8.0 g of sodium hydroxide pellets. Cool to room temperature and bring to volume. Store in an amber glass container and prepare fresh every two weeks.

7.4 Sodium Hypochlorite Solution

Dilute 100 mL of reagent grade Hypochlorite (5%) to 200 mL with distilled water. Commercial bleach (5%) may be used in the same manner.

7.5 Sodium Nitroprusside Solution (0.05%)

Dissolve 0.125 g of sodium nitroprusside in 250 mL of deionized water. Store in an amber container and prepare fresh monthly.

7.6 Disodium EDTA

Add 50 g of sodium EDTA to 800 mL of deionized water. Use a pH electrode to adjust the pH to 8.0 by slow addition of 50% sulfuric acid. Cool to room temperature and bring to volume. Add 1 mL of Brij-35 and mix.

7.7 Ammonia Stock Standard

Dry Ammonium Chloride at 105°C. Dissolve 3.819 g in 600 mL of deionized water, add 2 mL of concentrated sulfuric acid, and dilute to 1000 mL with deionized water. This solution is to be obtained from standards prep.

7.8 Intermediate Standard

Pipette 1.0 mL of stock standard into a 100 mL volumetric flask and dilute to volume with 0.2% sulfuric acid.

7.9 Working Standards

Prepare dilutions as indicated below. All dilutions should be made with 0.2% sulfuric acid.

Volume Intermediate Standard	Final Volume	Concentration mg/L as N
1.0 mL	100 mL	0.1
2.0 mL	100 mL	0.2
5.0 mL	100 mL	0.5
10.0 mL	100 mL	1.0
20.0 mL	100 mL	2.0
30.0 mL	100 mL	3.0

7.10 DCS Solution

Use ERA nutrients for DCS solutions. These must be preserved properly with sulfuric acid to 0.2%.

8. Procedure

8.1 Sample Preparation

8.1.1 Check the pH of all samples to ensure that they have been properly preserved. If necessary, add sulfuric acid to produce a final concentration of 0.2% and indicate on the benchsheet.

8.1.2 Soils and wastes must be taken through the D.I. Leach procedure before analysis. (LM-RMA-1074)

8.1.3 NPDES samples for ammonia must be distilled before analysis (LM-RMA-1085).

8.2 Instrument Set-up and Calibration

8.2.1 Turn on the colorimeter and allow 20 min. warmup time.

8.2.2 Set up the proportioning pump for pumping reagents. Pump all reagents except Nitroprusside. The pH needs to adjust before pumping this reagent. Pump the Nitroprusside after the other reagents have been pumping for five minutes.

8.2.3 Turn on the chart recorder and allow the baseline to stabilize.

8.3 Analysis

8.3.1 Standards are to be analyzed in order of decreasing concentration. A linear fit should be used.

8.3.2 Samples exceeding the high standard require a dilution. Samples below 0.1 mg/L are reported as ND.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A blank (0.2% sulfuric acid) is required with every batch of 20 or less samples.

9.1.2 Two DCS samples are required for each batch of 20 or less samples. Use ERA nutrients.

9.1.3 Duplicates may be required for project specific QC.

9.1.4 Spikes may be required for project specific QC. Dilute 1 mL Intermediate Standard to 10 mL with sample. The spiking concentration is 1 mg/L.

9.2 Acceptance Criteria

9.2.1 Blanks must be less than two times the reporting limit.

9.2.2 Average DCS recovery must be 93-107% .

9.2.3 The RPD for the DCS must be less than 10%

9.2.4 There are no acceptance criteria for project specific QC.

9.2.5 Standard checks must be within 10% of the expected value.

9.2.6 The calibration curve must have a correlation coefficient of 0.995 or greater.

9.3 Corrective Actions

9.3.1 Follow the corrective actions outlined in the current Enseco QAPP Manual.

9.3.2 If the blank is more than two times the reporting limit, the entire batch of samples must usually be reprepped and reanalyzed. Consult your supervisor.

9.3.3 Check for contamination in reagents and standards. Be sure the reagents were made correctly and have not exceeded their expiration dates.

9.3.4 All samples and standards must contain 0.2% sulfuric acid.

10. Calculations

10.1 Measure and record the peak height for each sample.

10.2 Prepare a calibration curve from the standard data. Use a linear least squares program.

10.3 Calculate the sample concentrations from the calibration curve and multiply by any dilution factors. Be sure to include dilutions made during prep.

11. Reporting Requirements

11.1 Reporting units are mg/L for waters and mg/kg for non-aqueous samples.

11.2 Limits

11.2.1 If the blank shows contamination (but is less than two times the reporting limit), the reporting limit is raised to the blank value.

11.2.2 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor.

11.3 Significant Figures

11.3.1

Range	Sig. Figures
< 10	2
10	3
> 10	3

11.3.2 Observed values below the detection limit must be reported as not detected (ND) .

11.4 LIMS Data Entry

The usual standards for data entry apply.

11.5 Anomalies

Document any non-standard procedures or anomalies. Some common anomalies are:

11.5.1 Sample diluted for color interference.

11.5.2 Sample pH was neutral preserved with sulfuric acid before analysis.

12. Review Requirements

12.1 All data packages must be complete. They must be properly signed and dated.

12.2 Confirm that the special instructions were appropriately observed.

12.3 Confirm that there were no unusual peaks by checking the chromatogram.

12.4 Check calculations.

12.5 Check the reporting limits. The standard reporting limit must be multiplied by any dilution factor or raised to any blank contamination value.

12.6 Verify that the results were correctly anomalized.

13. References

13.1 Source Method: EPA 350.1 Nitrogen, Ammonia (Colorimetric, Automated Phenate)

13.2 Related Documents

13.2.1 4500-NH3.H Automated Phenate Method Standard Methods

13.2.2 Enseco QAPP

13.2.3 M-EQA-0002 Internal QC Checks--Laboratory Performance QC

13.2.4 Enseco SOP LM-RMA-1039 Rev. 1.0 Nitrogen Ammonia 350.1 EPA

13.3 Deviations from Source Method and Rationale

13.3.1 The source method has been expanded to include soils and wastes through the use of a deionized water leach.

13.3.2 The EDTA solution is brought to a specific pH by pH electrode in order to maintain a consistent pH for all analysis.

(Attached are completed example benchsheet and appropriate diagrams or tables.)

TECHNICON

Analyst ANALYST Control (Example ERA 9932 10.6) Date ( )

Parameter NH_3 Spike 1.0 mg/l Units mg/l mg/kg

Cup #	Sample	pH	Bottle Type	Reading	Initial Conc.	Dilution Factor Prep	Dilution Factor Anlyt.	Final Conc.	% Recovery RPD
1	3.0								
2	2.0								
3	1.0								
4	.5								
5	.1								
6	0.0								
7	RN								
8	DCS 1								
9	DCS 2								
10	PBIK								
11	sample	<2	#2			1X	1X		
12			#2						
13			#2						
14			#2						
15									
16						5X	1X		
17									
18									
19									
20									
21	CCV								
22	CCB								

APPENDIX B-4

TOTAL DISSOLVED SOLIDS (ENSECO)

STANDARD
OPERATING
PROCEDURE

Subject or Title:		Page 1 of 11
Total Dissolved Solids (TDS)		
SOP No.: LM-RMA-1060	Revision: 4.0	Effective Date: May 21, 1991

Supersedes: 3.0

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1. Scope and Application

1.1 Analytes

This method is used for the determination of Total Dissolved Solids, also known as Filterable Residue.

1.2 Reporting Limits

The method reporting limit is 10 mg/L.

1.3 Applicable Matrices

This method is applicable to drinking, surface, and saline waters, domestic, and industrial wastes.

Prepared by	Date:
Tom J Stovall	May 22 1991
Management Approval:	Date:
David B Rohrbaugh	5-22-91
QA Officer Approval:	Date:
Bill Shaw	05/22/91

1.4 Dynamic Range

This method covers the range from 10 mg/L and up. It is a gravimetric method and there is no upper limit. As a practical matter, the final residue weight should be limited to about 200 mg.

1.5 Analysis Time

The analytical time is about 30 minutes per sample, not including drying time. The total drying time is variable and can be as long as 24 to 48 hours.

2. Method Summary

A well-mixed sample is filtered through a glass fiber filter. The filtrate is evaporated at 103°C and dried to a constant weight at 180°C.

3. Comments

3.1 Interferences

3.1.1 Some samples may contain hygroscopic salts which require prolonged drying, desiccation, and rapid weighing.

3.1.2 Samples containing high concentrations of bicarbonate may require careful prolonged drying to ensure that all the bicarbonate is converted to carbonate.

3.1.3 Too much residue in the pans will crust over, entrapping water that will not be driven off during drying. Total residue should be limited to about 200 mg.

3.1.4 Oil and grease in the samples will cause unreliable results due to difficulty in drying to constant weight.

3.1.5 Some samples may have fine suspended solids which pass through the glass fiber filter causing high results.

3.1.6 Aluminum pans should not be used. Components in some samples may react to form aluminum compounds causing unreliable results.

3.2 Helpful Hints

3.2.1 Conductance multiplied by a factor between 0.55 and 0.81 gives a rough approximation of TDS. The exact relationship depends on the compounds present in the samples and may not hold for very high concentrations or samples containing nonionic species or samples with conductances greater than 10,000 umho/cm or less than 50 umho/cm.

3.2.2 The filtrate from the TSS procedure (LM-RMA-1062) may be used for TDS determination.

4. Safety Issues

Normal laboratory safety procedures should be followed. There are no special concerns with this method.

5. Sample Collection, Preservation, Containers and Holding Times

5.1 Samples are to be collected in plastic bottles. A minimum of 100 mL is required and may or may not be filtered in the field.

5.2 Refrigerate all samples to 4°C.

5.3 The holding time for TDS is 7 days.

6. Apparatus

6.1 Glass fiber filter disks, 47 mm (Gelman Type A/E). The filters should be rinsed with deionized water and dried before use. Assemble the filtration apparatus, turn on the vacuum pump, and place a filter on the holder. Rinse the filter with three 20 mL portions of deionized water, dry at 103°C and store in a desiccator.

6.2 Vacuum filtration apparatus.

6.3 Vacuum pump equipped with moisture trap.

6.4 Porcelain evaporating dishes. These must be thoroughly cleaned, rinsed with deionized water, and baked at 180°C for at least one hour before use. Store in a desiccator.

6.5 Desiccators providing space for storage of samples in process separate from filters and evaporating dishes.

6.6 Drying ovens set at 103°C and 180°C. Separate ovens should be maintained at the appropriate temperatures if possible.

6.7 Steam bath

6.8 Thermometers, NBS traceable.

6.9 Analytical balance capable of measurements to 0.0001 g.

6.10 Conductivity meter and associated apparatus.

6.11 Tweezers, graduated cylinders, and other general laboratory apparatus. Glassware must be thoroughly washed with detergent and rinsed with deionized water.

7. Reagents and Standards

7.1 Deionized water (ASTM Type II or equivalent)

7.2 All reagents and standards required for measurement of conductance (see SOP LM-RMA-1062).

7.3 DCS Solution

Use an ERA Minerals Sample or a sample from another reliable source.

8. Procedure

8.1 Preparation

8.1.1 Check samples for special instructions, making copies to include with the data package. Obtain samples from sample receiving. Fill out the benchsheet. See example benchsheet.

8.1.2 Measure and record the conductance of each sample before analysis. An accurate value, corrected for temperature and cell constant, must be obtained. If the conductance has already been measured, this value may be used. Check the Short Holding Time Sheets; TDS has been placed on them in order to identify all samples requiring conductivity. **THE CONDUCTANCE MUST BE RECORDED ON THE BENCH SHEET FOR EACH SAMPLE.**

8.1.3 If samples are visibly oily, this should be noted on the benchsheet.

8.1.4 Mark the evaporating dishes with appropriate identification. Weigh dishes for each sample to the nearest 0.0001 g. Only use dishes which have previously been baked at 180°C and stored in a desiccator.

8.1.5 Determine the proper volume of each sample to be analyzed.

8.1.5.1 If the sample has a conductance less than 3,000 umho/cm, 100 mL should be used.

8.1.5.2 If the conductance is 3,000 to 10,000 umho/cm, use 20 mL of sample. Dilute volumes of <100 mL to 100 mL final volume with deionized water.

8.1.5.3 If the conductance is extremely high (more than about 10,000 umho/cm), smaller volumes may be used. Consult your supervisor.

8.1.5.4 If there is limited sample volume, a smaller amount of sample must be analyzed. This must be noted on an anomaly sheet.

8.2 Analysis

8.2.1 Thoroughly rinse the entire filtration apparatus with deionized water before filtering each sample. Assemble the filtration apparatus and turn on the vacuum pump. Only use filters which have been previously rinsed, dried, and stored in a desiccator. The patterned side of the filter must face down.

8.2.3 Shake the sample vigorously, measure the required volume into a graduated cylinder, and rapidly transfer to the filtration apparatus. All samples are to be filtered, whether or not they have been filtered in the field.

NOTE: If the sample contain large amounts of insoluble material, a portion may be poured off after mixing and allowed to settle or an aliquot maybe taken and centrifuged, before filtering. Do not pour directly

from the bottle without mixing. This will alter the sample composition and adversely affect subsequent analyses.

8.2.4 After the sample has been filtered, rinse the graduated cylinder into the filtration apparatus with three 10 mL portions of deionized water. Continue vacuum for three minutes after filtration is complete.

8.2.5 Transfer the filtered sample to the preweighed evaporating dish. Rinse the receiving flask into the dish to ensure complete transfer of the sample.

8.2.6 Place the samples in the drying oven set at 103°C and evaporate to dryness. Record the date and time that the samples were placed in the oven and the temperature of the oven on the benchsheet. Alternately, the samples may be placed on a steam bath and evaporated to dryness.

8.2.7 Prepare new filters and dishes to replace the ones you have used. Instructions are given in Sections 6.1 and 6.4 respectively.

8.2.8 When the samples are dry, transfer to the drying oven set at 180 +/- 2°C and heat for a minimum of one hour. If a number of samples are being dried simultaneously, additional time may be necessary. Record the date and time that the samples are placed in the drying oven. In addition, record the oven temperature on the bench sheet at the beginning and end of the drying period.

8.2.9 Cool the samples in a desiccator for two to three hours. Remove and weigh one tray crucibles at a time to prevent absorption of moisture. Record the date and time of this measurement on the bench sheet.

8.2.10 Return the samples to the oven and bake for another hour. Cool the samples in a desiccator and weigh. Record the date, time, and oven temperatures as above. Repeat this cycle again if the weight changes by more than 1.0 mg. If a constant weight is not achieved within three drying cycles, note this on an anomaly sheet. Do not continue the drying, cooling, weighing cycle beyond three times.

8.3 Conclusion

8.3.1 Clean all glassware, apparatus and the work area.

8.3.2 Return all samples to sample receiving.

8.3.3 Calculate the results following the calculation in section 10. Circle the lowest final weight of the two weights that varied between 1.0 mg. This is used as the final weight for calculating TDS. NOTE: If a constant weight is not achieved within three drying cycles, circle the lowest final weight. This is used as the final weight for calculating TDS.

8.3.4 Complete the data package making sure it is properly filled out and the results are highlighted. See sample example. Be sure to include copies of the special instructions.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A blank is required with every batch of 20 or less samples. The blank is prepared using 100 mL of deionized water. It is to be taken through all steps of the analysis, including filtration. It is not necessary to measure the conductance.

9.1.2 A DCS pair is required with each batch of 20 or less samples. These are to be taken through all steps of the analysis, including filtration. It is not necessary to measure their conductances. DCS are prepared using 100 mLs of standard.

9.1.3 Duplicates may be required for project specific QC.

9.1.4 Spikes may be required for project specific QC. Spikes may be analyzed by mixing equal volumes of DCS and sample. Calculation of the result should be based on the actual volume of sample used in the spike (one half the total volume). The "spiking concentration" is equal to the DCS value.

9.2 Acceptance Criteria

9.2.1 Weight change for blanks must be between -0.5 and 2 mg.

9.2.2 Average DCS recovery must be 90 - 110%.

9.2.3 The RPD for the DCS must be less than 10%.

9.2.5 There are no acceptance criteria for project specific QC.

9.3 Corrective Action Required

9.3.1 Follow the corrective actions outlined in the current Enseco QAPP Manual.

9.3.2 Poor results for the DCS's is usually due to contamination. Be sure all glassware and the filtration apparatus is clean and rerun the samples.

9.3.3 High blanks are also the result of contamination. Low blanks indicate that the evaporating dishes were not completely dry when the initial weight was measured. Rerun the samples.

9.3.4 If the TDS/COND ratio falls outside the limits (0.55-0.81) the sample should probably be rerun for conductance and then for TDS. Consult your supervisor.

10. Calculations

10.1 Calculate blank residue weight:

BLANK = (W2 - W1) X 1000

BLANK = Blank residue weight, mg
W2 = Final blank weight, g
W1 = Initial blank weight, g

10.2 Calculate samples and DCS's:

RESIDUE = (W2 - W1) X 1000

TDS = (RESIDUE) X 1000 / VOL

RESIDUE = Sample residue weight, mg
W2 = Final weight, g
W1 = Initial weight, g
TDS = TDS result, mg/L
VOL = Sample Volume, mL

10.3 Calculate the reporting limit

Rep. Limit (mg/L) = Residue (mg) X 1000 / Vol. (mL)

This is to be done only if the blank is greater than 1 mg or if a sample volume less than 100 mLs was used. Otherwise, the reporting limit is 10 mg/L.

If the blank is between 1 and 2 mg, the reporting limited is based on the blank residue weight. In all other cases, use 1 mg.

The reporting limit is based on either 100 mL OR the actual volume used, whichever is lower.

10.4 Calculate the TDS/COND ratio.

TDS/COND ratio = Measured TDS / Measured Conductivity

11. Reporting Requirements

11.1 Units

Report all results in mg/L

11.2 Limits

Results below the reporting limit are reported as ND.

11.3 Significant Figures

Significant figures to report are:

Range	Significant Figures
<10	2
≥10	3

DCS's are reported to three significant figures

11.4 LIMS Data Entry

Since drying times will vary, the analysis date reported will be the date on which the samples were filtered. The holding time is calculated from this analysis date.

11.5 Anomalies

Document any non-standard procedure or any anomalies. Some common anomalies are:

1. Insufficient sample. Reporting limits changed accordingly.

2. Filtrate is turbid.

3. TDS/COND ratio is outside the limits. TDS & COND were rerun and confirmed. The sample contains high concentration of TDS or may contain nonionic species. The conductivity is greater than 10,000 or less than 50 umho/cm so the rule does not apply.

4. Holding time missed.

5. Samples are visibly oily.

See the attached example benchsheet

12. Review Requirements

12.1 All data packages must be complete. They must be properly signed and dated. See example benchsheet.

12.2 Confirm that the special instructions were appropriately observed.

12.3 Check all sample calculations.

12.4 Check the reporting limits.

12.5 Verify that all anomalies are documented.

12.6 The TDS/COND ratio should fall between 0.55 - 0.81. This ratio may not hold for nonionic species or samples high in TDS. It also may not hold for samples with conductances greater than 10,000 or less than 50 umho/cm. Anomalize results that do not fall within these limits.

13. References

13.1 Source Method: EPA Method 160.1

13.2 Related Documents

13.2.1 Standard Methods "Total Dissolved Solids Dried at 180°C", 17th Edition, 1989.

13.2.2 Enseco QAPP

13.2.3 M-EQA-0002 Internal QC Checks--Laboratory Performance QC

13.2.4 LM-RMA-1071: pH, Specific Conductance, and Alkalinity: Automatic Titrator

13.2.5 LM-RMA-1062: Total Suspended Solids (TSS)

13.3 Deviations from Source Method and Rationale

13.3.1 A 103° oven may be used for the evaporation of the sample to dryness.

13.3.2 Constant weight, as defined by the Inorganic Technical Committee of Enseco, is a weight change of 1.0 mg between weighings.

13.3.3 Samples will not be taken through the drying, cooling, weighing cycle more than three times. This is a policy approved by the Enseco Inorganic Technical Committee.

13.4 Updates to SOP (Revision 3.0 to Revision 4.0)

13.4.1 Provision for evaporation of samples on steam bath included.

13.4.2 Maximum number of drying cycles stipulated.

13.4.3 Constant weight defined as weight change of 1.0 mg.

APPENDIX B-5

CHLORIDE, SULFATE, AND NITRATE NITROGEN (ENSECO)

Anion Analysis by Ion Chromatography

SOP No.:	Revision No.:	Effective Date:
LM-RMA-1025	2.0	12/15/90

Supersedes:
1.0 (3/16/88)

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1. Scope and Application

1.1 Analytes

This method is applicable to the determination of the following inorganic anions:

Fluoride	Nitrate (as N)
Chloride	Ortho-Phosphate (as P)
Bromide	Sulfate

Prepared by: Roxanne Sullivan	Date: November 19, 1990
Management Approval:	Date: 12/12/90
QA Officer Approval: Jan Rederbarger	Date: 12/12/90

1.2 Applicable Matrices

1.2.1 This method is applicable to drinking and surface waters as well as mixed domestic and industrial wastewaters.

1.2.2 The method may be applied to nonaqueous matrices by analyzing a deionized water leachate of the sample (SOP LM-RMA-1074 D.I. Leach).

1.3 Reporting Limits

Enseco's standard reporting limits are:

Analyte	Water (mg/L)
Fluoride	0.5
Chloride	3
Bromide	0.1
Nitrate (as N)	0.1
Ortho-Phosphate (as P)	0.5
Sulfate	5

Lower reporting limits may be available under special conditions. Consult the laboratory for details.

Nonaqueous reporting limits, when determined using DI leach SOP LM-RMA-1074, will be a factor of 5 higher than the above aqueous limits.

1.4 Dynamic Range

The dynamic range of the method is anion specific as indicated in the following table and can be extended by diluting the sample:

Analyte	Dynamic Range (mg/L)	Approximate Retention Times
Fluoride	0.5 - 50	1.0
Chloride	3 - 500	1.6
Nitrite	-------	<1.3
Bromide	0.1 - 50	2.3
Nitrate	0.1 - 50	3.2
Ortho-phosphate	0.5 - 50	5.9
Sulfate	5 - 500	6.5

1.5 Analysis Time

The approximate analysis time for water samples is 10 minutes, not including data reduction. Nonaqueous samples require a DI leach, whose approximate preparation time is 2 hours.

2. Method Summary

A small volume of sample is introduced into the Dionex ion chromatograph. The anions of interest are separated and measured using a system comprised of a guard column, separator column, suppressor column, and conductivity detector. Results are processed using Dionex software.

3. Comments

3.1 Interferences

3.1.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems.

3.1.2 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

3.1.3 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

3.2 Helpful Hints

3.2.1 The water dip or negative peak that elutes near, and can interfere with, the fluoride peak can be eliminated by the addition of the equivalent of 1 mL of concentrated eluent (7.3 100X) to 100 mL of each standard and sample.

3.2.2 Large concentrations of any one ion (such as chloride in brines) may affect the retention time of the other ions and mask peaks.

3.2.3 Molybdenum has the same retention time as sulfate; nitrite elutes very close to chloride.

3.2.4 This method is not recommended for leachate samples when acetic acid is used for pH adjustment. The retention times of the anions differ when large amounts of acetate are present.

3.2.5 This method is not recommended for fluoride. Any anion that is not retained by the column will elute in the area of fluoride and interfere.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco-RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any question or safety concerns, see your supervisor or safety officer.

4.2 Normal accepted laboratory safety practices should be followed during reagent preparation and instrument operation.

5. Sample Collection, Preservation, and Holding Times

5.1 Samples should be collected in scrupulously clean glass or polyethylene bottles.

5.2 Samples are to be preserved and analyzed in accordance with the following:

Analyte	Preservation	Holding Time
Fluoride	None	28 Days
Chloride	None	28 Days
Bromide	4°C	ASAP
Nitrate (as N)	4°C	48 Hrs
O-Phos (as P)	Filter, 4°C	48 Hrs
Sulfate	4°C	28 Days

6. Apparatus

6.1 Balance - Analytical, capable of accurately weighing to 0.0001 g.

6.2 Ion chromatograph - Analytical system complete with ion chromatograph and all required accessories.

6.2.1 Anion guard column: AG4A, 4X50mm, Dionex P/N 37042.

6.2.2 Anion separator column: AS4A, 4X250MM, Dionex P/N 37041.

6.2.3 Anion micromembrane suppressor (AMMS): Dionex P/N 38019.

6.2.4 Detector - Conductivity cell: approximately 6 mL volume.

6.3 Sample bottles: Glass or polyethylene of sufficient volume to allow replicate analyses of anions of interest.

6.4 Miscellaneous laboratory glassware and pipettes.

7. Reagents and Standards

7.1 Reagent water: Distilled or deionized water, free of the anions of interest and containing particles no larger than 0.20 microns.

7.2 Eluent solution: Sodium bicarbonate (CAS RN 144-55-8) 0.0025M, sodium carbonate (CAS RN 497-19-8) 0.00075M. Dissolve 1.0081 g sodium bicarbonate ($NaHCO_3$) and 1.0176 g of sodium carbonate (Na_2CO_3) in reagent water and dilute to 4 liters.

7.3 Regeneration solution (AMMS): Sulfuric acid (CAS RN 7664-93-9) 0.025N. Dilute 2.8 mL conc. sulfuric acid (H_2SO_4) to 4 liters with reagent water.

7.4 Stock standards: May be purchased as certified solutions or prepared from ACS reagent grade materials (dried at 105°C for 30 min) as listed below; may be prepared individually or as one stock standard.

7.4.1 Fluoride (F) 1000 mg/L: Dissolve 2.2100 g sodium fluoride (NaF CAS RN 7681-49-4) in reagent water and dilute to 1 L.

7.4.2 Chloride (Cl) 10,000 mg/L: Dissolve 16.485 g sodium chloride (NaCl CAS RN 7647-14-5) in reagent water and dilute to 1 L.

7.4.3 Bromide (Br) 1000 mg/L: Dissolve 1.288 g sodium bromide (NaBr CAS RN 7647-15-6) in reagent water and dilute to 1 L.

7.4.4 Nitrate (NO_3-N) 1000 mg/L: Dissolve 6.0679 g sodium nitrate ($NaNO_3$ CAS RN 7631-99-4) in reagent water and dilute to 1 L.

7.4.5 Phosphate (PO_4-P) 1000 mg/L: Dissolve 4.3937 g potassium phosphate (KH_2PO_4 CAS RN 7778-77-0) in reagent water and dilute to 1 L.

7.4.6 Sulfate (SO_4) 10,000 mg/L: Dissolve 18.141 g potassium sulfate (K_2SO_4 CAS RN 7778-80-5) in reagent water and dilute to 1 L.

These stock standards are stable for at least one month when stored at 4°C. Dilute working standards should be prepared weekly, except phosphate, which should be prepared fresh daily.

7.5 Working standards: Make up individual working standards for each anion as follows:

Analyte	Concentration (mg/L)	Stock Std. (mLs)	Final Volume (mLs)
Fluoride	100	10	100
Chloride	1000	10	100
Bromide	100	10	100
Nitrate	100	10	100
O-Phos	100	10	100
Sulfate	1000	10	100

7.6 Stock DCS solutions, 1000 ppm.

Stock DCS solutions may be purchased as certified solutions or prepared from ACS reagent grade materials (dried at 105° for 30 minutes) obtained from standards prep as listed below. The stocks may be prepared individually or as one stock DCS. 1000 mg/L DCS stock solutions should be made from a different source than the calibration stocks.

7.7 Working DCS solution

Dilute down the stock DCS solutions into one 100 mL volumetric as follows:

Analyte	Final Concentration (mg/L)	Stock Stds. (mLs)
Fluoride	10	1
Chloride	100	10
Bromide	10	1
Nitrate	10	1
O-Phos	10	1
Sulfate	100	10

7.8 CCV Solution

Prepare a CCV solution at the following concentrations from the working std. solutions in a 100 mL volumetric.

Analyte	Concentration	Working Std. (mLs)
Fluoride	25	2.5
Chloride	250	2.5
Bromide	25	2.5
Nitrate	25	2.5
O-Phos	25	2.5
Sulfate	250	2.5

8. Procedure

8.1 Preparation

8.1.1 Obtain samples from sample receiving. Check each sample for special instructions making copies to put along with the data package. Fill out your benchsheet.

8.1.2 Unless particulate matter is present, water samples are analyzed directly without advanced preparation. Samples containing particulate matter must be filtered through a 0.45 um filter prior to analysis.

8.1.3 Soil samples are prepared by leaching one part sample with five parts deionized water.
See SOP: LM-RMA- 1074 D.I. Leach.

8.2 Instrument Set-up and Calibration

8.2.1 Establish the following ion chromatographic operating parameters:

Columns - AS4A Separator
AGYA Guard
Anion Micromembrane Suppressor

8.2.2 Prepare a series of standards at the following concentrations. Each standard should be made up in 100 mL volumetric with eluent. This is the normal operating curve for Rocky. Other curves can be made to meet client request such as USGS.

Std. 1 is a eluent blank

Std. 2	Concentration (mg/L)	Working/Stock Std. to be diluted (ppm)	mLs
Fluoride	0.1	100	0.1
Chloride	1	1000	0.1
Bromide	0.1	100	0.1
Nitrate	0.1	100	0.1
O-Phos	0.1	100	0.1
Sulfate	1	1000	0.1

Anions by IC

SOP No.:	Revision No.:	Effective Date:
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Std. 3	Concentration (mg/L)	Working/Stock Std. to be diluted (ppm)	mLs
Fluoride	1	100	1
Chloride	10	1000	1
Bromide	1	100	1
Nitrate	1	100	1
O-Phos	1	100	1
Sulfate	10	1000	1

Std. 4	Concentration (mg/L)	Working/Stock Std. to be diluted (ppm)	mLs
Fluoride	10	100	10
Chloride	100	1000	10
Bromide	10	100	10
Nitrate	10	100	10
O-Phos	10	100	10
Sulfate	100	1000	10

Std. 5	Concentration (mg/L)	Working/Stock Std. to be diluted (ppm)	mLs
Fluoride	50	1000	5
Chloride	500	10,000	5
Bromide	50	1000	5
Nitrate	50	1000	5
O-Phos	50	1000	5
Sulfate	500	10,000	5

Eluent - 0.75 m M $NaHCO_3$ (0.0630 g/L)
2.00 m M Na_2CO_3 (0.2120 g/L)

Regenerant -1.025 N H_2SO_4
2.0 mL of 50% H_2SO_4
per 2000 mL
Sample Loop -100 mL

Flow Rate - 2.0 mL/min.

8.2.3 Using injections of 100 uL of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte.

8.2.4 The working calibration curve must be verified on each working day, or whenever the anion eluent is changed, and after every 10 samples. If the response or retention time for any analyte varies from the expected values by more than ± 10%, the test must be repeated, using fresh calibration standards. If the results are still more than ± 10%, an entire new calibration curve must be prepared for that analyte.

8.2.5 Non-linear response can result when the separator column capacity is exceeded (overloading). Maximum column loading (all anions) should not exceed about 5000 ppm.

8.2.6 Linear calibration curve will be used. A study was done using both linear and quadratic calibration and the curves were found to be equivalent.

8.3 Analysis

8.3.1 Check system calibration daily and, if required, recalibrate.

8.3.2 Load and inject a fixed amount of well mixed sample. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units. An automated constant volume injection system may also be used.

8.3.3 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for a compound. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

8.3.4 Different dilutions may be required to obtain the concentration of individual anions at the lowest possible reporting limit.

8.3.5 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, spike the sample with an appropriate amount of standard and reanalyze.

8.4 Conclusion

8.4.1 Shut off the I.C. Clean all glassware, apparatus, and the work area.

8.4.2 Return all samples to sample receiving.

8.4.3 Calculate the results according to the calculation section below if not done with the IC software.

8.4.4 Complete the data package making sure it is filled out properly and all results are highlighted. See the attached example. Be sure to include copies of the special instructions. Submit the package to your supervisor for approval.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A blank is required with every batch of 20 or less samples.

9.1.2 Duplicate laboratory control samples must be analyzed with every batch of 20 or less samples.

9.1.3 Continuing calibration blank (CCB) and continuing calibration verification (CCV) samples must be analyzed after every tenth sample. A CCV and CCB must also be analyzed at the completion of the day's analyses.

9.1.4 Duplicates may be required for project specific QC.

9.1.5 Spikes may be required for project specific QC.

9.2 Acceptance Criteria

9.2.1 Blanks must be less than two times the reporting limit.

9.2.2 Average DCS requirements are summarized in the following table:

Analyte	Accuracy Control Limits*
Fluoride	88 - 112
Chloride	92 - 108
Bromide	90 - 110
Nitrate	91 - 109
Ortho-Phos	75 - 125
Sulfate	93 - 107

9.2.3 The requirements for the RPD for the DCS are summarized in the following table:

Analyte	Precision Control Limits*
Fluoride	15% RPD
Chloride	10% RPD
Bromide	20% RPD
Nitrate	20% RPD
Ortho-Phos	20% RPD
Sulfate	15% RPD

* Subject to change in accordance with Enseco QAPP.

9.2.4 For the DCS data to be considered acceptable, 80% of the acceptance criteria in Sections 9.2.2 and 9.2.3 must be achieved if an IC scan is required.

9.2.5 Standard checks must be within 10% of the expected value.

9.2.6 There are no acceptance criteria for project specific QC.

9.2.7 The calibration curve for each analyte must have a correlation coefficient of 0.995 or greater.

9.3 Corrective Actions

9.3.1 If acceptance criteria described in Section 9.2 are not met, the analysis sequence must be terminated until the source of the problem can be identified and corrected.

9.3.2 If the blank is more than two times the reporting limit, the entire batch of samples must usually be reprepped/reanalyzed or anomalized. Consult your supervisor.

9.3.3 Follow the corrective actions outlined in the current Enseco QAPP Manual.

10. Calculations

10.1 Prepare separate calibration curves for each anion of interest by plotting peak size in area or peak height units of standards against concentration values.

10.2 Compute sample concentration by comparing sample peak response with the standard curve.

10.3 Calculations can be done automatically using the Nelson software.

11. Reporting Requirements

11.1 Units

Report results in mg/L for aqueous samples and mg/kg for soil samples.

11.2 Limits

11.2.1 The reporting limits for the target anions are listed in the following table:

Analyte	Reporting Limit Water (mg/L)
Fluoride	0.5
Chloride	3
Bromide	0.1
Nitrate	0.1
Ortho-Phos	0.5
Sulfate	5

11.2.2 If dilutions were required for any reason the reporting limit is multiplied by the dilution factor.

11.2.3 If the blank shows contamination (but less than two times the reporting limit) the reporting limit is raised to the blank value.

11.3 Significant Figures

11.3.1

Range	Sig. Figures
< 10	2
10	3
> 10	3

11.3.2 Observed values below the detection limit must be reported as not detected (ND).

11.4 LIMS Data Entry

The usual standards for data entry apply.

11.5 Document any non-standard procedure or any anomalies.

12. Review Requirements

12.1 All data packages must be complete. They must be properly signed and dated.

12.2 Confirm that the special instructions were appropriately observed.

12.3 Check any calculations.

12.4 Check the reporting limits. The standard reporting limit must be multiplied by any dilution factor or raised to any blank contamination value.

12.5 Verify that the results were correctly anomalized.

13. References

13.1 Method Source

This method is based on EPA Test Method 300.0 (Method A) entitled, "The Determination of Inorganic Anions in Water by Ion Chromatography," December 1989.

13.2 Related Documents

13.2.1 Enseco QAPP

13.2.2 M-EQA-0002 Internal QC checks--Laboratory Performance QC

13.2.3 LM-RMA-1074 D.I. Leach

13.3 Deviations from Source Method and Rationale

13.3.1 The columns specified in the referenced method have been substituted. This change was motivated by advances in the technology which occurred after publication of the method.

13.3.2 Although the source method does not recommend fluoride analysis by IC it will be used occasionally by an experienced chromatographer knowledgable in the particular problems.

13.3.3 Nitrite has been deleted from the list of target anions because it elutes so close to chloride.

13.3.4 Enseco's Quality Assurance Plan will be followed instead of the quality program outlined in Method 300.0.

13.4 SOP Updates (Revision 1.0 to Revision 2.0)

13.4.1 Format updated.

13.4.2 Range was extended to 500 mg/L for chloride.

13.4.3 The SOP has been modified to reflect the revised Method 300.0 (Method A), December 1989.

ION CHROMATOGRAPHY

Access File: 112990A.FRN Analyst: Steve P Date: 11-3-

SBP

UNITS: mg/l Checked & Approved:

Sample Name	DF	Chloride (0.2 RPD)	Bromide (1.0 RPD)	Sulfate (1.0 RPD)		
ICB	1	ND(3.0)	ND(0.20)	ND(5.0)		
DCS 1A	1	99.0(99)	19.3(97)	203(102)		
DCS 2A	1	98.8(99)	19.1(96)	201(101)		
12171-07 X50	50	4860	19.1	4810		
12171-07 X10	10	OverRange	224	ND(50)		
BLANK	1	ND(3.0)	ND(0.20)	5.4		
12171-08 X25	25	3600	10.7	3110		
12171-08 X5	5	31.2	9.8	OverRange		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
12171-09 X50	50	3540	11.1	2960		
12171-09 X10	10	OverRange	11.4	3240		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
12171-10 X50	50	4100	11.5	4080		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCB	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCV	1	102(102)	9.5(95)	98.9(99)		
12171-10 X10	10	OverRange	11.5	4430		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
12470-01 X2	2	43.7	0.80	ND(10)		
SBP 12470-01	1	45.6	0.80	ND(5.0)		
12470-03 X5	5	73.1	ND(1.0)	1410		
12470-03	1	79.2	0.47	ND(5.0)		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
12470-05 X10	10	138	ND(2.0)	2740		
12470-05	1	152	1.3	ND(5.0)		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCB	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCV	1	100(100)	9.2(92)	97.1(97)		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
ICB	1	ND(3.0)	ND(0.20)	ND(5.0)		
DCS 1B	1	105(105)	20.0(100)	212(106)		
DCS 2B	1	102(102)	19.6(98)	206(103)		
12470-07 X5	5	65.1	ND(1.0)	ND(25)		
12470-07	1	66.8	0.33	ND(5.0)		
12470-08 X5	5	65.6	ND(1.0)	ND(25)		
12470-08	1	67.9	0.33	ND(5.0)		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
12429-01 X2	2	11.3	ND(0.40)	571		
12429-01	1	10.9	ND(0.20)	OverRange		
12432-01	1	ND(3.0)	ND(0.20)	33.4		
12432-02	1	ND(3.0)	ND(0.20)	33.7		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCB	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCV	1	105(105)	9.6(96)	100(100)		
12432-04 X5	5	ND(15)	ND(1.0)	2270		
12432-04	1	8.1	ND(0.20)	ND(5.0)		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCB	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCV	1	104(104)	9.5(95)	101(101)		

APPENDIX B-6

NITRITE NITROGEN (ENSECO)

Subject or Title: NITRITE AS N

SOP No.: LM-RMA-1041

Revision No.: 2.0

Effective Date: May 16, 1991

Supersedes: Revision No. 1

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1. Scope and Application

1.1 Analytes

This method is applicable to the determination of nitrite.

1.2 Detection Limit

The detection limit is 0.01 mg/L as N.

1.3 Applicable Matrices

This method is applicable to drinking, surface and saline waters, domestic and industrial wastes. Nitrite on soils requires a D.I. leach prep. This prep is outlined in SOP: LM-RMA-1074.

Prepared by:	Date:
Tony J. Stowell	May 24, 1991
Management Approval:	Date:
David B Roberts Jr	5-24-91
QA Officer Approval:	Date:
Bill Glazer	05/27/91

1.4 Dynamic Range

The range extends to 0.2 mg/L as N. Higher concentrations may be analyzed by dilution of the sample.

1.5 Approximate analytical time is 20 minutes per sample. A number of samples may be analyzed concurrently.

2. Method Summary

Nitrite in acidic solution forms a diazonium compound with sulfanilamide. The diazonium compound then reacts with N-(1-naphthyl)-ethylenediamine dihydrochloride to produce a reddish purple dye which is measured in a spectrophotometer at 540 nm.

3. Comments

3.1 Interferences

3.1.1 Strong oxidizing and reducing agents will affect the nitrite concentration.

3.1.2 High alkalinity (> 600 mg/L) will give cause low results due to a shift in the pH.

3.2 Helpful Hints

None

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outline in the Enseco/RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see you supervisor or safety officer.

4.2 Wear gloves and apron when handling concentrated acids, bases and solvents. Transport only in approved carriers. Avoid breathing fumes and vapors; handle in a fume hood. Neutralize and clean up any spills immediately. In case of skin contact, flush affected area with water for at least 15 minutes. Notify your supervisor or safety officer of any spills or exposures.

5. Samples Collection, Preservation, Containers and Holding Times

   5.1 Samples are to be collected in plastic bottles and refrigerated to 4 C.

   5.2 The holding time for nitrite is 48 hours.

6. Apparatus

   6.1 Spectrophotometer for use at 540 nm.

   6.2 Miscellaneous laboratory apparatus and glassware.

7. Reagents and Standards

   7.1 Buffer/Color Reagent

   Add 105 mL concentrated hydrochloric acid to 250 mL deionized water and mix. Add 5.0 g sulfanilamide and 0.5 g N-(1-naphthyl) ethylenediamine dihydrochloride. Stir until dissolved. Dilute to 500 mL with deionized water. Store in a dark bottle in a refrigerator. This solution is stable for one month.

   7.2 Calibration Stock Standard, 100 mg/L as N

   Dry anhydrous sodium nitrite in a dessicator for 24 hours. Dissolve 0.4926 g in deionized water and dilute to 1000 mL. Add 2 mL chloroform to preserve.

   7.3 Intermediate Standard, 1.0 mg/L as N

   Dilute 1.0 mL of the 100 mg/L stock to 100 mL with deionized water. Prepare fresh daily.

   7.4 Calibration Standards

   Dilute the intermediate standard with deionized water as follows:

Vol. Intermediate Std.	Final Volume	Concentration
1.0 mL	100 mL	0.01 mg/l as N
2.0 mL	100 mL	0.02 mg/l as N
4.0 mL	100 mL	0.04 mg/l as N
8.0 mL	100 mL	0.08 mg/l as N
20.0 mL	100 mL	0.20 mg/l as N

   Prepare fresh daily.

7.5 Spiking standard, 10.0 mg/L as N

Dilute 10.0 mL of the 100 mg/L stock to 100 mL with deionized water. **Prepare only if matrix spike is required.**

7.6 DCS Stock, 100 mg/L as N

Prepare as in 7.2 above, but independently, to provide a reference material.

7.7 Intermediate DCS, 1.0 mg/L as N

Dilute 1.0 mL of 100 mg/L DCS Stock to 100 mL with deionized water. Prepare fresh daily.

7.8 Working DCS, 0.10 mg/L

Dilute 10.0 mLs of Intermediate DCS to 100 mL with deionized water. Prepare fresh daily.

7.9 Hydrochloric Acid, 25%

Carefully add 100 mL concentrated hydrochloric acid to 300 mL deionized water. Mix well.

8. Procedure

8.1 If the pH of the sample is greater than 10 or if high alkalinity is suspected, add 25% hydrochloric acid to adjust the pH to about 6.

8.2 If necessary, the sample may be filtered to remove particulates.

8.3 Place 10 mL aliquots of sample into two test tubes. One aliquot will be used for analysis and the other for a color blank. (Color blanks are not required for standards and DCS's).

8.4 Add 0.4 mL buffer/color reagent and mix. For the color blank add 0.4 mL 1 N HCL instead of the buffer/color reagent.

8.5 After 15 minutes, measure the absorbance at 540 nm. Use the reagent blank to zero the spectrophotometer.

8.6 Dilute and reanalyze any samples which have higher absorbances than the 0.20 mg/L standard. Reanalyze color blanks at the same dilution as the samples.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A blank (deionized water) is required with every batch of 20 or less samples.

9.1.2 Two DCS samples are required with each batch of 20 or less samples.

9.1.3 A blank and standard check are required after every 10 or less samples.

9.1.4 Duplicates may be required for project specific QC.

9.1.5 Spikes may be required for project specific QC. Add 0.1 mL of the 10.0 mg/L spiking standard to 10 mL sample. The spiking concentration is 0.10 mg/L.

9.2 Acceptance Criteria

9.2.1 If the prep blank reads greater than 2 times the detection limit, consult your supervisor.

9.2.2 DCS recovery must be 90 - 110%.

9.2.3 The RPD for DCS samples must be less than 10%.

9.2.4 Standard checks must be within 10% of the expected value.

9.2.5 There are no acceptance criteria for project specific QC.

9.2.6 The calibration curve must have a correlation coefficient of 0.995 or greater.

9.3 Corrective Action Required

If problems are encountered, check the dilutions made when preparing the standards and DCS. Also check the pH of the samples after color development; it should be 1.5 - 2.0. If problems persist, consult your supervisor.

10. Calculations

10.1 Enter the standard data into linear least squares program to generate a calibration curve.

10.2 Subtract the color blank absorbances from the sample absorbances. Calculate the sample concentration from the net absorbance and multiply by any dilution factors.

10.3 If the prep blank is more than the detection limit, the detection limit must be raised to the blank value.

10.4 If dilutions are necessary the reporting limit must be raised by the dilution factor.

11. Reporting Requirements

11.1 Reporting units are mg/L as N.

11.2 Samples less than the detection limit are reported as N.D.

11.3 Significant Figures:

0.01 - 9.99: 2 significant figures
10 + : 3 significant figures

11.4 LIMS Data Entry

The usual standards for data entry apply.

12. References

12.1 Method source: EPA Method 354.1

12.2 Additional information: Standard Methods #419 (Sixteenth Edition)

12.3 Deviations from source method and rationale

12.3.1 The sample volume was reduced by a factor of 5. The reagent volumes were reduced accordingly.

12.3.2 The source method does not include a provision for the analysis of soils.

12.3.3 Provision for the analysis of sample color blanks has been included.

12.4 Updates to SOP

Revision 1.0 was updated to Revision 2.0 to include provision for nitrite determination in soil, changes in standard curve preparation, provision for the analysis of sample color blanks, preparation and use of a spiking standard, changes in QA/QC and clerical changes.

APPENDIX B-7

TOTAL ORGANIC CARBON - WATER (ENSECO)

STANDARD
OPERATING
PROCEDURE

Subject or Title: Total Organic Carbon (TOC) (0.I. 700)		Page 1 of 9
SOP No.: LM-RMA-1066	Revision No.: 1.0	Effective Date: August 1, 1988
Supersedes: Original		

1. Scope and Application

1.1 Analytes

This method covers the determination of various forms of carbon using the O.I. Corporation Model 700 TOC Analyzer. The instrument is capable of measuring various forms of carbon. The specific forms measured depend on sampling technique and subsequent handling in the laboratory. Unless special requirements are specified before the analysis is started, samples will be homogenized, the solids allowed to settle, and the liquid decanted for analysis.

The definition of organic carbon depends on the preliminary treatment of the samples. Unless otherwise specified, the result reported will NOT include purgeable organic carbon or particulate carbon.

Purgeable organic carbon and inorganic carbon may also be determined but are not reported unless specifically requested.

1.2 The detection limit is 0.1 mg/L.

1.3 Applicable Matrices

This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.

Prepared by: Lindsay Breyer	Date: 7/18/88
Management Approval: [signature]	Date: 11/21/88
QA Officer Approval: [signature]	Date: 11/22/88

1.4 Dynamic Range

Instrument response is linear to 50 mg/L. Higher concentrations may be analyzed by dilution of the samples.

1.5 Approximate analytical time is 10 minutes per sample.

2.0 Summary of Method

2.1 The sample is acidified and sparged to convert inorganic carbon (carbonates and bicarbonates) to carbon dioxide, which is trapped on a molecular seive. Purgeable organic carbon is also removed by sparging and trapped on a purgeables trap. The molecular seive is thermally desorbed and the carbon dioxide is detected by a nondispersive IR detector. The purgeables trap is then desorbed and the compounds pyrolyzed in an oxygen atmosphere to carbon dioxide, which is measured by the same detector. The sample is then digested with sodium persulfate at 100°C. Organic carbon is converted to carbon dioxide, which is trapped on the molecular seive trap. It is then thermally desorbed and the carbon dioxide measured by the nondispersive IR detector.

3. Comments

3.1 Interferences

3.1.1 Particulates in the sample have been found to cause erratic results even when the samples are homogenized. They will also cause carry-over to subsequent samples.

3.1.2 As noted above, the manner of preliminary treatment of the sample defines the types of carbon which are measured. Specific sample requirements must be defined when the samples are logged in for analysis.

3.1.3 Purgeable organic carbon will be lost if the samples are not collected in VOA vials with the headspace eliminated.

3.2 Helpful Hints

This SOP was written specifically for the OI 700 TOC Analyzer.

4. Safety Issues

Follow normal laboratory precautions.

5. Sample Collection and Preservation

5.1 Samples are collected in glass bottles and preserved by adding sulfuric acid to pH less than 2.

5.2 Samples requiring purgeable organic carbon must be collected in VOA vials with headspace eliminated.

5.3 The holding time for TOC is 28 days.

6. Apparatus

6.1 OI 700 TOC Analyzer with POC option, autosampler, etc.

6.2 Disposable glass autosampler tubes.

6.3 Volumetric pipettes, volumetric flasks, beakers, etc.

7. Reagents and Standards

7.1 Phosphoric Acid, 5% v/v

Carefully add 50 mL phosphoric acid to about 600 mL deionized water, mix, and dilute to 1000 mL.

7.2 Sodium Persulfate, 10% w/v

Dissolve 100 g sodium persulfate in deionized water and dilute to 1000 mL.

7.3 TOC Stock Standard, 1000 mg/L

Dissolve 2.128 g potassium hydrogen phthalate in about 600 mL deionized water, add 2 mL concentrated sulfuric acid, and dilute to 1000 mL.

7.4 TOC Working Standards

Dilute the stock standard with deionized water as follows:

Aliquot	Final Volume	TOC Concentration
1.0 mL	200 mL	5.0 mg/L
5.0 mL	200 mL	25.0 mg/L
10.0 mL	200 mL	50.0 mg/L

7.5 LCS Solution

Weigh an empty 250 mL glass bottle covered with a VOA cap. Fill with a diluted ERA demand sample and recap, eliminating all headspace. Inject 10.0 uL chloroform through the septum and reweigh. Shake vigorously until the chloroform has dissolved. The TOC concentration will be the true value of the ERA sample. The POC concentration is calculated from:

POC = 1491 / (WT2 - WT1)

POC = POC concentration, mg/L
WT2 = Weight of full bottle + cap, g
WT1 = Weight of empty bottle + cap, g

If POC is not required, it is unnecessary to spike the ERA sample with chloroform.

8. Procedure

8.1 Instrument Set-Up

8.1.1 The instrument should be left on with gases flowing in order to reduce the need for detector maintenance. If the instrument has been turned off for any reason, turn it back on and allow it to warm up until the IR output stabilizes (may be several hours).

8.1.2 Check gas pressures. Nitrogen should be at 30 psi and oxygen at 10 psi.

8.1.3 Check gas flows. Purge gas flow should be 6.0 and detector gas flow 4.5 on the rotameters. If these flows cannot be achieved, inform your supervisor.

8.1.4 Fill the deionized water reservoir and set the flow rate to keep the wash station full.

8.1.5 Fill the acid reservoir with 5% phosphoric acid and the oxidant reservoir with 10% sodium persulfate. Check to be sure that the reagent bottles are being sparged.

8.1.6 Open the keypad drawer and press the PRIME ACID button 10 to 20 times to prime the acid pump.

8.1.7 Press the PRIME OXIDANT button 10 to 20 times to prime the oxidant pump.

8.2 Reagent Blanks

8.2.1 Select Display Mode NORMAL PPM C and Analysis Mode POC/IC/TOC.

8.2.2 Press SET SYSTEM CONFIG 1. Set the instrument parameters as follows:

ACID INJECTION PUMP: Enabled
OXID INJECTION PUMP: Enabled
SAMPLE PUMP: Disabled
SAMPLE LOOP VALVE: Disabled
AUTO-RUN ANALYSIS: Enabled
AUTO-PRINT: Enabled
AUTO-SAMPLER: Disabled

Press CLEAR ENTRY when the proper system configuration has been established.

8.2.3 Press RUN/STOP. The instrument will automatically run blanks (acid and oxidant) and cleanse itself.

8.2.4 When the blanks show consistent results, press RUN/STOP and CLEAR.

8.2.5 If the instrument has already been calibrated, the results printed should be less than the detection limit. If not, as might happen with new reagents, it may be necessary to recalibrate. Compute the average millivolt reading for the last three blanks. Compute the average for all three forms of carbon.

8.3 Calibration

8.3.1 Check calibration by analyzing the 25 mg/l standard. If it is within 10%, recalibration is not necessary.

8.3.2 Press SET SYSTEM CONFIG and set the following parameters:

ACID INJECTION PUMP: Enabled
OXID INJECTION PUMP: Enabled
SAMPLE PUMP: Enabled*
SAMPLE LOOP VALVE: Enabled*
AUTO-RUN ANALYSIS: Enabled
AUTO-PRINT: Enabled
AUTO-SAMPLER: Disabled

*Changed from reagent blank configuration.

Press CLEAR ENTRY when finished.

8.3.3 Disconnect the sample line from the sampler and insert into the 25 mg/L calibration standard.

8.3.4 Press RUN/STOP. The instrument will analyze the standard repeatedly. Press RUN/STOP and CLEAR after at least 3 runs have been completed.

8.3.5 Compute the average mV response for organic carbon.

8.3.6 Press SELECT DISPLAY MODE twice to get to CALIBRATION CONSTANTS.

8.3.7 Press SELECT NEXT DISPLAY. Choose option 2, organic carbon calibration.

8.3.8 Enter the average blank mV for each form of carbon. If a value is already entered, add it to your average before entry. Also add it to the average mV for the standard.

8.3.9 ENTER STANDARD MASS: Multiply the standard concentration (mg/L) by the loop volume (mL) and enter the result. With a 1.010 mL loop and 25 mg/L standard, the standard mass is 25.25.

8.3.10 Press SELECT NEXT DISPLAY. Enter the mV average for the standard. The scaling factor will be displayed.

8.3.11 Press SELECT NEXT DISPLAY. Then press SELECT DISPLAY MODE to return to NORMAL PPM C. Reattach the sample line to the sampler.

8.4 Analysis

8.4.1 Mix samples well, fill sampler tubes, and load the autosampler. Deionized water should be first, followed by the standards and the LCS samples. Record the order in which the samples will be analyzed (the sampler moves counterclockwise). If POC is required, use the racks supplied for VOA vials and load them directly onto the sampler. Do not open VOA vials.

8.4.2 Press SET SYSTEM CONFIG 1. Set the following parameters:

ACID INJECTION PUMP: Enabled
OXID INJECTION PUMP: Enabled
SAMPLE PUMP: Enabled
SAMPLE LOOP VALVE: Enabled
AUTO-RUN ANALYSIS: Enabled
AUTO-PRINT: Enabled
AUTO-SAMPLER: Enabled
READY/STANDBY STATUS OVERRIDE: Disabled
SAMPLE ID NUMBER: 01
SAMPLE STOP NUMBER: **
NUMBER OF REPS PER SAMPLE: 1

**Position of last sample on the autosampler

8.4.3 Advance the sampler so that the first sample is under the sample line.

8.4.4 Press RUN/START. The run will proceed automatically and stop when the last sample has been analyzed.

8.4.5 Dilute and reanalyze any samples more than 50 mg/L. Turn off the deionized water reservoir when done.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 Analyze one sample of deionized water with every batch of 20 or less samples.

9.1.2 Two LCS samples are required with every batch of 20 or less samples.

9.1.3 Linearity check standards (5, 25, 50 mg/L) are required at the start of every run. Calibration checks (25 mg/L) are required after every 10 or less samples and at the end of the run.

9.1.4 Duplicates may be required as project specific QC.

9.1.5 Spikes may be required as project specific QC. Dilute 1.0 mL stock standard to 100 mL with sample. The spiking concentration is 10 mg/L TOC. See your supervisor if spikes are required for POC.

9.2 Acceptance Criteria

9.2.1 Control limits are:

	Accuracy	Precision
TOC	91 - 109%	20%
POC	70 - 130%	20%

9.2.2 Standard checks must be within 10% of the expected value.

9.2.3 There are no acceptance criteria for project specific QC.

9.3 Corrective Action Required

Verify that the instrument is properly calibrated. Check autosampler tubes for contamination. Be sure that the sample pump and reagent pumps are operating correctly. Recalibrate and reanalyze samples as necessary.

Check for nonlinearity and also the IR output. If it is too high, the cell may need cleaning. Consult your supervisor.

Check gas flows with and without heating of the primary trap and POC trap. If the flow drops below 4.5 (primary trap) or 4.0 (POC trap) when heat is applied, the traps need maintenance. See your supervisor.

10. Calculations

Record the results printed out for each sample. Multiply by any dilution made to get the final result.

11. Reporting

11.1 Reporting units are mg/L

11.2 Reporting Limits

Samples less than 0.1 mg/L are reported as ND.

11.3 Significant Figures

0.1 to 0.9 mg/L	1 significant figure
> 1.0 mg/L	2 significant figures

11.4 LDMS Data Entry

The usual standards for data entry apply.

12. References

12.1 Method Source: EPA Method 415.1

12.2 Deviations from Source Method and Rationale

12.1 The source method is vague as to specific procedures to follow and references the instrument manual.

12.2 Solids are excluded from analysis unless specifically requested on the samples. Solids preclude the use of an autosampler and cause carryover and erratic response.

APPENDIX B-8

TOTAL ORGANIC CARBON - SOIL (ETC)

ETC

SOP # ED522902
EFFECTIVE DATE: 10/19/90
SUPERCEDES DATE: [illegible]
PAGE 1 of 10
APPROVED [signature] 10/19/90
GB [signature] 10/19/90

Method for the Determination of Total Carbon and Total Organic Carbon in Sediment, Sludge and Soil Samples

Re: USEPA 415.1, SW846 9060, EPA AD/A103 788, Section 3-73

1.0 METHOD SUMMARY

1.1 Total Organic Carbon is measured using the Dohrman DC-80 carbon analyzer coupled with the PRG-1 furnace module.

1.2 A known weight of sample is combusted in an oxygen atmosphere at 800 C, in the presence of a copper catalyst. Carbonaceous materials are converted to carbon dioxide. The C02 formed is quantified using an NDIR detector.

1.3 Intact samples are taken for analysis to minimize loss of volatile components. Results are reported on a "wet weight basis".

1.4 Data is reported in mg/kg. Method detection limit is 100 mg/kg.

1.5 If the results are to be reported on a "dry weight basis", a % solids determination is required and is applied to data calculations.

2.0 APPARATUS

2.1 Dohrman DC-80 Carbon Analyzer

2.2 Dohrman PRG-1 Furnace Module

2.3 Dohrman Sludge/Sediment Sampler Accessory

2.4 50 ul syringe (unimetrics)

2.5 Chann Electro Balance

2.6 Quartz Wool and Sample Boats

2.7 Stainless Steel Forcepts/Spatula

2.8 Dohrmann Oxidation Promotor (PN 511-883)

3.0 REAGENTS

3.1 Carbon stock solution (10,000 mg/l) - dissolve 21.2800 grams of primary standard grade potassium hydrogen phthalate (anhydrous, dried @ 105°C for 2 hours, cooled in a dessicator) into 800 ml of DIHOH (1 ml = 10.0 mg). Add 2 ml 1+1 H2SO4, then dilute to 1 litre with DIHOH. Stock solution is stable for 6 months.

3.2 4000 mg/l standard - dilute 40 ml of the "stock" solution. Add 2 ml of 1+1 H2SO4, then dilute to 100 ml.

3.3 2000 mg/l standard - dilute 20 ml of the "stock" solution. Add 2 ml of 1+1 H2SO4, then dilute to 100 ml.

3.4 1000 mg/l standard - dilute 10 ml of the stock solution. Add 2 ml 1+1 H_2SO_4, then dilute to 100 ml

3.5 100 mg/l standard - dilute 1.0 ml of the stock solution. Add 2 ml of 1+1 H_2SO_4, then dilute to 100 ml.

3.6 Standards for soil analysis must be prepared fresh weekly.

3.7 H_2SO_4 solution (1+1) (V/V) - Add with constant mixing in an ice bath. 100 ml of concentrated ACS AR grade sulfuric acid to 100 ml of deionized water

3.8 Potassium presulfate - phosphoric acid reagent (2%). Dissolve 20.0g of reagent grade potassium persulfate ($K_2S_2O_8$), add 2.0 ml of concentrated (85%) phosphoric acid (H_2PO_4). Mix thoroughly and dilute to 1 litre with DIHOH. Prepare fresh monthly.

3.9 Hydrochloric acid solution (1+9) - add 10 ml of conc. ACS (AR) HCL to 90 ml of DIHOH. Mix thoroughly.

4.0 INTERFERENCES

4.1 Inorganic carbon (carbonates and bi-carbonates) represent positive interferences. Inorganic carbon is removed by treating a known weight of sample with several drops of 1+9 HCL solution. Additional acid is added dropwise until inorganic carbonate removal is complete.

4.2 Non-homogenous matrices may yield poor reproducibility Homogenize the intact sample by grinding to obtain a representative aliquot.

5.0 ELECTRO BALANCE CALIBRATION

5.1 Check and document the calibration of the electro balance as per the instrument manual.

5.2 Identical weighing pans should be in place on the "B" and "tare" positionns of the balance.

5.3 Set the range at the "B" 1200 mg position. The readout should be 0.00 mg. Close the balance. Allow to stabilize, then depress the tare. The readout should be stable at 0.00 mg.

5.4 Place a 1000 mg Class "S" weight on to Pan "B". Record the result. Depress the "CAL" button to calculate to 1000 mg. Record the result. Repeat the procedure and confirm calibration.

5.5 Then weigh and record the values obtained from the following Class "S" weights:

100.0 mg
10.0 mg
5.0 mg

5.6 All values obtained should be within 1% of the expected value. If not, recalibrate the balance as per Section 5.0.

6.0 CARBON ANALYZER - PRELIMINARY SET UP FOR SOIL ANALYSIS

6.1 Check all instrument components as per the Dohrman DC-81 instrument manual. Furnace inlet tube to bulkhead #5, furnace outlet tube is connected to bulkhead #4. Also see attached assembly drawing.

6.2 Position the boat carrage under the sample inlet block. Place the sample boat into the carrier using forcepts. Place a piece of quartz wool into the boat and close the inlet block.

6.3 Turn on the power to the PRG-1 furnace module.

6.4 If fresh oxidation promotor (PN 511-883) is used, condition by heating for 1 hour with the furnace exit tube immersed in basic DIHCH. After 1 hour, connect the furnace exit tube to the UV module at bulkhead #4. Verify flow within the reactor.

6.5 If "pre-conditioned" oxidation promotor is used, connect the furnace exit tube to the UV reactor module at bulkhead #4. Verify flow within the reactor. Allow the system to come up to full temperature and stabilize for 30 minutes.

6.6 Verify the following:

- Oxygen is flowing in the reactor
- Normal system back pressure
- UV lamp is turned off
- Reactor is filled with reagent
- Quartz wool is in the boat
- Mode is TOC
- Volume is 40 ml
- Pump is on
- Baseline is stable at 0.0100 units in "DET" mode

6.7 Set the IR mode to DET. Decalibrate the instrument by depressing the CALIB button for 3 seconds.

6.8 Adjust the baseline to 0.0100 units. It should be stable after the furnace temperature has reached 800°C ± 0.0002 units.

6.9 Decontaminate the sample boat by pushing it into the combustion zone for 2 minutes. This will remove any organic residues on the boat. Instrument baseline will return to normal when boat is clean.

7.0 CALIBRATION

7.1 Inject 40 ul of the 2000.0 mg/l carbon standard into the cooled sample boat via the liquid injection port. Slide the boat into the combustion zone.

7.2 Depress the start button. Then on completion of the cycle, record the PPM response obtained on the LED. Bring the boat out of the furnace into the inlet block and allow to cool. Inject three additional 40 ul volumes on the 2000 mg/l calibration standard. The response values should be within 5% of the mean. If not, repeat the calibration procedure.

7.3 Press the "calibrate" button and record the mean response value and the "adjusted" PPM value from the calibration sequence. The value should be 2000.0 PPM C ± 5%.

8.0 CHECK STANDARD ANALYSIS

8.1 Analyze single 40 ul injections of each of the following acidified and sparged standard solutions.

Method blank
100 mg/l carbon standard
1000 mg/l carbon standard
2000 mg/l carbon standard
4000 mg/l carbon standard

8.2 Calculate the % Recovery. Recoveries should be within 10% of the known standard value. If not, re-analyze the standard.

$$\% \text{ REC} = \frac{\text{Observed Result}}{\text{Known Result}} \times 100$$

9.0 SAMPLE ANALYSIS

9.10 Remove the quartz wool and tare the cool, clean sample boat on pan "B" (1200 mg range) of the electro balance. Remove the tared boat from the balance, then add a homogenous amount of sample to the boat. Reweigh and record the weight in mg.

9.11 If organic carbon is to be determined, remove inorganic carbonates by treating a known weight of sample with several drops of 1+9 HCL. Wait until effervescing is completed and add additional acid. Continue this process until the incremental addition of acid causes no further effervescence. Proceed to 9.3.

9.2 If total carbon is to be determined, omit the addition of HCL reagent to the sample. Report data as "Total Carbon". Proceed to 9.3.

9.3 Transfer the "loaded boat" to the sample carriage via the inlet block. Allow the system to stabilize for 30 seconds after closing the inlet block.

9.4 Push the start button and slide the boat into the furnace combustion zone. Allow the instrument to

complete the cycle. Record the PPM value obtained from the LED display.

9.5 Analyze all samples in duplicate.

9.6 Calculate the carbon concentration for the sample using the following:

$$mg/kg = \frac{40\ (\text{LED ppm})}{\text{Sample weight mg}}$$

All data is reported to three significant figures.

10.0 QUALITY CONTROL

10.1 Four calibration standards are analyzed for each analytical run. The % recoveries are calculated for each.

10.2 A 2000 PPM check standard is analyzed every 10-15 samples and at the end of each analytical run. The % recovery is calculated.

10.3 A method blank (40.0 ul of DIHOH) is analyzed once per batch. Results must be less than the MDL.

10.4 An EPA reference QC sludge sample is analyzed in duplicate for each batch and the % recovery is calculated. The value obtained should be within the EPA limits.

10.5 All samples are run in duplicate at minimum.

ETC

Total Organic Carbons in Soils

QW- Batch: _____ Method Ref: EPA 415.1, SW-846 9060 Page _____ of _____

QC-Batch: _____ EPA AD/A103 788 (Combustion /IR) Date: _____

Verified: _____ MDL: 100 mg/kg Time: _____

Instrument: _____ Matrix: Soil/Solids Analyst: _____

Job #	Weight mg	HCL Added	Inject #	LED Value	Conc mg/kg Wet Wt.	% Solids	Conc mg/kg Dry Wt	Comments

BALANCE CALIBRATION DATA:

1000 mg Initial = _____ 10 mg CLASS "S" = _____

1000 mg CALIB = _____ 5 mg CLASS "S" = _____

100 mg CLASS "S" = _____

$$mg/kg = \frac{40 \times LED}{mg} \div \frac{\%\ Solids}{100}$$

CV-12 5/90 Rev. 0

ATTACHMENT #ED522902

APPENDIX B-9

LOW LEVEL METALS BY ICP (ENSECO)

Ensec

STANDARD
OPERATING
PROCEDURE

Subject or Title: CLP Inductively Coupled Plasma Analysis (ICP)		Page 1 of 15
SOP No.: LE-RMA-2000	Revision No.: Original	Effective Date: November 22, 1989
Supersedes: N/A		

1.0 Scope and Application

1.1 This method is applicable for the determination of metals required under the CLP SOW (Al, Sb, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Ag, Na, V, and Zn) in water and soil samples. This method may also be used for the determination of Non - CLP metals (B, Mo, Si, Sr, and Ti).

1.2 Detection Limits

1.2.1 Contract Required Detection Limits (CRDL)

Detection limits are those listed in current SOW.

1.2.2 Instrument Detection Limits (IDL)

1.2.2.1 Detection limits are calculated quarterly according to current SOW protocol.

1.2.2.2 A table of current quarterly IDL's are distributed to the analytical staff, data review, and report generation upon completion of quarterly IDL's.

1.3 Analytical Range

1.3.1 Linear ranges are checked quarterly according to the current SOW.

Prepared by: Scott Hoatson	Date: October 11, 1989
Management Approval: Kathy K. Ohonyak	Date: 11/22/89
QA Officer Approval: Lena M Ligon	Date: 11/22/89

1.3.2 Aluminum : 0 - 600 mg/L Al in solution.

1.3.3 Iron : 0 - 500 mg/L Fe in solution.

1.3.4 Cations (Ca, Mg, K, Na) : 0 - 1000 mg/L of analyte in solution.

1.3.5 All other metals : 0 - 100 mg/L of analyte in solution.

1.4 The approximate analytical time of one sample is 3 min.

2.0 Summary of Method

2.1 Aqueous samples or digests are nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through a quartz torch and injects it into the plasma. The plasma of ionized argon is sustained at temperatures as high as 10,000°K by a Radio Frequency (RF) generation source.

2.2 Once the aerosol reaches the plasma, the sample is desolvated, decomposed, the elements are excited and emit their characteristic radiation.

2.3 The emitted light energy is then directed through the entrance slit, defracted by the grating, refocused on the exit slits and projected on photomultiplier tubes (PMT) specific for each element. The PMT's convert the light energy to an electrical signal that is digitized and processed by the instrument's computer.

3.0 Comments

3.1 Interferences

3.1.1 Background emission may be caused by, among other things, continuum emission and viscosity differences. Background correction points are required for each element (see ICAP 61 operator's manual).

3.1.2 Interelement 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. Interelement corrections (IEC's) must be applied to the analyte to remove the effects of these unwanted emissions. To calculate an IEC, standardize the ICP, set analysis mode to Standardized Intensity Ratio and analyze the "interfering element" at its upper linear range. Divide the concentration of the analyte by the concentration of the "interfering element". That number is the value entered in the IEC table for the analyte.

3.2 Helpful Hints

3.2.1 Use the correct digest for analysis, it is marked "HCl/HNO_3 ICP"

3.2.2 Follow the prepared ICP RAW DATA Checklist.

3.2.3 When correcting a mistake, draw ONE line through the mistake, make CORRECT entry, and INITIAL the correction.

3.2.4 Use correct ICP method, EPA2 for normal RAS analysis, and EPA1 when analytes other than RAS elements are to be analyzed.

3.2.5 Have a copy of the prep sheet handy during analysis. The prep sheet has blank and duplicate sample information as well as matrix specific QC information and other special instructions on it. Samples can be marked off as they are analyzed so 10 samples are not exceeded between calibration checks.

3.2.6 Verify that the "SB" and "FAT" lights are lit on the front of the spectrum shifter controller. If there was a power interruption, they won't be lit. To reset, see section 9.5.

4.0 Safety Issues

4.1 Many of the metals determined by this method are extremely toxic and/or carcinogenic. Care should be exercised when preparing standards from concentrated stock solutions. Wear gloves, safety glasses, and a lab coat.

4.2 All of the samples may contain unknown contaminants at unknown levels other than the analytes being tested. Appropriate care should be taken when handling them.

4.3 The plasma emits strong UV light and is very harmful to vision. **AVOID LOOKING DIRECTLY AT THE PLASMA.**

5.0 Apparatus

5.1 Thermo Jarrell Ash ICAP 61 Simultaneous Plasma Emission Spectrometer. It consists of a 0.75 meter Polychromator that accepts up to 63 channels, an R.F. generator, an inductively coupled argon plasma (ICAP) excitation source and an IBM compatible Data Acquisition System.

5.2 Gilson peristaltic pump.

5.3 Eppendorf 10 - 100 uL and 100 - 1000 uL adjustable pipettors.

5.4 Oxford 1 - 5 mL and 5 -10 mL adjustable pipettors.

6.0 Reagents, Standards, and Supplies

6.1 EPA ICP CCV solution prepared by a standards prep analyst at RMAL.

6.2 EPA ICP CRI stock solution prepared by a standards prep analyst at RMAL.

6.3 ICV concentrate solutions are prepared by EPA Las Vegas/ UNLV. Working solutions are prepared daily according to the dilution instructions accompanying the concentrates.

6.4 Interference Check solutions A & B are prepared by EPA Las Vegas.

6.5 Calibration standards prepared monthly by Environmental Resource Associates.

6.6 Approximately 5 ppm Cd profiling solution prepared at RMAL.

6.7 ICAP check standards prepared by EPA - Cincinnati (ICAP-19 and ICAP-7.

6.8 1000 ppm Yttrium stock solution.

6.9 Five dram snap cap vials.

6.10 Disposable 0.45 um syringe filters and syringes (Gelman or equivalent).

7.0 Procedure

7.1 Initiating the Plasma

7.1.1 Turn on the coolant water, argon, and exhaust fan.

7.1.2 Confirm that the argon supply is sufficient.

7.1.3 Turn on the mass flow controller.

7.1.4 Open the coolant and plasma toggle switches.

7.1.5 Set the coolant flow to 1.55 using the mass flow controller.

7.1.6 Turn on the peristaltic pump.

7.1.7 Aspirate DI rinse water for approx. 2 min. to humidify the spray chamber.

7.1.8 Using the sample flow knob on the front of the ICP, turn off the sample flow for 1 to 2 minutes.

7.1.9 Confirm: A. automatic power control set to automatic
B. forward power is on

7.1.10 Press red RF ON button.

7.1.11 Slowly turn the FORWARD POWER control knob clockwise until the forward power meter reads 0.5 kilowatts.

7.1.12 Press ignitor button and slowly turn FP knob fully clockwise.

7.1.13 Slowly turn up sample flow volume and close plasma toggle switch.

7.1.14 Using the mass flow controller, set the sample flow. Aspirate 1000 ppm yttrium and adjust sample flow until pink area extends 8 mm from the top of the coil.

7.1.15 Allow the ICP to warm up for a minimum of 30 minutes before profiling.

7.2 Using Jarrell Ash Software

7.2.1 Turn computer on.

7.2.2 The computer will automatically run the AUTOEXEC.BAT file, which places user in the MAIN MENU.

7.2.3 From the MAIN MENU, choose the OPERATION option (by pressing the ENTER key).

7.2.4 From the OPERATION menu choose the ANALYSIS option (also pressing the **ENTER** key).

7.2.5 The computer will then prompt the user for which method to use: Press the ENTER key to default to the current method shown. F6 will call up a list of methods currently on the hard drive.

7.2.6 In the ANALYSIS mode, the user has a choice of functions to execute (at the bottom right of the screen).

7.2.7 To Standardize: press the F3 key.

a. Make sure to standardize in order of standards shown.
b. Press F1 to start analyzing the standard.
c. Press F4 to print exposures just run.
d. Press F9 to keep the exposures and go on to the next standard.
e. Repeat until all standards are done.
f. The computer will display the statistical data for the standardization. Press F2 to print this data.
g. When the statistical data is printed, press F9 to save the data and return to the ANALYSIS mode.

7.2.8 To Analyze Samples: press the F1 key.

a. Enter the name of the sample and press ENTER.
b. Enter a comment if necessary and press ENTER.
c. Enter the user ID and press ENTER.

d. Enter the Correction factor(Dilution factor) and press ENTER.
e. Any of the last four steps can be eliminated if the default values are already set as desired.
f. Press F1 to initiate exposures.
g. There are two ways to SAVE and PRINT the analysis data.

   1. If this is the first time data is saved, press ALT F4 to store the data to the hard drive. Enter the name of the file and press ENTER. The name of the file is determined by the date and the shift the analysis takes place during. ex: DAY shift analysis on 6/23/88 file name is D0623, NIGHT shift is N0623. After the data is stored press ALT F2 to send the data to the printer.

   2. If data is saved to a file previously named, press CTRL F3 to execute the Macro. Enter the name of the Macro and press ENTER. Just press ENTER if the desired Macro is the default value. The Macro named " SAVE" will store and print the analysis data and return the user to the analysis menu ready to enter the name of the next sample.

h. To exit the software press CTRL F9. This returns the user to the main menu then press E to exit to DOS.

7.2.9 Archiving Data

a. Make sure that you are in the MAIN MENU (press CTRL F9 if you are not sure) and press I (for IMS, Information Management System).
b. From the IMS menu press F (for FILER).
c. From the FILER menu use the ARROW keys to move the Light Bar to 12. SAMPLES ARCHIVAL and press ENTER.
d. Press F1 for ARCHIVE.
e. Use the ARROW keys to move the Light Bar to FILE NAME(S) and press F6 to list the files available.
f. Press F2 to deselect all of the file names, then enter the number that corresponds to the file to archive and press ENTER. Press F9 to keep the file.

g. Make sure there is a blank formatted disk in drive "A" and press F9 to begin archiving.
h. To FORMAT a floppy disk, **at the analysis screen** press CTRL F3 and enter "FORMATA", press ENTER and follow the prompts. This a Macro that allows the user to format disks while in the Jarrell Ash software. If using a high density floppy to store data onto, press CTRL F3 and enter "FORMATB", place the floppy in Drive B.
i. If the high density disk has been used to store other data, exit to DOS to check the filename(s) on the disk. To do this, type "B:", then type "DIR" to view the files directory. If ANADATA.ARC is in the directory it needs to be renamed. If it is not in the directory then data can be archived onto the floppy as is.
j. To rename ANADATA.ARC type"RENAME ANADATA.ARC,FILENAME.ARC" (FILENAME.ARC is a generic reference to an ICAP data file i.e. D0815.ARC). Now data can be archived to the floppy. After data has been archived, rename the file in the above manner.
k. Press CTRL F9 to return to the MAIN MENU from anywhere in the program.

7.3 Standardization

7.3.1 There are 4 solutions for instrument standardization:
a. 2.5% HNO_3, 2.5% HCl BLANK
b. 1 ppm Silver in 4% HNO_3
c. 1 ppm Heavy Metals (10 ppm Al, Fe, Se) in 2.5% HNO_3/HCl
d. 100 ppm Cations (Ca, Mg, K, Na) in 2.5% HNO_3/HCl

7.3.2 These standards are prepared monthly by ERA (Environmental Resource Associates. The source and Lot number is to be recorded on the front page of the standardization data printout.

7.3.3 The software is set up to run 2 exposures for each standard.

7.3.4 See the above section on the software for standardization procedure.

7.4 Profiling

7.4.1 Aspirate the 5 ppm Cd profiling solution.

7.4.2 From the Analysis screen on the computer, press the appropriate Function keys to initiate Manual Instrument Profile. Follow step by step instructions that appear on the computer monitor (Note: the directions given for turning the micrometer are backwards, so turn the micrometer in the opposite direction.)

7.5 Sample Analysis

7.5.1 All EPA inorganic cases in house are listed on a computer generated Sample Analysis Status Report. They are listed according to due dates.

7.5.2 The analyst refers to the assignment board in the lab supervisors office to determine which cases need to be analyzed. Then he or she checks the EPA project files kept in the inorganic open area.

7.5.3 The prep sheet, included in the file, lists all of the sample numbers, prep date and volumes, dilutions, and special instructions.

7.5.4 The prepared samples are stored on shelves A-0 to E-5 located between the AA and ICP areas.

7.5.5 See analysis procedure above in SOFTWARE section.

7.5.6 Quality control and sample analyses run during one shift comprise the analytical run.

7.5.7 The following quality control operations must be performed during each analytical run:

a. Initial Calibration Verification
b. Initial Calibration Blank
c. CRI (every 8 hours or at beginning and end of analytical run)*
d. ICSA (every 8 hours or at beginning and end of analytical run)*

e. ICSAB(every 8 hours or at beginning and end of analytical run)*
f. Preparation Blank (with each RMA project number)*
g. DCS (with each RMA project number)*
h. Duplicate Sample Analysis (with each RMA project number)*
i. Matrix Spike Analysis (with each RMA project number)*
j. 5X Serial Dilution analysis (with each RMA project number)*
k. Continuing Calibration Verification (CCV) (every 10 samples and at the end of the analysis run after the final ICSAB)*
l. Continuing Calibration Blank (CCB) (every 10 samples)*

7.5.8 At the close of each analytical run, archive the data using the procedure described above.

* For explanations and control limits See exhibit E of SOW 787 attached. It also contains actions to be taken if any of the QC requirements are not met.

7.5.9 Include photocopies of of the standardization,initial calibration checks and interference check data with each RMA project. Also include any pertinent CCV/CCB data from a previous project. Write the RMA project number and the disk number at the top of the raw data.

7.5.10 The originals of the standardization data are kept in a file in the Data Review office.

7.6 Nightly Shutdown

7.6.1 Archive data from analysis run.

7.6.2 Turn down forward power control knob until plasma goes out.

7.6.3 Turn off everything mentioned in section on "Initiating the Plasma".

8.0 Changing Torch Assembly

8.1 Removal

8.1.1 Turn plasma off.

8.1.2 Turn RF power off.

8.1.3 Allow torch to cool for 1-2 min. (a hot torch looks just like a cold torch).

8.1.4 Open torch access door.

8.1.5 Using a penny as a screwdriver,unscrew and remove the left side of cowl that holds torch alignment.

8.1.6 From access where nebulizer is housed, reach behind nebulizer and remove the black spring clamp from the base of the torch.

8.1.7 Carefully lift the torch up and out through the nebulizer access area. Take care not to bump torch on anything as it is very fragile and is likely to chip or break.

8.1.8 Detach the gas hoses from the torch, pay attention to which line attaches where for later installation.

8.1.9 Inspect torch and tip for damage or clogs.

8.2 Installation

8.2.1 Choose torch/tip assembly from supply. If original torch is still in good condition then just replace the torch tip.

8.2.2 Match torch number with tip number.

8.2.3 Insert new tip into torch. Align the line on the fritted portion at the bottom of the torch with the line on the fritted portion of the torch tip.

8.2.4 Look down on the torch assembly from the top. visually assure that the tip is centered in the torch. Rotate the tip as necessary to center the tip. Attach the rubber band around the hooks at the bases of the torch and tip.

8.2.5 Replace the torch assembly into the ICP by reversing the steps in section 8.1.

8.2.6 See section 7.1 on Initiating the Plasma.

9.0 Troubleshooting - Common Problems

9.1 If RF alarm goes off while the plasma is lit, immediately press the BLUE (RF off) BUTTON on the center panel of the ICP. This alarm indicates there is a problem with the system. Turning the power off quickly can help save the components from damage.

9.2 Blanks are reading high in certain elements, usually Cu, Fe, or Zn (normally after the interference check or a high sample), or these elements taking a long time to flush out of the system.

9.2.1 Insert sample tubing into 10% HNO_3/HCl and allow to flush for several minutes. Then flush with DI water for approximately 1 minute.

9.2.2 If the problem still exists, then the torch tip is most likely contaminated or has a particulate occlusion. Open torch access door and visually inspect the torch tip for discoloration and signs of blockage.

9.2.3 Follow the instructions in section 8.0 on changing the torch/tip assembly. Only replace the tip.

9.2.4 Relight the plasma, reprofile, and restandardize the instrument.

9.3 Sample takes a long time to travel through the tubing to the nebulizer.

9.3.1 Check for a plug in the nebulizer by releasing the clamp on the peristaltic pump and checking the sample flow through the tubing. There should be a steady sample flow without the help of the pump. If there isn't then there is probably a restriction (usually a "fuzzball" at the junction of the sample tubing and the nebulizer inlet). This kind of a clog may also cause a small "drip" at the aforementioned junction or cause the tubing to detach itself due to back pressure. Turn off the plasma before trying to remove the restriction.

9.3.2 If there is not a plug in the nebulizer, then the pump tubing may be worn. Unclamp the tubing from the peristaltic pump. Roll the center of the tubing between your fingers to check for a "Flat Side". If this is the case then replace the tubing.

9.3.3 Another possible cause to the problem, albeit an unlikely one, is not enough Argon flow through the sample line. Normally this can be readily spotted from the reading on the front of the Mass Flow Controller. If there is a leak in the gas line, then the Mass Flow Controller will read correctly but the flow to the nebulizer will be greatly reduced or nonexistent (this situation may also cause the melting of torches). To check for this situation, turn off the plasma and remove the gas line from the center of the nebulizer (leave the gas flow on) and plug the end with a finger. The gas flow reading should drop to zero. If there is no drop in the gas flow reading on the Mass Flow Controller, then there is a leak in the line somewhere. Use "Snoop" to find the leak, repair the leak and relight the plasma.

9.4 Cadmium readings are significantly negative in samples and blanks.

9.4.1 The system was not flushed well enough after Profiling and there was Cadmium carryover in calibration standard 1 (Blank). As time goes by and the system gets flushed out cadmium results will go negative.

9.4.2 Contamination in calibration sample number 1 (Blank) will cause the same symptoms. Try using a new calibration standard 1.

9.5 There are no intensty readings when profiling or standardizing.

9.5.1 The sample tube is not far enough in the standard bottle, no sample is being aspirated.

9.5.2 Is the "POWER" button on the front of the spectrum shifter controller lit ? If it is not, press the "POWER" button and the light should come on.

9.5.3 Open the back panel of the spectrum shifter controller and check the circuit breaker to see if it is tripped. Reset the circuit breaker if necessary and press the "RESET" button in the back. Close the back panel.

9.5.4 Press the BLUE button on the front of the spectrum shifter controller above the N+1 dynode. This is also a reset button. The instrument should now be ready for use.

9.6 Profile intensities change significantly from previous day.

9.6.1 Torch may be out of alignment. Realign torch, see supervisor if needed.

9.6.2 Gas flows may be incorrect. Check flows using 1000 ppm Yttrium (See 7.1.14).

9.6.3 Mirrors may be out of alignment. See supervisor prior to adjustment for the first time.

9.6.4 A new torch may have been installed, check logbook to see if entry has been made.

9.6.5 Changes in profile intensity are common and they need to be investigated. However as long as the Cu/Ti ratio remains relatively constant then the instrument parameters are still constant and the IEC's won't be affected.

10.0 References

10.1 ICAP 61 Operator's Manual - Thermo Jarrell Ash Corporation, January 1988. Copyright 1987

10.2 SOW 787/788, United States Environmental Protection Agency.

10.3 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 2nd Edition, USEPA, 1982.

SECTION I

GENERAL QA/QC PRACTICES

Standard laboratory practices for laboratory cleanliness as applied to glassware and apparatus must be adhered to. Laboratory practices with regard to reagents, solvents, and gases must also be adhered to. For additional guidelines regarding these general laboratory procedures, see Sections 4 and 5 of the Handbook for Analytical Quality Control in Water and Wastewater Laboratories EPA-600/4-79-019, USEPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, March 1979.

SECTION II

SPECIFIC QA/QC PROCEDURES

The quality assurance/quality control (QA/QC) procedures defined herein must be used by the Contractor when performing the methods specified in Exhibit D. When additional QA/QC procedures are specified in the methods in Exhibit D, the Contractor must also follow these procedures. NOTE: The cost of performing all QA/QC procedures specified in this Statement of Work is included in the price of performing the bid lot, except for duplicate, spike, and laboratory control sample analyses, which shall be considered separate sample analyses.

The purpose of this document is to provide a uniform set of procedures for the analysis of inorganic constituents of samples, documentation of methods and their performance, and verification of the sample data generated. The program will also assist laboratory personnel in recalling and defending their actions under cross examination if required to present court testimony in enforcement case litigation.

The primary function of the QA/QC program is the definition of procedures for the evaluation and documentation of sampling and analytical methodologies and the reduction and reporting of data. The objective is to provide a uniform basis for sample collection and handling, instrument and methods maintenance, performance evaluation, and analytical data gathering and reporting. Although it is impossible to address all analytical situations in one document, the approach taken here is to define minimum requirements for all major steps relevant to any inorganic analysis. In many instances where methodologies are available, specific quality control procedures are incorporated into the method documentation (Exhibit D). Ideally, samples involved in enforcement actions are analyzed only after the methods have met the minimum performance and documentation requirements described in this document.

The Contractor is required to participate in the Laboratory Audit and Intercomparison Study Program run by EPA EMSL-Las Vegas. The Contractor can expect to analyze two samples per calendar quarter during the contract period.

The Contractor must perform and report to SMO and EMSL/LV as specified in Exhibit B quarterly verification of instrument detection limits (IDL) by the method specified in Exhibit E, by type and model for each instrument used on this contract. All the IDLs must meet the CRDLs specified in Exhibit C. For ICP methods, the Contractor must also report, as specified in Exhibit B, linearity range verification, all interelement correction factors, wavelengths used, and integration times.

In this Exhibit, as well as other places within this Statement of Work, the term "analytical sample" is used in discussing the required frequency or placement of certain QA/QC measurements. The term "analytical sample" is defined in the glossary, Exhibit G. As the term is used, analytical sample includes all field samples, including Performance Evaluation samples, received from an external source, but it also includes all required QA/QC samples (matrix spikes, analytical/post-digestion

spikes, duplicates, serial dilutions, LCS, ICS, CRDL standards, preparation blanks and linear range analyses) except those directly related to instrument calibration or calibration verification (calibration standards, ICV/ICB, CCV/CCB). A "frequency of 10%" means once every 10 analytical samples. Note: Calibration verification samples (ICV/CCV) and calibration verification blanks (ICB/CCB) are not counted as analytical samples when determining 10% frequency.

In order for the QA/QC information to reflect the status of the samples analyzed, all samples and their QA/QC analysis must be analyzed under the same operating and procedural conditions.

If any QC measurement fails to meet contract criteria, the analytical measurement may not be repeated prior to taking the appropriate corrective action as specified in Exhibit E.

The Contractor must report all QC data in the exact format specified in Exhibits B and H.

This section outlines the minimum QA/QC operations necessary to satisfy the analytical requirements of the contract. The following QA/QC operations must be performed as described in this Exhibit:

1. Instrument Calibration
2. Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)
3. CRDL Standards for AA (CRA) and ICP (CRI)
4. Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses
5. ICP Interference Check Sample (ICS) Analyses
6. Spike Sample Analysis (S)
7. Duplicate Sample Analysis (D)
8. Laboratory Control Sample (LCS) Analysis
9. ICP Serial Dilution Analysis (L)
10. Instrument Detection Limit (IDL) Determination
11. Interelement Corrections for ICP (ICP)
12. Linear Range Analysis (LRA)
13. Furnace AA QC Analyses

1. <u>Instrument Calibration</u>

Guidelines for instrumental calibration are given in EPA 600/4-79-020 and/or Exhibit D. Instruments must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument standardization date and time must be included in the raw data.

For atomic absorption systems, calibration standards are prepared by diluting the stock metal solutions at the time of analysis. Date and time of preparation and analysis must be given in the raw data.

Calibration standards must be prepared fresh each time an analysis is to be made and discarded after use. Prepare a blank and at least three calibration standards in graduated amounts in the appropriate range. One atomic absorption calibration standard must be at the CRDL except for mercury. The calibration standards must be prepared using the same type of acid or combination of acids and at the same concentration as will result in the samples following sample preparation.

Beginning with the blank, aspirate or inject the standards and record the readings. If the AA instrument configuration prevents the required 4-point calibration, calibrate according to instrument manufacturer's recommendations, and analyze the remaining required standards immediately after calibration. Results for these standards must be within ± 5% of the true value. Each standards concentration and the calculations to show that the ±5% criterion has been met, must be given in the raw data. If the values do not fall within this range, recalibration is necessary.

The ± 5% criteria does not apply to the atomic absorption calibration standard at the CRDL.

Calibration standards for AA procedures must be prepared as described in Exhibit D.

Baseline correction is acceptable as long as it is performed after every sample or after the continuing calibration verification and blank check; resloping is acceptable as long as it is immediately preceded and immediately followed by CCV and CCB. For cyanide and mercury, follow the calibration procedures outlined in Exhibit D. One cyanide calibration standard must be at the CRDL. For ICP systems, calibrate the instrument according to instrument manufacturer's recommended procedures.. At least two standards must be used for ICP calibration. One of the standards must be a blank.

2. <u>Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)</u>

a. Initial Calibration Verification (ICV)

Immediately after each of the ICP, AA and cyanide systems have been calibrated, the accuracy of the initial calibration shall be verified and documented for every analyte by the analysis of EPA Initial Calibration Verification Solution(s) at each wavelength used for analysis. When measurements exceed the control limits of Table 1-Initial and Continuing Calibration Verification Control Limits for Inorganic Analyses (in Exhibit E), the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

If the Initial Calibration Verification Solution(s) are not available from EPA, or where a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for instrument calibration, but within the calibration range. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.

For ICP, the Initial Calibration Verification Solution(s) must be run at each wavelength used for analysis. For CN, the initial calibration verification standard must be distilled. The Initial Calibration Verification for CN serves as a Laboratory Control Sample; thus it must be distilled with the batch of samples analyzed in association with that ICV. This means that an ICV must be distilled with each batch of samples analyzed and that the samples distilled with an ICV must be analyzed with that particular ICV. The values for the initial and subsequent continuing calibration verifications shall be recorded on FORM II-IN for ICP, AA, and cyanide analyses, as indicated.

b. Continuing Calibration Verification (CCV)

To ensure calibration accuracy during each analysis run, one of the following standards is to be used for continuing calibration verification and must be be analyzed and reported for every wavelength used for the analysis of each analyte, at a frequency of 10% or every 2 hours during an analysis run, whichever is more frequent. The standard must also be analyzed and reported for every wavelength used for analysis at the beginning of the run and after the last analytical sample. The analyte concentrations in the continuing calibration standard must be one of the following solutions at or near the mid-range levels of the calibration curve:

1. EPA Solutions
2. NBS SRM 1643a
3. A Contractor-prepared standard solution

TABLE 1. INITIAL AND CONTINUING CALIBRATION VERIFICATION CONTROL LIMITS FOR INORGANIC ANALYSES

Analytical Method	Inorganic Species	% of True Value (EPA Set) Low Limit	High Limit
ICP/AA	Metals	90	110
Cold Vapor AA	Mercury	80	120
Other	Cyanide	85	115

The same continuing calibration standard must be used throughout the analysis runs for a Case of samples received.

Each CCV analyzed must reflect the conditions of analysis of all associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV). The duration of analysis, rinses and other related operations that may affect the CCV measured result may not be applied to the CCV to a greater extent than the extent applied to the associated analytical samples. For instance, the difference in time between a CCV analysis and the blank immediately following it as well as the difference in time between the CCV and the analytical sample immediately preceding it may not exceed the lowest difference in time between any two consecutive analytical samples associated with the CCV.

If the deviation of the continuing calibration verification is greater than the control limits specified in Table 1-Initial and Continuing Calibration Verification Control Limits for Inorganic Analyses, the analysis must be stopped, the problem corrected, the instrument must be recalibrated, the calibration verified and the reanalysis of preceding 10 analytical samples or all analytical samples analyzed since the last good calibration verification must be performed for the analytes affected. Information regarding the continuing verification of calibration shall be recorded on FORM II-IN for ICP, AA and cyanide as indicated.

3. <u>CRDL Standards for ICP (CRI) and AA (CRA)</u>

To verify linearity near the CRDL for ICP analysis, the Contractor must analyze an ICP standard (CRI) at two times the CRDL or two times the IDL, whichever is greater, at the beginning and end of each sample analysis run, or a minimum of twice per 8 hour working shift, whichever is more frequent, but not before Initial Calibration Verification. This standard must be run by ICP for every wavelength used for analysis, except those for Al, Ba, Ca, Fe, Mg, Na and K.

To verify linearity near the CRDL for AA analysis, the Contractor must analyze an AA standard (CRA) at the CRDL or the IDL, whichever is greater, at the beginning of each sample analysis run, but not before the Initial Calibration Verification.

Specific acceptance criteria for the two standards will be set by EPA in the future. In the interim, the Contractor must analyze and report these Standards on FORM II(PART 2)-IN.

4. <u>Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses</u>

a. Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Analyses

A calibration blank must be analyzed at each wavelength used for analysis immediately after every initial and continuing calibration verification, at a frequency of 10% or every 2 hours

during the run, whichever is more frequent. The blank must be analyzed at the beginning of the run and after the last analytical sample. Note: A CCB must be run after the last CCV that was run after the last analytical sample of the run. The results for the calibration blanks shall be recorded on FORM III-IN for ICP, AA and cyanide analyses, as indicated. If the magnitude (absolute value) of the calibration blank result exceeds the IDL, the result must be so reported in ug/L on FORM III-IN, otherwise report as IDL-U. If the absolute value blank result exceeds the CRDL (Exhibit C), terminate analysis, correct the problem, recalibrate, verify the calibration and reanalyze the preceding 10 analytical samples or all analytical samples analyzed since the last good calibration blank.

b. Preparation Blank (PB) Analysis

At least one preparation blank (or reagent blank), consisting of deionized distilled water processed through each sample preparation and analysis procedure (See Exhibit D, Section III), must be prepared and analyzed with every Sample Delivery Group, or with each batch[1] of samples digested, whichever is more frequent.

The first batch of samples in an SDG is to be assigned to preparation blank one, the second batch of samples to preparation blank two, etc. (see FORM III-IN). Each data package must contain the results of all the preparation blank analyses associated with the samples in that SDG.

This blank is to be reported for each SDG and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner:

1) If the absolute value of the concentration of the blank is less than or equal to the Contract Required Detection Limit (Exhibit C), no correction of sample results is performed.

2) If any analyte concentration in the blank is above the CRDL, the lowest concentration of that analyte in the associated samples must be 10x the blank concentration. Otherwise, all samples associated with the blank with the analyte's concentration less than 10x the blank concentration and above the CRDL, must be redigested and reanalyzed for that analyte (except for an identified aqueous soil field blank). The sample concentration is not to be corrected for the blank value.

3) If the concentration of the blank is below the negative CRDL, then all samples reported below 10x CRDL associated with the blank must be redigested and reanalyzed.

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[1] A group of samples prepared at the same time.

The values for the preparation blank must be recorded in ug/L for aqueous samples and in mg/Kg for solid samples on FORM III-IN for ICP, AA, and cyanide analyses.

5. <u>ICP Interference Check Sample (ICS) Analysis</u>

To verify interelement and background correction factors, the Contractor must analyze and report the results for the ICP Interference Check Samples at the beginning and end of each analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent, but not before Initial Calibration Verification. The ICP Interference Check Samples must be obtained from EPA (EMSL/LV) if available and analyzed according to the instructions supplied with the ICS.

The Interference Check Samples consist of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively (starting with Solution A) for all wavelengths used for each analyte reported by ICP.

Results for the ICP analyses of Solution AB during the analytical runs must fall within the control limit of ±20% of the true value for the analytes included in the Interference Check Samples. If not, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the analytical samples analyzed since the last good ICS. If true values for analytes contained in the ICS and analyzed by ICP are not supplied with the ICS, the mean must be determined by initially analyzing the ICS at least five times repetitively for the particular analytes. This mean determination must be made during an analytical run where the results for the previously supplied EPA ICS met all contract specifications. Additionally, the result of this initial mean determination is to be used as the true value for the lifetime of that solution (i.e., until the solution is exhausted).

If the ICP Interference Check Sample is not available from EPA, independent ICP Check Samples must be prepared with interferent and analyte concentrations at the levels specified in Table 2-Interferent and Analyte Elemental Concentrations Used for ICP Interference Check Sample. The mean value and standard deviation must be established by initially analyzing the Check Samples at least five times repetitively for each parameter on FORM IV-IN. Results must fall within the control limit of ±20% of the established mean value. The mean and standard deviation must be reported in the raw data. Results from the Interference Check Sample analyses must be recorded on FORM IV-IN for all ICP parameters.

TABLE 2. INTERFERENT AND ANALYTE ELEMENTAL CONCENTRATIONS USED FOR ICP INTERFERENCE CHECK SAMPLE

Analytes	(mg/L)	Interferents	(mg/L)
Ag	1.0	Al	500
Ba	0.5	Ca	500
Be	0.5	Fe	200
Cd	1.0	Mg	500
Co	0.5		
Cr	0.5		
Cu	0.5		
Mn	0.5		
Ni	1.0		
Pb	1.0		
V	0.5		
Zn	1.0		

6. Spike Sample Analysis (S)

The spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and measurement methodology. The spike is added before the digestion (i.e., prior to the addition of other reagents) and prior to any distillation steps (i.e., CN-). At least one spike sample analysis must be performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) or for each Sample Delivery Group.[2]

If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations must be performed using the results of the sample designated as the "original sample" (see section 7, Duplicate Sample Analysis). The average of the duplicate results cannot be used for the purpose of determining percent recovery. Samples identified as field blanks cannot be used for spiked sample analysis. EPA may require that a specific sample be used for the spike sample analysis.

The analyte spike must be added in the amount given in Table 3-Spiking Levels for Spike Sample Analysis, for each element analyzed. If two analytical methods are used to obtain the reported values for the same element within a Sample Delivery Group (i.e. ICP, GFAA), spike samples must be run by each method used.

[2]EPA may require additional spike sample analysis, upon Project Officer request, for which the Contractor will be paid.

If the spike recovery is not at or within the limits of 75-125%, the data of all samples received associated with that spike sample and determined by the same analytical method must be flagged with the letter "N" on FORMs I-IN and V-IN. An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the percent recovery does not meet the 75-125% recovery criteria.

For flame AA, ICP, and CN analyses, when the pre-digestion/pre-distillation spike recovery falls outside the control limits and the sample result does not exceed 4x the spike added, a post-digestion/post-distillation spike must be performed for those elements that do not meet the specified criteria (exception: Ag). Spike the unspiked aliquot of the sample at 2x the indigenous level or 2x CRDL, whichever is greater. Results of the post-digestion/post-distillation spike must be reported on FORM V(PART 2)-IN. Note: No post digest spike is required for Hg.

In the instance where there is more than one spike sample per matrix and concentration per method per SDG, if one spike sample recovery is not within contract criteria, flag all the samples of the same matrix, level, and method in the SDG. Individual component percent recoveries (%R) are calculated as follows:

$$\%\text{Recovery} = \frac{(SSR-SR)}{SA} \times 100$$

Where, SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

When sample concentration is less than the instrument detection limit, use SR = 0 only for purposes of calculating % Recovery. The spike sample results, sample results and % Recovery (positive or negative) must be reported on FORM V-IN for ICP, AA and cyanide analyses, as indicated.

The units for reporting spike sample results will be identical to those used for reporting sample results in FORM I-IN (i.e., ug/L for aqueous and mg/Kg dry weight basis for solid).

TABLE 3. SPIKING LEVELS FOR SPIKE SAMPLE ANALYSIS

	For ICP/AA (ug/L)		For Furnace AA (ug/L)		Other[1] (ug/L)
Element	Water	Soil[2]	Water	Soil	
Aluminum	2,000	*			
Antimony	500	500	100	100	
Arsenic	2,000	2,000	40	40	
Barium	2,000	2,000			
Beryllium	50	50			
Cadmium	50	50	5	5	
Calcium	*	*			
Chromium	200	200			
Cobalt	500	500			
Copper	250	250			
Iron	1,000	*			
Lead	500	500	20	20	
Magnesium	*	*			
Manganese	500	500			
Mercury					1
Nickel	500	500			
Potassium	*	*			
Selenium	2,000	2,000	10	10	
Silver	50	50			
Sodium	*	*			
Thallium	2,000	2,000	50	50	
Vanadium	500	500			
Zinc	500	500			
Cyanide					100

NOTE: Elements without spike levels and not designated with an asterisk, must be spiked at appropriate levels.

[1]Spiking level reported is for both water and soil/sediment matrices.

[2]The levels shown indicate concentrations in the final digestate of the spiked sample (200 mL final volume).

*No spike required.

7. Duplicate Sample Analysis (D)

One duplicate sample must be analyzed from each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) or for each Sample Delivery Group.[3] Duplicates cannot be averaged for reporting on FORM I-IN.

Duplicate sample analyses are required for percent solids. Samples identified as field blanks cannot be used for duplicate sample analysis. EPA may require that a specific sample be used for duplicate sample analysis. If two analytical methods are used to obtain the reported values for the same element for a Sample Delivery Group (i.e., ICP, GFAA), duplicate samples must be run by each method used.

The relative percent differences (RPD) for each component are calculated as follows:

$$RPD = \frac{|S - D|}{(S+D)/2} \times 100$$

Where, RPD = Relative Percent Difference
S = First Sample Value (original)
D = Second Sample Value (duplicate)

The results of the duplicate sample analyses must be reported on FORM VI-IN in ug/L for aqueous samples and mg/Kg dry weight basis for solid original and duplicate samples. A control limit of 20% for RPD shall be used for original and duplicate sample values greater than or equal to 5x CRDL (Exhibit C). A control limit of (±) the CRDL must be used for sample values less than 5x CRDL, and the absolute value of the control limit (CRDL) must be entered in the "Control Limit" column on FORM VI-IN.

If one result is above the 5x CRDL level and the other is below, use the ± CRDL criteria. If both sample values are less than the IDL, the RPD is not calculated on FORM VI-IN. For solid sample or duplicate results < 5x CRDL, enter the absolute value of the CRDL, corrected for sample weight and percent solids, in the "Control Limit" column.

If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an "*" on FORMs I-IN and VI-IN. In the instance where there is more than one duplicate sample per SDG, if one duplicate result is not within contract criteria, flag all samples of the same matrix, concentration, and method in the SDG. The percent difference data will be used by EPA to evaluate the long-term precision of the methods for each parameter. Specific control limits for each element will be added to FORM VI-IN at a later date based on these precision results.

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[3]EPA may require additional duplicate sample analyses, upon Project Officer request, for which the Contractor will be paid.

8. Laboratory Control Sample (LCS) Analysis

Aqueous and solid Laboratory Control Samples (LCS) must be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the EPA samples received. The aqueous LCS solution must be obtained from EPA (if unavailable, the Initial Calibration Verification Solutions may be used). One aqueous LCS must be prepared and analyzed for every group of aqueous samples in a Sample Delivery Group, or for each batch of aqueous samples digested, whichever is more frequent. An aqueous LCS is not required for mercury and cyanide analysis.

The EPA-provided solid LCS must be prepared and analyzed using each of the procedures applied to the solid samples received (exception: percent solids determination not required). If the EPA solid LCS is unavailable, other EPA Quality Assurance Check samples or other certified materials may be used. One solid LCS must be prepared and analyzed for every group of solid samples in a Sample Delivery Group, or for each batch of samples digested, whichever is more frequent.

All LCS results and percent recovery (%R) will be reported on FORM VII-IN. If the percent recovery for the aqueous LCS falls outside the control limits of 80-120% (exception: Ag and Sb), the analyses must be terminated, the problem corrected, and the samples associated with that LCS redigested and reanalyzed.

If the results for the solid LCS fall outside the control limits established by EPA, the analyses must be terminated, the problem corrected, and the samples associated with that LCS redigested and reanalyzed.

9. ICP Serial Dilution Analysis (L)

Prior to reporting concentration data for the analyte elements, the Contractor must analyze and report the results of the ICP Serial Dilution Analysis. The ICP Serial Dilution Analysis must be performed on a sample from each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) or for each Sample Delivery Group, whichever is more frequent. Samples identified as field blanks cannot be used for Serial Dilution Analysis.

If the analyte concentration is sufficiently high (minimally a factor of 50 above the instrumental detection limit in the original sample), the serial dilution (a five fold dilution) must then agree within 10% of the original determination after correction for dilution. If the dilution analysis for one or more analytes is not at or within 10%, a chemical or physical interference effect must be suspected, and the data for all affected analytes in the samples received associated with that serial dilution must be flagged with an "E" on FORM IX-IN and FORM I-IN.

The percent differences for each component are calculated as follows:

$$\% \text{ Difference} = \frac{|I - S|}{I} \times 100$$

where, I = Initial Sample Result
S = Serial Dilution Result (Instrument Reading x 5)

In the instance where there is more than one serial dilution per SDG, if one serial dilution result is not within contract criteria, flag all the samples of the same matrix and concentration in the Sample Delivery Group. Serial dilution results and "E" flags must be reported on FORM IX-IN.

10. Instrument Detection Limit (IDL) Determination

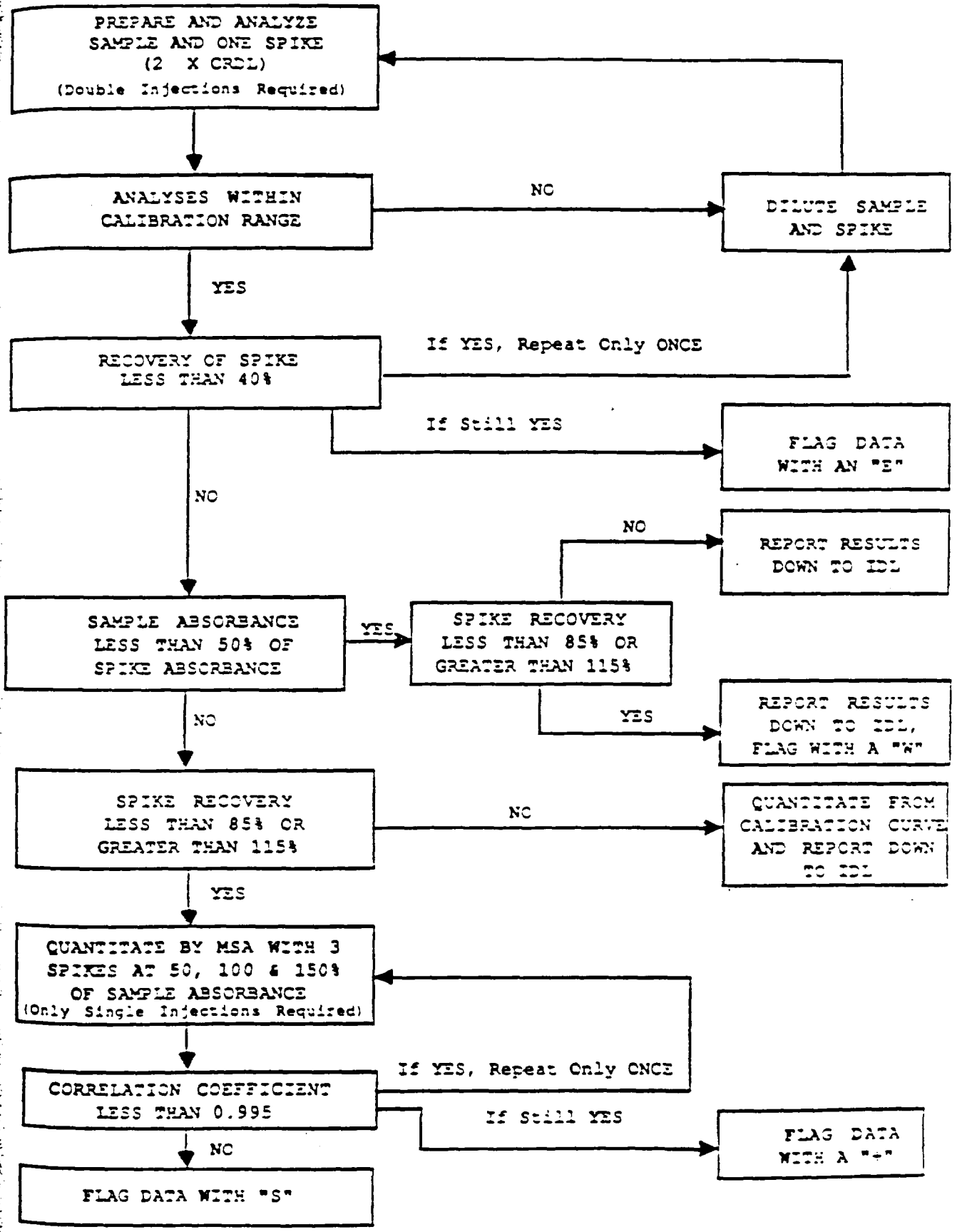
Before any field samples are analyzed under this contract, the instrument detection limits (in ug/L) must be determined for each instrument used, within 30 days of the start of contract analyses and at least quarterly (every 3 calendar months), and must meet the levels specified in Exhibit C.

The Instrument Detection Limits (in ug/L) shall be determined by multiplying by 3, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x-5x the instrument manufacturer's suggested IDL, with seven consecutive measurements per day. 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 normally performed between the analysis of separate samples). IDL's must be determined and reported for each wavelength used in the analysis of the samples.

The quarterly determined IDL for an instrument must always be used as the IDL for that instrument during that quarter. If the instrument is adjusted in anyway that may affect the IDL, the IDL for that instrument must be redetermined and the results submitted for use as the established IDL for that instrument for the remainder of the quarter.

IDLs must be reported for each instrument used on FORM X-IN submitted with each data package. If multiple AA instruments are used for the analysis of an element within a Sample Delivery Group, the highest IDL for the AAs must be used for reporting concentration values for that Sample Delivery Group. The same reporting procedure must be used formultiple ICPs.

Figure 1
FURNACE ATOMIC ABSORPTION ANALYSIS SCHEME



11. Interelement Corrections for ICP

Before any field samples are analyzed under this contract, the ICP interelement correction factors must be determined prior to the start of contract analyses and at least annually thereafter. Correction factors for spectral interference due to Al, Ca, Fe, and Mg must be determined for all ICP instruments at all wavelengths used for each analyte reported by ICP. Correction factors for spectral interference due to analytes other than Al, Ca, Fe, and Mg must be reported if they were applied.

If the instrument was adjusted in anyway that may affect the ICP interelement correction factors, the factors must be redetermined and the results submitted for use. Results from interelement correction factors determination must be reported on FORM XI(PART 1)-IN and FORM XI(PART 2)-IN for all ICP parameters.

12. Linear Range Analysis (LRA)

For all ICP analyses, a linear range verification check standard must be analyzed and reported quarterly (every 3 calendar months) for each element on FORM XII-IN. The standard must be analyzed during a routine analytical run performed under this contract. The analytically determined concentration of this standard must be within ± 5% of the true value. This concentration is the upper limit of the ICP linear range beyond which results cannot be reported under this contract without dilution of the analytical sample.

13. Furnace Atomic Absorption (AA) QC Analyses

Because of the nature of the Furnace AA technique, the special procedures summarized in Figure 1-Furnace AA Analysis Scheme ("MSA Tree") will be required for quantitation. (These procedures do not replace those in Exhibit D of this SOW, but supplement the guidance provided therein.)

a. All furnace analyses must fall within the calibration range. In addition, all analyses, except during full methods of Standard Addition (MSA), will require duplicate injections. The absorbance and concentration of each injection must be reported in the raw data as well as the average absorbance and concentration values. Average concentration values are used for reporting purposes. A maximum of 10 full sample analyses to a maximum 20 injections may be performed between each consecutive calibration verifications and blanks. The raw data package must contain absorbance and concentration values for both injections, the average value and the relative standard deviation (RSD) or coefficient of variation (CV). For concentrations greater than CRDL, the duplicate injection readings must agree within 20% RSD or CV, or the analytical sample must be rerun once (i.e., two additional burns). If the readings are still out, flag the value reported on FORM I-IN with an "M". The "M" flag is required for the analytical spike as well as the sample. If the analytical spike for a sample requires an "M" flag, the flag must be reported on FORM I- IN for that sample.

b. All furnace analyses for each analytical sample, including those requiring an "M" flag, will require at least an analytical spike to determine if the MSA will be required for quantitation. The analytical spike[4] will be required to be at a concentration (in the sample) 2x CRDL. This requirement for an analytical spike will include the LCS and the preparation blank. (The LCS must be quantitated from the calibration curve and corrective action, if needed, taken accordingly. MSA is not to be performed on the LCS or preparation blank, regardless of spike recovery results.) If the preparation blank analytical spike recovery is out of control (85-115%), the spiking solution must be verified by respiking and rerunning the preparation blank once. If the preparation blank analytical spike recovery is still out of control, correct the problem and reanalyze all analytical samples associated with that blank. An analytical spike is not required on the pre-digestion spike sample.

The analytical spike of a sample must be run immediately after that sample. The percent recovery (%R) of the spike, calculated by the same formula as Spike Sample Analyses (see item 6, this section), will then determine how the sample will be quantitated, as follows:

1) If the spike recovery is less than 40%, the sample must be diluted and rerun with another spike. Dilute the sample by a factor of 5 to 10 and rerun. This step must only be performed once. If after the dilution the spike recovery is still <40%, report data and flag with an "E" to indicate interference problems.

2) If the spike recovery is greater than or equal to 40% and the sample absorbance is less than 50% of the "spike"[5], report the sample results to the IDL. If the spike recovery is less than 85% or greater than 115%, flag the result with a "W".

3) If the sample absorbance is greater than or equal to 50% of the spike and the spike recovery is at or between 85% and 115%, the sample must be quantitated directly from the calibration curve and reported down to the IDL.

4) If the sample absorbance is greater than or equal to 50% of the spike and the spike recovery is less than 85% or greater than 115%, the sample must be quantitated by MSA.

---

[4]Analytical Spikes are post-digestion spikes to be prepared prior to analysis by adding a known quantity of the analyte to an aliquot of the <u>digested</u> sample. The unspiked sample aliquot must be compensated for any volume change in the spike samples by addition of deionized water to the unspiked sample aliquot. The volume of the spiking solution added must not exceed 10% of the analytical sample volume; this requirement also applies to MSA spikes.

[5]"Spike" is defined as [absorbance of spike sample] minus [absorbance of the sample].

c. The following procedures will be incorporated into MSA analyses.

1) Data from MSA calculations must be within the linear range as determined by the calibration curve generated at the beginning of the analytical run.

2) The sample and three spikes must be analyzed consecutively for MSA quantitation (the "initial" spike run data is specifically excluded from use in the MSA quantitation). Only single injections are required for MSA quantitation.

   Each full MSA counts as two analytical samples towards determining 10% QC frequency (i.e., five full MSAs can be performed between calibration verifications).

3) For analytical runs containing only MSAs, single injections can be used for QC samples during that run. For instruments that operate in an MSA mode only, MSA can be used to determine QC samples during that run.

4) Spikes must be prepared such that:

   a) Spike 1 is approximately 50% of the sample absorbance.

   b) Spike 2 is approximately 100% of the sample absorbance.

   c) Spike 3 is approximately 150% of the sample absorbance.

5) The data for each MSA analysis must be clearly identified in the raw data documentation (using added concentration as the x-variable and absorbance as the y-variable) along with the slope, x-intercept, y-intercept and correlation coefficient (r) for the least squares fit of the data. The results must be reported on FORM VIII-IN. Reported values obtained by MSA must be flagged on the data sheet (FORM I-IN) with the letter "S" if the correlation coefficient is greater than or equal to 0.995.

6) If the correlation coefficient (r) for a particular analysis is less than 0.995, the MSA analysis must be repeated once. If the correlation coefficient is still less than 0.995, report the results on FORM I-IN from the run with the best "r" and flag the result with a "+" on FORM VIII-IN and FORM I-IN.

ATTACHMENT II

Instrument Detection Limit and Spiking Level for Drinking Water

Compound	Required Instrument Detection Limit[1] ug/L			Required Matrix Spike Concentrations ug/L		
	GFAA	ICP	Other	GFAA	ICP	Other
Metal:						
1. Aluminum		100			2000	
2. Antimony [2]	5			20	500	
3. Arsenic	5			20		
4. Barium		50			2000	
5. Beryllium		5			50	
6. Cadmium [2]	0.5			2	50	
7. Calcium [3]		1000			50,000	
8. Chromium		10			200	
9. Cobalt		10			500	
10. Copper		10			250	
11. Iron		100			1000	
12. Lead [2]	2			20	500	
13. Magnesium [3]		1000			25,000	
14. Manganese		10			200	
15. Mercury			0.2			1.0
16. Nickel		20			400	
17. Potassium [3]		2000			20,000	
18. Selenium	2			10		
19. Silver		5			50	
20. Sodium [3]		1000			50,000	
21. Thallium	2			20		
22. Vanadium		10			500	
23. Zinc		20			200	
24. Cyanide			10			100

[1] Instrument Detection Limits (IDL) must be met before any samples are analyzed. The Lab may submit their quarterly Form XI with each case if all IDLs meet the detection limits. If Detection Limits cannot be met by using ICP, USE OF GFAA REQUIRED.

[2] ICP analysis results may only be reported for Sb, Cd and Pb, if the concentration is ≥ 10 times the IDL of instrument used. If ICP results are reported, all ICP audits are required including matrix spike.

[3] Report Ca, Mg, Na and K on separate Form V for Matrix Spike if a separate aliquot is used for this spike.

Rev. ~~4.0~~ ~~5.0~~ 6.0
~~5/88~~ ~~4/88~~ 6/89

APPENDIX B-10

LOW LEVEL METALS BY AA (ENSECO)

Subject or Title:

CLP ATOMIC ABSORPTION

SOP No.:	Revision No.:	Effective Date:
LM-RMA-2033	1.0	August 1, 1989

Supersedes: **Original**

1 Scope and Application

1.1 These methods are applicable for the determination of Arsenic, Lead, Selenium, Thallium, Cadmium, and Antimony.

1.2 Detection Limits

1.2.1 Contract Required Detection Limits (CRDL)

1.2.1.1 Arsenic, Thallium, Antimony (SAS): 10 ug/L.

1.2.1.2 Selenium, Lead: 5 ug/L

1.2.1.3 Cadmium (SAS): 0.5 ug/L

1.2.2 Instrument Detection Limits (IDL)

1.2.2.1 Detection Limits are calculated quarterly according to SOW 787 protocol, see appendix.

1.2.2.2 Arsenic, Selenium, Thallium, Lead, and Antimony: IDL range: 1 - 3 ug/L.

1.2.2.3 Cadmium IDL range: 0.1 - 0.3 ug/L

Prepared by:	Date:
Scott Hoatson	August 1, 1989
Management Approval: Kathryn K Okazaki	Date: 8/10/89
QA Officer Approval: Tina M Rigoni	Date: 8/10/89

1.3 The methods are suited for the determination of trace concentrations of the elements listed in water and soil samples.

1.4 The range of the analysis is from 0 - 100 ug/L of analyte in solution.

1.5 The approximate analytical time of one sample with proper QC is 6 - 8 min.

2. Summary of Method

2.1 Known aliquots of sample are injected into an electrothermally heated graphite furnace. The furnace is programmed to drive off solvents and interferents by stepping through a series of drying and charring stages. The analyte is then atomized by a rapid increase in the temperature of the furnace. The atoms produced by this atomization absorb atomic emission from a source lamp. The absorbance is compared to that of a standard of known concentration. An elemental concentration is then calculated by the instrument and displayed on a digital readout.

3. Comments

3.1 Interferences

3.1.1 Chemical and physical interferences are prevalent when analyzing samples using these methods. Care must be taken to minimize interferences by adhering precisely to the protocols outlined in the appendix.

3.1.2 Matrix modifier is added to the samples prior to the determination of the analytes. The modifier prevents premature loss of the analytes as volatile salts and/or helps prevent signal suppression during atomization due to formation of high temperature stable complexes.

3.1.3 Background interferences must be compensated for by the use of either deuterium or Zeeman background correction.

3.2 Helpful Hints

3.2.1 Use the correct digest for aliquoting, it is marked HGA/HNO3.

3.2.2 The dilution factor for soil digests is 200X unless otherwise noted on the prep sheet.

3.2.3 Be sure to read the prep sheet for SPECIAL INSTRUCTIONS.

3.2.4 Use UNLV made standards for ICV's and LCS's.

3.2.5 Show absorbances or concentrations for THREE calibration standards.

3.2.6 Analyze a calibration standard and calibration blank every twenty sample injections.

3.2.7 Use duplicate injections and set the instrument to print the average and coefficient of variation (CV). The CV must be less than or equal to 20% or the sample less than the CRDL.

3.2.8 There is an analytical spike concentrate already prepared. Spike 100 ul of this solution into 10 ml of sample.

3.2.9 If the analytical spike recovery is less than 40%, dilute the sample 10x, one time, and respike the sample.

3.2.10 Analytical spike concentrations are two times the CRDL.

3.2.11 Matrix spike concentrations are as follows: As 40 ppb, Se 10 ppb, Tl 50 ppb, Pb 20 ppb.

3.2.12 When correcting a mistake, draw one line through the mistake, make correct entry, and initial the correction.

3.2.13 All reruns must be explained on bench sheet.

4. Safety Issues

4.1 Many of the metals determined by the methods are extremely toxic and/or carcinogenic. Care should be exercised when preparing standards from concentrated stock solutions. Wear gloves, safety glasses, and a lab coat.

4.2 All of the samples may contain unknown contaminants at unknown levels other than the analytes being tested for. Appropriate care should be taken when handling them.

4.3 Emission from hollow cathode (HCL) and electrodeless discharge (EDL) lamps is often intense and emitted at wavelengths that are hazardous to vision. **AVOID LOOKING DIRECTLY AT THE LAMPS WHEN IN USE.**

4.4 Zeeman background correction uses an extremely strong magnetic field for its operation. This magnetic field is HARMFUL TO PACEMAKER WEARERS.

5. Apparatus

5.1 Perkin Elmer model 2380 atomic absorption spectrophotometer equipped with a HGA 400 graphite furnace, an AS 40 autosampler, and Deuterium background correction.

5.2 Perkin Elmer model 5000 atomic absorption spectrophotometer equipped with a HGA 500 graphite furnace, an AS 40 autosampler, and Zeeman background correction.

5.3 Perkin Elmer model 5100 atomic absorption spectrophotometer equipped with a HGA 600 graphite furnace, an AS 60 autosampler, and Zeeman background correction. The 5100 is interfaced with an Epson Equity III+ computer with a mouse pointer.

5.4 Eppendorf 10 - 100 ul and 100 - 1000 ul adjustable pipettors.

5.5 Oxford 1 - 5 ml and 5 - 10 ml adjustable pipettors

6. Reagents, Standards and Supplies

6.1 Nitric Acid, Baker Instra-Analyzed or equivalent.

6.2 Deionized Water

6.3 Nickel powder, 99.99%, Spex Industries Inc. or equivalent.

6.4 EPA HGA Analytical Spike solution prepared by RMAL QA/QC Standards Prep Department.

6.5 EPA HGA Calibration Standard stock solution prepared by RMAL QA/QC Standards Prep Department.

6.6 EPA CCV stock solution prepared by RMAL QA/QC Standards Prep Department.

6.7 ICV solution concentrates supplied by EPA UNLV.

6.8 2% Nickel Nitrate solution, prepared by dissolving 20 g of Nickel powder in approximately 250 ml of 20% Nitric Acid (use extreme caution and only add a small amount of nickel at a time until it is all dissolved). Filter solution and bring final volume up to 1 liter.

6.9 Five dram snap cap vials.

6.10 Two ml auto analyzer sample cups.

6.11 Disposable .45 um syringe filters and syringes. Gelman or equivalent.

7. Procedure

7.1 Aliquoting

7.1.1 Obtain the RMA numbers of the next case to be aliquoted from the board in the lab supervisor's office.

7.1.2 Go to the file cabinets and pull the Graphite Furnace worksheets and the Prep sheet from the appropriate file folder. Photocopy all of the sheets and replace the original Prep sheet back in the file folder.

7.1.3 The sample digests are located on shelves A-0 through E-5 between the AA and ICP areas. Remove the digests from the shelves and place them in order along the top of a bench. Place two 5 dram vials in front of each digest bottle.

7.1.4 Label each vial with the correct RMA project number, and sample number. Follow the sample number with an "A" to denote the analytical spike. Place the correct dilution factor on the vials if the sample is diluted beyond the prep dilution.

7.1.5 Using a calibrated 100ul pipettor, add 100ul of 2% nickel nitrate solution to each of the vials. Add 100ul of analytical spike concentrate to each of the vials marked with an "A". With a 10ml pipettor add 10ml of sample to each vial. Cap vial and shake. If there is low sample volume, reduce the volume aliquoted and spiked, while maintaining the same ratios.

7.1.6 Replace the sample digests to the same shelf they were removed from. Place the aliquots into a box, label box with RMA project number, and place box on rolling aluminum shelves.

7.1.7 Place the worksheets along with the copy of the Prep sheet in the accordion file folder. Label the slot in the accordion folder with the RMA project number and place an "A" on the assignment board in lab supervisor's office

7.2 Instrument Set Up (General)

7.2.1 Turn on power and utilities.

7.2.2 Put lamp into instrument.

7.2.3 Light lamp in AA ONLY mode.

7.2.4 Set correct wavelength for analyte.

7.2.5 Optimize instrument energy after a 5-10 minute warm-up time

7.2.6 Check to see that lamp energy can be turned up past autogain energy. It is recommended that the lamp energy exceed EE.

7.2.6.1 If minimum energy cannot be achieved then reoptimize instrument and try again.

7.2.6.2 If minimum energy still cannot be achieved then the lamp is probably faulty and needs to be replaced at this point.

7.2.7 Check baseline by setting time to 1 sec. and setting the instrument mode to CONTINUOUS. Please note that the instrument is still set at AA ONLY.

7.2.7.1 If baseline is unstable, reoptimize the AA and check the baseline again.

7.2.7.2 If baseline is still unstable, there may be a problem with the lamp and the lamp should be replaced at this point.

7.2.8 Switch background correction to AA-BKGRND. If the lamp is an EDL then set the lamp wattage to 80% of the recommended wattage.

7.2.9 After approximately 10 minutes check the baseline again. Follow step 7 except keep instrument setting at AA-BKGRND.

7.2.9.1 If baseline is unstable, reoptimize the AA and check the baseline again.

7.2.9.2 If baseline is still unstable, visually inspect the furnace to verify alignment (2380 only). If alignment is out, realign furnace (see 7.5)

7.2.9.3 If the problem persists then the D2 lamp may be the problem. The power on the EDL power supply may be turned up 1-2 watts or the D2 lamp may need to be replaced at this point. Consult with your supervisor on this matter if not sure which way to go.

7.2.10 Program the autosampler, furnace, and AA in accordance with standard methods (see attachment)

7.2.11 Analyze blanks until baseline is stable before attempting standardization.

7.3 Calibration

7.3.1 Prepare calibration standards (As, Se, Tl, Pb) daily by diluting the EPA Calibration stock solution as follows:

7.3.1.1 Blank - add 100 ul of 2% nickel solution to 10 ml of 1% HNO_3 in a 5 dram vial.

7.3.1.2 10 ppb - add 10 ul of stock and 100 ul of 2% nickel solution to 10 ml of 1% HNO_3 in a 5 dram vial.

7.3.1.3 50 ppb - add 50 ul of stock and 100 ul of 2% nickel solution to 10 ml of 1% HNO_3 in a 5 dram vial.

7.3.1.4 100 ppb - add 100 ul of stock and 100 ul of 2% nickel solution to 10 ml of 1% HNO3 in a 5 dram vial.

7.3.1.5 For Cadmium, make up standards at 0.5, 2.0, and 10 ppb levels. Do not use 2% nickel solution.

7.3.1.6 For Antimony, make up standards at the same levels as As, Se, Tl, and Pb. Use the 2% nickel solution.

7.3.2 Pour the calibration standards into 2 ml auto sampler vials. Place the vials on the tray in this order:

7.3.2.1 Blank in position marked AZ

7.3.2.2 10 (0.5 for Cd) ppb in position marked S1

7.3.2.3 50 (2.0 for Cd) ppb in position marked S2

7.3.2.4 100 (10.0 for Cd) ppb in position marked S3

7.3.3 Set the AA to average two injections of each sample and report the coefficient of variation (RSD).

7.3.4 Place the samples to be run in the sample tray in the order listed on the Graphite Furnace worksheet.

7.3.5 Start the analysis by pressing the start/stop key on the front of the AS-40 controller panel (2380, 5000). For 5100 use see below.

7.3.6 The AA will automatically calculate the calibration curve. The calibration is verified according to the quality assurance requirements of SOW 787 (see Attachment).

7.3.7 To shut down the instrument, reverse the process outlined in section 7.2.

7.4 Calibration and Analysis - 5100 PC Operation

7.4.1 Turn on the power to:

7.4.1.1 The 5100 component

7.4.1.2 The Zeeman magnet component

7.4.1.3 The HGA 600

7.4.1.4 The computer, monitor, and printer

7.4.2 The first screen menu will be the DESKTOP menu. Using the "mouse", set the arrow pointer to 5100_PC.EXEC and "click" twice.

7.4.3 The next menu will be the BENCHTOP menu. Using the mouse, move the arrow pointer to the desired element and "click" twice.

7.4.4 The next screen inquires which programs to be run. Using the mouse move the arrow pointer to ELEMENTS and "click" once.

7.4.5 Select the elements that you want to run by moving the arrow pointer to the desired element and "click" twice. The computer will call up all of the necessary information stored for that element (note: Each element has its own lamp position on the lamp turret. The computer will automatically turn the turret to that position. When this is complete the EDL lamp can now be lit.).

7.4.6 To align lamps move the arrow pointer to the top of the screen to the word WINDOWS, a list of windows will drop down. Move the arrow pointer to ALIGN LAMPS and "click" once. Adjust the lamp alignment to give the maximum energy. If the energy goes offscale then press F2 on the keyboard to reset the autogain (or move the arrow to the box marked AGC/AIC and "click" once).

7.4.7 The figures in the appendix refer to a typical set-up of the ELEMENT PARAMETERS file found under WINDOWS. The only changes normally made are found in figure 2. Line 4 refers to the final concentration units: set to ug/l for waters and mg/L for soils. Line 5 refers to the number of replicates or "burns". The most common change would be from 2 replicates to 1 replicate for MSA analyses. Note that the volume for the standards in figure 5 must match the sample volume in figure 4.

7.4.8 The sample names are entered next. Call up WINDOWS again and "click" once on ID/WEIGHT PARAMETER. A blank file will appear. A file exists which has the first 5 control samples and the position numbers 1-35 already entered. This file is named INITSTD.A60. Call up this file by either typing in the name or moving the arrow pointer as before. Figure 7 illustrates how the top portion should look(the top portion being the portion in brackets). Do Not change this, the final concentration will not be reported correctly. The dilution factor (prep or analytical) should be entered in the DILUTION column with each sample individually.

7.4.9 To start an analysis run, call up AS-60 CONTROL window (figure 8). This window should already be on the screen (The screen holds up to 5 windows. Any window on the screen may be called up touching a part of the window with the arrow pointer and "clicking" once.).

Figure 8 shows the AS-60 control window. Note that the information in section 1 is called up automatically. DO NOT enter anything on the line labeled DATA FILE as of yet. Enter the name of your ID/WEIGHT file where shown. Enter the locations of the samples to run on the next line. Using the mouse, turn the PRINTER on (leave the SAVE DATA box off). Section 3 controls the autosampler. RUN ALL will cause the instrument to standardize <u>and</u> analyze the samples entered in section 2. RUN SAMPLES will cause the instrument to only analyze the samples entered in section 2 <u>without</u> standardizing.

7.4.10 The HGA methods are the similar to the other instruments with the following exceptions.

7.4.10.1 Instrument Model Z5100

7.4.10.2 Background Correction - Zeeman

7.4.10.3 Integration - Peak Height with 37 pt. peak smoothing. (Arsenic-Peak Area)

7.4.10.4 HGA model 600

7.4.10.5 Graphite tube - pyro/platform

The furnace programs for the different elements are shown in figures 9-13.

7.5 Furnace Alignment (2380)

7.5.1 Insert and light a Copper HCL (a good EDL lamp may be used instead) lamp according to PE recommendations.

7.5.2 Set wavelength to 324.7 nm.

7.5.3 Peak in lamp energy.

7.5.4 Set SIGNAL to Absorbance.

7.5.5 Be sure print function is turned of.

7.5.6 Set Mode to Continuous.

7.5.7 Set BACKGROUND CORRECTION to AA-ONLY.

7.5.8 Set integration time to 0.5 seconds.

7.5.9 Remove Quartz windows from furnace.

7.5.10 Swing entire furnace out of the light path. Be sure water lines in back are also out of the way.

7.5.11 Autozero the instrument.

7.5.12 Swing furnace back into position.

7.5.13 If absorbance readings are at or near zero (+-.003), stop, the furnace is aligned.

7.5.14 If absorbance readings are negative, return to step 10.

7.5.15 If absorbance readings are positive, continue.

7.5.16 Unlock Furnace by moving the locking bar to the left. The locking bar is not readily visible. It is located directly in front of the furnace assembly below the heavy electric cables.

7.5.17 Turn the horizontal and vertical adjustment knobs until absorbance readings are at or near zero.

7.5.18 Lock the furnace into place by moving the locking bar to the right.

7.5.19 Replace quartz windows and make entry into instrument log book.

8. QA/QC Requirements.

8.1 Requirements are stated in Section E of SOW 787 (see Attachment).

8.2 Data must be recorded on bench sheet and readily available for review.

8.3 Units and sample type must be clearly identified on the bench sheets.

8.4 Care must be taken not to exceed the working range of the instrument. Self absorption occurs with any atomic absorption technique at higher concentrations. The upper limit of quantification varies by element.

9. Calculations

9.1 Waters - sample result x dilution factor = final result ug/L

9.2 Soils - sample result x dilution factor / 1000 = final result mg/Kg.

9.3 Method of Standard Addition HP - 11C Use.

9.3.1 Turn on calculator and clear statistics register. Press f;clear sum.

9.3.2 Set calculator to 4 decimal places. Press f;fix;4.

9.3.3 Key in data pairs for MSA as follows:

9.3.3.1 Concentration from instrument; ENTER;

9.3.3.2 Concentration Added; sum +.

9.3.3.3 Enter in all 4 data pairs for MSA in this way.

9.3.4 To determine the correlation coefficient (r); Press f;$\hat{y}$,r;x≷y.

9.3.5 To determine the y-intercept; Press f;L.R.

9.3.6 The slope is determined by following STEP 5 and pressing x≷y.

9.3.7 The final concentration is determined by following STEP 6 and pressing the DIVIDE key.

10. Reporting

10.1 Reporting requirements are stated in Section E of SOW 787 (see Attachment).

10.2 Significant Figures

10.2.1 Values less than 10 - 2 significant figures.

10.2.2 Values greater than or equal to ten - 3 significant figures.

11. Scheduling

11.1 Projects are listed on the scheduling board according to the rules of the department KANBAN (see 13.0).

11.2 The analyst chooses which samples to analyze according to due date priority and instrument availability.

12. EPA Metals Kanban

12.1 Purpose

This is a system that is intended to improve the flow of work through the group from log-in to the copy room. Projects should be completed in a more timely fashion with a reduction of work in progress at any given time. The estimated process time is approximately 4 days from start to finish.

12.2 Definitions

12.2.1 A KANBAN will consist of approximately 40 CLP and Industrial CLP samples. The size of the KANBAN may be adjusted in the future if it is decided that this is necessary.

12.2.2 A SAMPLE is a billable sample according to the weekly backlog sheet. To be prepped for, and analyzed by, HGA, CVAA, CN, ICP, and % solids.

12.2.3 ANDON card is a red 3x5 card with a blue 3x5 card clipped to it. The blue card contains the following information: the project numbers of the samples in the KANBAN, the samples matrices, the required parameters, the project due dates, the numbers of samples in each project, and the number of samples in the total KANBAN.

12.3 Process

12.3.1 Samples are logged in and project folders completed.

12.3.2 Completed project folders are placed in the COMPLETED FOLDER basket for folder review. Any industrial folders will be given to the appropriate PA by the login technician and will be placed in the COMPLETED FOLDER basket by the login technician after review.

12.3.3 Each folder is reviewed for completeness and correctness, and then it is placed in the project filing cabinet next to the wet chem lab, and each project is entered in the blue prep book.

12.3.4 A blue KANBAN card is filled out for each KANBAN. The projects are assigned to a KANBAN on basis of due date using the backlog to determine the order.

12.3.5 The completed blue KANBAN cards are then placed in the "KANBAN In Waiting" clip on the side of the project folder filing cabinet.

12.3.6 The data review analyst will place the folders for a KANBAN completed by operations along with its blue card in the top basket of a two tiered basket located on top of the project folder filing cabinet. The buffer KANBAN in data review can then also be reviewed and placed in the lower of the two tiered baskets. No further KANBANS of data can be reviewed until report writing "pulls" the first of these completely reviewed KANBANS.

12.3.7 The flow of a KANBAN through prep, analysis, review, and report writing is determined by a "pull" system initiated by report writing. When report writing has completed writing and reviewing its previous KANBAN, their supervisor will come back and pull the first KANBAN from the completed basket. If report writing does not already have a buffer KANBAN, the report writing supervisor can take the buffer KANBAN with them also.

12.3.8 The data review analyst can then pull a KANBAN of data from the "KANBAN #1" basket in the metals analyst supervisor's office.

12.3.9 The analysts then move any data that is in the "KANBAN #2" basket into the "KANBAN #1" basket.

12.3.10 The analysts also then pull the next KANBAN from prep by moving the top blue KANBAN card from the "KANBAN in Waiting" clip to the "KANBAN to be prepped"clip.

12.3.11 When the data review analyst pulls a KANBAN of data from the metals analysts, the metals lab is ready for a buffer KANBAN:

12.3.11.1 A metals analyst will get the ready buffer KANBAN from the prep lab.

12.3.11.2 The analyst will remove the blue card from the ANDON card and tape this card to the tracking board next to the project numbers it applies to.

12.3.12 The metals analysis can be started on the buffer KANBAN when all the analyses are completed or in progress for the projects in the "KANBAN #1" basket.

12.3.13 When the prep lab task group determines that they have completed all the necessary parameters for their previous KANBAN including ICP and furnace preps, Hg preps and analysis, CN- repreps, etc., the prep supervisor will take the blue card from the "KANBAN to be prepped" clip and attach it to the red ANDON card. The cards will be placed in the appropriate window with the blue card facing the prep lab to signal that a KANBAN is in progress.

12.3.14 Sample preparation puts the HGA and ICP completed preps for the KANBAN on the counter by the door in the soils prep lab. When the task group has determined that they have completed all parameters for that KANBAN, they will turn the ANDON card around so the RED side faces into the soils lab. This indicates that no more preps are to be started. The BLUE side is therefore facing the metals lab and indicates to the analysts that another KANBAN is ready for analysis.

12.4 Important Principles

12.4.1 No more than 2 KANBANS of work are allowed at any station (not including RUSH or REWORK). This will facilitate a smoother flow of work throughout the system and insure that no group starts "pushing" work through.

12.4.2 The KANBANS include metals or metals + CN projects only.

12.4.3 Quick turnaround projects are NOT included in the KANBAN. A quick turnaround is a turnaround of 1 week or less.

12.4.4 The KANBAN projects MUST be started at each step as soon as possible. They will take priority over other projects unless the other projects are coming against holding times or due dates.

12.4.5 Repreps and rework on a KANBAN must have top priority. They must be scheduled before any other preps or analyses. The entire KANBAN will be held up from proceeding to report writing until this data is turned in and reviewed.

12.5 Documentation Procedures for Repreps and Reanalyses

12.5.1 It is required that a "reprep request form" be filled out every time a reprep is needed. The reprep request forms will be put in the appropriate file folders along with the data.

12.5.2 A prep sheet must be filled out every time repreps are performed for any parameter. The prepsheet is the sheet used to document preps for HGA, ICP, Hg, CN, and % solids.

12.5.3 The required procedures for processing reprep documentation follow:

12.5.3.1 When a Hg reprep is required, the Hg analyst or the data review chemist will fill out a reprep request form. A prepsheet needs to be filled out for the reprep. As with all repreps, make sure that any dilution factors, reduced volumes, etc. are correctly noted on the reprep sheet. After the reanalysis is complete, the data along with the reprep request sheet and the reprep sheet need to be turned into the data review chemist.

12.5.3.2 When a CN reprep is required, the wet chem supervisor will submit a reprep request form to the prep lab. The analyst doing the redistillation needs to fill out the necessary reprep forms. A reprepsheet needs to be filled out and put in the file folder. The CN distillation log that gets turned in to the wet chem supervisor also has to be filled out. When the distillation is complete, place the distillation log along with the attached reprep request form in the "CN prep sheet folder" in the hanging file outside of the wet chem supervisor's office.

12.5.3.3 When HGA and/or ICP repreps are required, a reprep request form will be turned into the prep lab. When the repreps are completed, a reprep sheet needs to be filled out. This form along with its attached reprep request form need to be put in the box along with the samples and brought to the metals lab. When the analyst has finished the reprep analysis, they will place the data along with the reprep sheet and the reprep request form in the appropriate data basket in the metals supervisor's office.

12.5.4 The data review chemist will be filling out a "reanalysis request form" when she finds data problems as a result of review. The analyst who completes the reanalyses will turn the data in to the appropriate data basket with the reanalysis request form attached to it.

13. References

13.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 2nd Edition, USEPA, 1982.

13.2 Analytical Methods for Furnace Atomic Absorption Spectrometry, Publication B332, Perkin Elmer & Co., Ueberlingen, West Germany, January 1984.

13.3 Instruction manuals for Perkin Elmer Instrumentation, Models 2380 and 5000 atomic absorption spectrophotometers, AS 40 Autosampler, and the HGA 400 and HGA 500 Graphite furnaces.

13.4 Analytical Methods for AA Spectrometry, Perkin Elmer & Co., Norwalk, CT, revised 9/76.

13.5 SOW 787, United States Environmental Protection Agency.

13.6 Users Manual and Reference Manual for Perkin Elmer Model 5100PC Atomic Absorption Spectrometer.

ATTACHMENTS

SOW 787 SECTION E - QA/QC

APPENDIX B-10
LOW LEVEL METALS BY AA (ENESCO)

PAGES 22, 23, AND 24

NOT AVAILABLE

AS OF 12/18/00

SECTION II

SPECIFIC QA/QC PROCEDURES

The quality assurance/quality control (QA/QC) procedures defined herein must be used by the Contractor when performing the methods specified in Exhibit D. When additional QA/QC procedures are specified in the methods in Exhibit D, the Contractor must also follow these procedures. NOTE: The cost of performing all QA/QC procedures specified in this Statement of Work is included in the price of performing the bid lot, except for duplicate, spike, and laboratory control sample analyses, which shall be considered separate sample analyses.

The purpose of this document is to provide a uniform set of procedures for the analysis of inorganic constituents of samples, documentation of methods and their performance, and verification of the sample data generated. The program will also assist laboratory personnel in recalling and defending their actions under cross examination if required to present court testimony in enforcement case litigation.

The primary function of the QA/QC program is the definition of procedures for the evaluation and documentation of sampling and analytical methodologies and the reduction and reporting of data. The objective is to provide a uniform basis for sample collection and handling, instrument and methods maintenance, performance evaluation, and analytical data gathering and reporting. Although it is impossible to address all analytical situations in one document, the approach taken here is to define minimum requirements for all major steps relevant to any inorganic analysis. In many instances where methodologies are available, specific quality control procedures are incorporated into the method documentation (Exhibit D). Ideally, samples involved in enforcement actions are analyzed only after the methods have met the minimum performance and documentation requirements described in this document.

The Contractor is required to participate in the Laboratory Audit and Intercomparison Study Program run by EPA EMSL-Las Vegas. The Contractor can expect to analyze two samples per calendar quarter during the contract period.

The Contractor must perform and report to SMO and EMSL/LV as specified in Exhibit B quarterly verification of instrument detection limits (IDL) by the method specified in Exhibit E, by type and model for each instrument used on this contract. All the IDLs must meet the CRDLs specified in Exhibit C. For ICP methods, the Contractor must also report, as specified in Exhibit B, linearity range verification, all interelement correction factors, wavelengths used, and integration times.

For QA/QC procedures, two different types of samples are specified. A "sample received" is a field sample or PE sample, each of which has an EPA Sample Number and is accompanied by sample Traffic Report documentation. An "analytical sample" is each "analysis" performed (i.e.,

each cuvette, cup, tube or container in auto samplers). A "frequency of 10%" means once every 10 analytical samples. NOTE: Calibration blanks and calibration verification samples are not counted as analytical samples when determining 10% frequency.

If any QC measurement fails to meet contract criteria, the analytical measurement may not be repeated prior to taking the appropriate corrective action as specified in Exhibit E.

The Contractor must report all QC data in the exact format specified in Exhibits B and H.

This section outlines the minimum QA/QC operations necessary to satisfy the analytical requirements of the contract. The following QA/QC operations must be performed as described in this Exhibit:

1. Instrument Calibration
2. Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)
3. CRDL Standards for AA (CRA) and ICP (CRI)
4. Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses
5. ICP Interference Check Sample (ICS) Analyses
6. Spike Sample Analysis (S)
7. Duplicate Sample Analysis (D)
8. Laboratory Control Sample (LCS) Analysis
9. ICP Serial Dilution Analysis (L)
10. Instrument Detection Limit (IDL) Determination
11. Interelement Corrections for ICP (ICP)
12. Linear Range Analysis (LRA)
13. Furnace AA QC Analyses

1. <u>Instrument Calibration</u>

Guidelines for instrumental calibration are given in EPA 600/4-79-020 and/or Exhibit D. Instruments must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument standardization date and time must be included in the raw data.

For atomic absorption systems, calibration standards are prepared by diluting the stock metal solutions at the time of analysis. Date and time of preparation and analysis must be given in the raw data.

Calibration standards must be prepared fresh each time an analysis is to be made and discarded after use. Prepare a blank and at least three calibration standards in graduated amounts in the appropriate range.

One atomic absorption calibration standard must be at the CRDL except for mercury. The calibration standards must be prepared using the same type of acid or combination of acids and at the same concentration as will result in the samples following sample preparation.

Beginning with the blank, aspirate or inject the standards and record the readings. If the AA instrument configuration prevents the required 4-point calibration, calibrate according to instrument manufacturer's recommendations, and analyze the remaining required standards immediately after calibration. Results for these standards must be within ± 5% of the true value. Each standards concentration and the calculations to show that the ±5% criterion has been met, must be given in the raw data. If the values do not fall within this range, recalibration is necessary.

The ± 5% criteria does not apply to the atomic absorption calibration standard at the CRDL (CRA). Specific acceptance criteria will be set for this standard by EPA in the future. In the interim, the Contractor must observe and quantitate this standard at a level above their stated instrument detection limit during Atomic Absorption (AA) analysis. The CRDL standard must be reported on FORM II(PART 2)-IN.

Calibration standards for AA procedures must be prepared as described in Exhibit D.

Baseline correction is acceptable as long as it is performed after every sample or after the continuing calibration verification check. For cyanide and mercury, follow the calibration procedures outlined in Exhibit D. One cyanide calibration standard must be at the CRDL. For ICP systems, calibrate the instrument according to instrument manufacturer's recommended procedures. At least two standards must be used.

2. <u>Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)</u>

a. Initial Calibration Verification (ICV)

Immediately after each of the ICP, AA and cyanide systems have been calibrated, the accuracy of the initial calibration shall be verified and documented for every analyte by the analysis of EPA Initial Calibration Verification Solution(s) at each wavelength used for analysis. When measurements exceed the control limits of Table 1-Initial and Continuing Calibration Verification Control Limits for Inorganic Analyses, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

If the Initial Calibration Verification Solution(s) are not available from EPA, or where a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for

instrument calibration, but within the calibration range. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.

For ICP, the Initial Calibration Verification Solution(s) must be run at each wavelength used for analysis. For CN, the initial calibration verification standard must be distilled. The values for the initial and subsequent continuing calibration verifications shall be recorded on FORM II-IN for ICP, AA, and cyanide analyses, as indicated.

b. Continuing Calibration Verification (CCV)

To ensure calibration accuracy during each analysis run, one of the following standards is to be used for continuing calibration verification and must be be analyzed for every wavelength used for the analysis of each analyte, at a frequency of 10% or every 2 hours during an analysis run, whichever is more frequent. The standard must also be analyzed for every wavelength used for analysis at the beginning of the run and after the last analytical sample. The analyte concentrations in the continuing calibration standard must be one of the following solutions at or near the mid-range levels of the calibration curve:

1. EPA Solutions
2. NBS SRM 1643a
3. A Contractor-prepared standard solution

TABLE 1. INITIAL AND CONTINUING CALIBRATION VERIFICATION CONTROL LIMITS FOR INORGANIC ANALYSES

Analytical Method	Inorganic Species	% of True Value (EPA Set) Low Limit	High Limit
ICP/AA	Metals	90	110
Cold Vapor AA	Mercury	80	120
Other	Cyanide	85	115

The same continuing calibration standard must be used throughout the analysis runs for a Case of samples received.

If the deviation of the continuing calibration verification is greater than the control limits specified in Table 1-Initial and Continuing Calibration Verification Control Limits for Inorganic

Inorganic Analyses, the instrument must be recalibrated and the preceding 10 analytical samples or all analytical samples analyzed since the last good calibration verification must be performed for the analytes affected. Information regarding the continuing verification of calibration shall be recorded on FORM II-IN for ICP, AA and cyanide as indicated.

3. CRDL Standards for ICP (CRI)

To verify linearity near the CRDL for ICP analysis, the Contractor must analyze an ICP standard at two times the CRDL or two times the IDL, whichever is greater, at the beginning and end of each sample analysis run, or a minimum of twice per 8 hour working shift, whichever is more frequent, but not before Initial Calibration Verification. This standard must be run by ICP for every wavelength used for analysis, except those for Al, Ba, Ca, Fe, Mg, Na and K.

Specific acceptance criteria for the standard will be set by EPA in the future. In the interim, the Contractor must analyze and report these Standards on FORM II(PART 2)-IN.

4. Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses

a. Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Analyses

A calibration blank must analyzed at each wavelength used for analysis immediately after every initial and continuing calibration verification, at a frequency of 10% or every 2 hours during the run, whichever is more frequent. The blank must be analyzed at the beginning of the run and after the last analytical sample. The results for the calibration blanks shall be recorded on FORM III-IN for ICP, AA and cyanide analyses, as indicated. If the magnitude (absolute value) of the calibration blank result exceeds the IDL, the result must be so reported in ug/L on FORM III-IN, otherwise report as IDL-U. If the absolute value blank result exceeds the CRDL (Exhibit C), terminate analysis, correct the problem, recalibrate and reanalyze the preceding 10 analytical samples or all analytical samples analyzed since the last good calibration blank.

b. Preparation Blank (PB) Analysis

At least one preparation blank (or reagent blank), consisting of deionized distilled water processed through each sample preparation and analysis procedure (See Exhibit D, Section III), must be prepared and analyzed with every Sample Delivery Group, or with each batch[1] of samples digested, whichever is more frequent.

[1]A group of samples prepared at the same time.

The first batch of samples in an SDG is to be assigned to preparation blank one, the second batch of samples to preparation blank two, etc. (see FORM III-IN). Each data package must contain the results of all the preparation blank analyses associated with the samples in that SDG.

This blank is to be reported for each SDG and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner:

1) If the absolute value of the concentration of the blank is less than or equal to the Contract Required Detection Limit (Exhibit C), no correction of sample results is performed.

2) If any analyte concentration in the blank is above the CRDL, the lowest concentration of that analyte in the associated samples must be 10x the blank concentration. Otherwise, all samples associated with the blank with the analyte's concentration less than 10x the blank concentration and above the CRDL, must be redigested and reanalyzed for that analyte (except for an identified aqueous soil field blank). The sample concentration is not to be corrected for the blank value.

3) If the concentration of the blank is below the negative CRDL, then all samples reported below 10x CRDL associated with the blank must be redigested and reanalyzed.

The values for the preparation blank must be recorded in ug/L for aqueous samples and in mg/Kg for solid samples on FORM III-IN for ICP, AA, and cyanide analyses.

5. <u>ICP Interference Check Sample (ICS) Analysis</u>

To verify interelement and background correction factors, the Contractor must analyze and report the results for the ICP Interference Check Samples at the beginning and end of each analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent, but not before Initial Calibration Verification. The ICP Interference Check Samples must be obtained from EPA (EMSL/LV) if available and analyzed according to the instructions supplied with the ICS.

The Interference Check Samples consist of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively (starting with Solution A) for all wavelengths used for each analyte reported by ICP.

Results for the ICP analyses of Solution AB during the analytical runs must fall within the control limit of ±20% of the true value for the analytes included in the Interference Check Samples. If not, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the analytical samples analyzed since the last good ICS. If true values for analytes contained in the ICS and analyzed by ICP are

not supplied with the ICS, the mean must be determined by initially analyzing the ICS at least five times repetitively for the particular analytes. This mean determination must be made during an analytical run where the results for the previously supplied EPA ICS met all contract specifications. Additionally, the result of this initial mean determination is to be used as the true value for the lifetime of that solution (i.e., until the solution is exhausted).

If the ICP Interference Check Sample is not available from EPA, independant ICP Check Samples must be prepared with interferent and analyte concentrations at the levels specified in Table 2-Interferent and Analyte Elemental Concentrations Used for ICP Interference Check Sample. The mean value and standard deviation must be established by initially analyzing the Check Samples at least five times repetitively for each parameter on FORM IV-IN. Results must fall within the control limit of ±20% of the established mean value. The mean and standard deviation must be reported in the raw data. Results from the Interference Check Sample analyses must be recorded on FORM IV-IN for all ICP parameters.

TABLE 2. INTERFERENT AND ANALYTE ELEMENTAL CONCENTRATIONS USED FOR ICP INTERFERENCE CHECK SAMPLE

Analytes	(mg/L)	Interferents	(mg/L)
Ag	1.0	Al	500
Ba	0.5	Ca	500
Be	0.5	Fe	200
Cd	1.0	Mg	500
Co	0.5		
Cr	0.5		
Cu	0.5		
Mn	0.5		
Ni	1.0		
Pb	1.0		
V	0.5		
Zn	1.0		

6. Spike Sample Analysis (S)

The spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and measurement methodology. The spike is added before the digestion (i.e., prior to

the addition of other reagents) and prior to any distillation steps (i.e., CN-). At least one spike sample analysis must be performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) or for each Sample Delivery Group, whichever is more frequent.[2]

If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations must be performed using the results of the sample designated as the "original sample" (see section 7, Duplicate Sample Analysis). The average of the duplicate results cannot be used for the purpose of determining percent recovery. Samples identified as field blanks cannot be used for spiked sample analysis. EPA may require that a specific sample be used for the spike sample analysis.

The analyte spike must be added in the amount given in Table 3-Spiking Levels for Spike Sample Analysis, for each element analyzed. If two analytical methods are used to obtain the reported values for the same element within a Sample Delivery Group (i.e. ICP, GFAA), spike samples must be run by each method used.

If the spike recovery is not within the limits of 75-125%, the data of all samples received associated with that spike sample and determined by the same analytical method must be flagged with the letter "N" on FORMs I-IN and V-IN. An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the percent recovery does not meet the 75-125% recovery criteria.

For flame AA, ICP, Hg, and CN analyses, when the pre-digestion/pre-distillation spike recovery falls outside the control limits and the sample result does not exceed 4x the spike added, a post-digestion/post-distillation spike must be performed for those elements that do not meet the specified criteria (exception: Ag). Spike the unspiked aliquot of the sample at 2x the indigenous level or 2x CRDL, whichever is greater. Results of the post-digestion/post-distillation spike must be reported on FORM V(PART 2)-IN.

In the instance where there is more than one spike sample per matrix and concentration per method per SDG, if one spike sample recovery is not within contract criteria, flag all the samples of the same matrix, level, and method in the SDG. Individual component percent recoveries (%R) are calculated as follows:

$$\%\text{Recovery} = \frac{(SSR-SR)}{SA} \times 100$$

[2] EPA may require additional spike sample analysis, upon Project Officer request, for which the Contractor will be paid.

Where, SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

When sample concentration is less than the instrument detection limit, use SR = 0 only for purposes of calculating % Recovery. The spike sample results, sample results and % Recovery (positive or negative) must be reported on FORM V-IN for ICP, AA and cyanide analyses, as indicated.

The units for reporting spike sample results will be identical to those used for reporting sample results in FORM I-IN (i.e., ug/L for aqueous and mg/Kg dry weight basis for solid).

TABLE 3. SPIKING LEVELS[1] FOR SPIKE SAMPLE ANALYSIS

	For ICP/AA (ug/L)		For Furnace AA (ug/L)		Other[2] (ug/L)
Element	Water	Soil	Water	Soil	
Aluminum	2,000	*			
Antimony	500	500	100	100	
Arsenic	2,000	2,000	40	40	
Barium	2,000	2,000			
Beryllium	50	50			
Cadmium	50	50	5	5	
Calcium	*	*			
Chromium	200	200			
Cobalt	500	500			
Copper	250	250			
Iron	1,000	*			
Lead	500	500	20	20	
Magnesium	*	*			
Manganese	500	500			
Mercury					
Nickel	500	500			
Potassium	*	*			
Selenium	2,000	2,000	10	10	
Silver	50	50			
Sodium	*	*			
Thallium	2,000	2,000	50	50	
Vanadium	500	500			
Zinc	500	500			
Cyanide					100

NOTE: Elements without spike levels and not designated with an asterisk, must be spiked at appropriate levels.

[1]The levels shown indicate concentrations in the final digestate of the spiked sample (200 ml final volume).

[2]Spiking level reported is for both water and soil/sediment matrices.

*No spike required.

7. Duplicate Sample Analysis (2)

One duplicate sample must be analyzed from each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) or for each Sample Delivery Group, whichever is more frequent.[3] Duplicates cannot be averaged for reporting on FORM I-IN.

Duplicate sample analyses are required for percent solids. Samples identified as field blanks cannot be used for duplicate sample analysis. EPA may require that a specific sample be used for duplicate sample analysis. If two analytical methods are used to obtain the reported values for the same element for a Sample Delivery Group (i.e., ICP, GFAA), duplicate samples must be run by each method used.

The relative percent differences (RPD) for each component are calculated as follows:

$$RPD = \frac{|S - D|}{(S+D)/2} \times 100$$

Where, RPD = Relative Percent Difference
S = First Sample Value (original)
D = Second Sample Value (duplicate)

The results of the duplicate sample analyses must be reported on FORM VI-IN in ug/L for aqueous samples and mg/Kg dry weight basis for solid original and duplicate samples. A control limit of 20% for RPD shall be used for original and duplicate sample values greater than or equal to 5x CRDL (Exhibit C). A control limit of (±) the CRDL must be used for sample values less than 5x CRDL, and the absolute value of the control limit (CRDL) must be entered in the "Control Limit" column on FORM VI-IN.

If one result is above the 5x CRDL level and the other is below, use the ± CRDL criteria. If both sample values are less than the IDL, the RPD is not calculated on FORM VI-IN. For solid sample or duplicate results < 5x CRDL, enter the absolute value of the CRDL, corrected for sample weight and percent solids, in the "Control Limit" column.

If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an "*" on FORMs I-IN and VI-IN. In the instance where there is more than one duplicate sample per SDG, if one duplicate result is not within contract criteria, flag all samples of the same matrix, concentration, and method in the SDG. The percent difference data will be used by EPA to evaluate the long-term precision of the methods for each parameter. Specific control limits for each element will be added to FORM VI-IN at a later date based on these precision results.

[3] EPA may require additional duplicate sample analyses, upon Project Officer request, for which the Contractor will be paid.

8. Laboratory Control Sample (LCS) Analysis

Aqueous and solid Laboratory Control Samples (LCS) must be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the EPA samples received. The aqueous LCS solution must be obtained from EPA (if unavailable, the Initial Calibration Verification Solutions may be used). One aqueous LCS must be prepared and analyzed for every group of aqueous samples in a Sample Delivery Group, or for each batch of aqueous samples digested, whichever is more frequent. An aqueous LCS is not required for mercury and cyanide analysis.

The EPA-provided solid LCS must be prepared and analyzed using each of the procedures applied to the solid samples received (exception: percent solids determination not required). If the EPA solid LCS is unavailable, other EPA Quality Assurance Check samples or other certified materials may be used. One solid LCS must be prepared and analyzed for every group of solid samples in a Sample Delivery Group, or for each batch of samples digested, whichever is more frequent.

All LCS results and percent recovery (%R) will be reported on FORM VII-IN. If the percent recovery for the aqueous LCS falls outside the control limits of 80-120% (exception: Ag and Sb), the analyses must be terminated, the problem corrected, and the previous samples associated with that LCS redigested and reanalyzed.

If the results for the solid LCS fall outside the control limits established by EPA, the analyses must be terminated, the problem corrected, and the previous samples associated with that LCS redigested and reanalyzed.

9. ICP Serial Dilution Analysis (L)

Prior to reporting concentration data for the analyte elements, the Contractor must analyze and report the results of the ICP Serial Dilution Analysis. The ICP Serial Dilution Analysis must be performed on each group of samples of a similar matrix type (i.e., water, soil and concentration (i.e., low, medium) or for each Sample Delivery Group, whichever is more frequent. Samples identified as field blanks cannot be used for Serial Dilution Analysis.

If the analyte concentration is sufficiently high (minimally a factor of 50 above the instrumental detection limit in the original sample), an analysis of a 5-fold dilution must agree within 10% of the original determination. If the dilution analysis for one or more analytes is not within 10%, a chemical or physical interference effect must be suspected, and the data for all affected analytes in the samples received associated with that serial dilution must be flagged with an "E" on FORM IX-IN and FORM I-IN.

The percent differences for each component are calculated as follows:

$$\% \text{ Difference} = \frac{|I - S|}{I} \times 100$$

where, I = Initial Sample Result
S = Serial Dilution Result (Instrument Reading x 5)

In the instance where there is more than one serial dilution per SDG, if one serial dilution result is not within contract criteria, flag all the samples of the same matrix and concentration in the Sample Delivery Group. Serial dilution results and "E" flags must be reported on FORM IX-IN.

10. Instrument Detection Limit (IDL) Determination

Before any field samples are analyzed under this contract, the instrument detection limits (in ug/L) must be determined for each instrument used, within 30 days of the start of contract analyses and at least quarterly (every 3 calendar months), and must meet the levels specified in Exhibit C.

The Instrument Detection Limits (in ug/L) shall be determined by multiplying by 3, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x-5x IDL, with seven consecutive measurements per day. 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 normally performed between the analysis of separate samples). IDL's must be determined and reported for each wavelength used in the analysis of the samples.

IDLs must be reported for each instrument used on FORM XI-IN submitted with each data package. If multiple AA instruments are used for the analysis of an element within a Sample Delivery Group, the highest IDL for the AAs must be used for reporting concentration values for that Sample Delivery Group. The same reporting procedure must be used for multiple ICPs.

11. Interelement Corrections for ICP

Before any field samples are analyzed under this contract, the ICP interelement correction factors must be determined within six months of the start of contract analyses and at least annually thereafter. Correction factors for spectral interference due to Al, Ca, Fe, and Mg must be determined at all wavelengths used for each analyte reported by ICP. Correction factors for spectral interference due to analytes other than Al Ca, Fe, and Mg must be reported if they were applied. Results from interelement correction factors determination must be reported on FORM XII(PART 1)-IN and FORM XII(PART 2)-IN for all ICP parameters.

12. Linear Range Analysis (LRA)

For ICP analysis, a linear range verification check standard must be analyzed and reported quarterly (every 3 calendar months) for each element on FORM XII-IN. The standard must be analyzed during a routine analytical run performed under this contract. The analytically determined concentration of this standard must be within ± 5% of the true value. This concentration is the upper limit of the ICP linear range beyond which results cannot be reported under this contract without dilution of the analytical sample.

13. Furnace Atomic Absorption (AA) QC Analyses

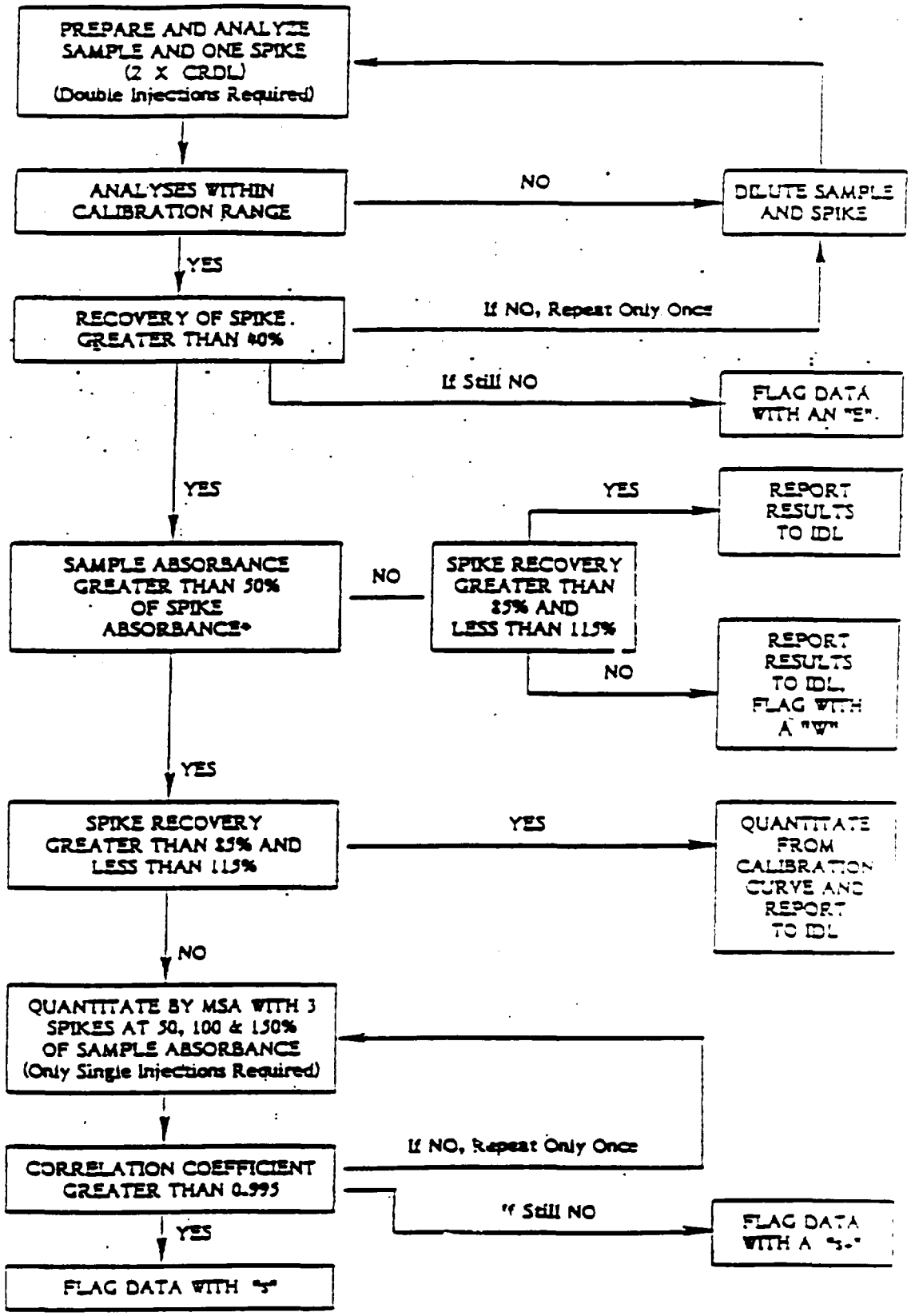
Because of the nature of the Furnace AA technique, the special procedures summarized in Figure 1-Furnace AA Analysis Scheme will be required for quantitation. (These procedures do not replace those in Exhibit D of this SOW, but supplement the guidance provided therein.)

a. All furnace analyses, except during Full Method of Standard Addition (MSA), will require duplicate injections for which the average absorbance or "concentration" will be reported. All analyses must fall within the calibration range. The raw data package must contain absorbance or "concentration" values for both injections, the average value and the relative standard deviation (RSD) or coefficient of variation (CV). For concentrations greater than CRDL, the duplicate injection readings must agree within 20% RSD or CV, or the sample must be rerun once (i.e., two additional burns). If the readings are still out, flag the value with an "M" on FORM I-IN.

   If the preparation blank analytical spike recovery is out of control (85-115%), the spiking solution must be verified by respiking and rerunning the preparation blank once. If the preparation blank analytical spike recovery is still out of control, correct the problem and reanalyze all analytical samples associated with that blank.

b. All furnace analyses for each analytical sample will require at least an analytical spike to determine if the MSA will be required for quantitation. This requirement for an analytical spike will include the LCS and the preparation blank. (The LCS must be quantitated from the calibration curve and corrective action, if needed, taken accordingly. MSA is not to be performed on the LCS or preparation blank, regardless of spike recovery results.) An analytical spike is not required on the pre-digestion spike sample

Figure 1.
FURNACE ATOMIC ABSORPTION ANALYSIS SCHEME



*Spike absorbance defined as (absorbance of spike sample) minus (absorbance of the sample..

when the pre-digestion spike sample recovery is within the specified control limits of 75-125% or when the sample concentration is greater than 4x the pre-digestion spike concentration. The spike[4] will be required to be at a concentration (in the sample) 2x CRDL.

The analytical spike of a sample must be run immediately after that sample. The percent recovery (%R) of the spike, calculated by the same formula as Spike Sample Analyses (see item 6, this section), will then determine how the sample will be quantitated, as follows:

1) If the spike recovery is less than 40%, the sample must be diluted and rerun with another spike. Dilute the sample by a factor of 5 to 10 and rerun. This step must only be performed once. If after the dilution the spike recovery is still <40%, report data and flag with an "E" to indicate interference problems.

2) If the spike recovery is greater than 40% and the sample absorbance or concentration is <50% of the "spike"[5], report the sample results to the IDL. If the spike recovery is less than 85% or greater than 115%, flag the result with a "W".

3) If the sample absorbance or concentration is >50% of the spike and the spike recovery is between 85% and 115%, the sample must be quantitated directly from the calibration curve and reported to the IDL.

4) If the sample absorbance or concentration is >50% of the spike and the spike recovery is less than 85% or greater than 115%, the sample must be quantitated by MSA.

c. The following procedures will be incorporated into MSA analyses.

1) Data from MSA calculations must be within the linear range as determined by the calibration curve generated at the beginning of the analytical run.

2) The sample and three spikes must be analyzed consecutively for MSA quantitation (the "initial" spike run data is specifically excluded from use in the MSA quantitation). Only single injections are required for MSA quantitation.

---

[4]Spikes are post-digestion spikes to be prepared prior to analysis by adding a known quantity of the analyte to an aliquot of the digested sample. The unspiked sample aliquot must be compensated for any volume change in the spike samples by addition of deionized water to the unspiked sample aliquot. The volume of the spiking solution added must not exceed 10% of the analytical sample volume.

[5]"Spike" is defined as [absorbance or concentration of spike sample] minus [absorbance or concentration of the sample].

Each full MSA counts as two analytical samples towards determining 10% QC frequency (i.e., five full MSAs can be performed between calibration verifications).

3) For analytical runs containing only MSAs, single injections can be used for QC samples during that run. For instruments that operate in an MSA mode only, MSA can be used to determine QC samples during that run.

4) Spikes must be prepared such that:

   a) Spike 1 is approximately 50% of the sample absorbance.

   b) Spike 2 is approximately 100% of the sample absorbance.

   c) Spike 3 is approximately 150% of the sample absorbance.

5) The data for each MSA analysis must be clearly identified in the raw data documentation (plotting added concentration on the x-axis and absorbance or found concentration on the y- axis) along with the slope, x-intercept, y-intercept and correlation coefficient (r) for the least squares fit of the data. The results must be reported on FORM VIII-IN. Reported values obtained by MSA must be flagged on the data sheet (FORM I-IN) with the letter "S" if the correlation coefficient is greater than or equal to 0.995.

6) If the correlation coefficient (r) for a particular analysis is less than 0.995, the MSA analysis must be repeated once. If the correlation coefficient is still less than 0.995, report the results on FORM I-IN from the run with the best "r" and flag the result with a "+" on FORM VIII-IN and FORM I-IN.

EXAMPLES OF PE 5100 WINDOWS

FIGURE 1

Element Parameter - C:\AA_USER\5100\ELEMENT\PB.GEL

Main | Calib | Inst | HGA | AS-60/1 | AS-60/2 | AS-60/3

Element: Pb Analyst: MP

PRINTER: ☐ Means Only ☒ Main Data ☐ Main+Suppl.

OPTIONS: ☒ Calib. Curves ☒ Peak Plots ☒ Sampler Volumes

PEAK STORAGE: ☒ None ☐ All ☐ 1 Repl./Sample

REMARKS:
STD PREP DATE:
STD PREP TIME:
ANALYST:
PROJECT: MATRIX:
CASE/SAS: INST: Z5100-A

FIGURE 2

Element Parameter - C:\AA_USER\5100\ELEMENT\PB.GEL

Main | Calib | Inst | HGA | AS-60/1 | AS-60/2 | AS-60/3

1 CALIBRATION TYPE: ☐ Linear ☐ Meth. of Add. ▶ Nonlinear ☐ Meth. of Add. Calib.

2 STANDARD UNITS: ☐ g/L ☐ mg/L ▶ ug/L ☐ ng/L ☐ pg/L ☐ ____ : Factor _____

3 STANDARD CONC.: S1: 10.0____ S2: 50.0____ S3: 100.0____ S4: _____
S5: _____ S6: _____ S7: _____ S8: _____ Rslp: _____

4 SAMPLE UNITS: ☐ g/L ☐ mg/L ▶ ug/L ☐ ng/L ☐ pg/L ☐ mg/g ☐ ug/g
☐ ng/g ☐ wt% ☐ ____ : ▶ Wt/Vol ☐ Wt/Wt : Factor _____

5 REPLICATES: ☐ 1 ☐ 3 ☐ 5 ▶ 2_ (1-99)

☐ if conc < _____ use __ if between _____ & _____ use __
if conc > 100.0____ use 1_ else use 2_ (1-99)

FIGURE 3

Element Parameter - C:\AA_USER\5100\ELEMENT\PB.GEL

Main | Calib | Inst | HGA | AS-60/1 | AS-60/2 | AS-60/3

INTEGRATION TIME (sec): ☐ 1 ☐ 3 ▶ 5 ☐ 10 ☐ ___ (1-60)

READ DELAY (sec): ▶ 0 ☐ 2 ☐ 5 ☐ ___ (0-29)

WAVELENGTH (nm): ▶ 283.3 ☐ ___ (190-860)

☐ Slew ▶ Peak Rollover (Abs.): ___

SLIT (nm): ☐ 0.02 ☐ 0.07 ☐ 0.20 ▶ 0.70 ☐ 2.00

BOC TIME (sec): ☐ 1 ☐ 2 ☐ 3 ▶ 4 ☐ 5

MEASUREMENT TYPE: ☐ Peak Area ▶ Peak Height

PEAK HEIGHT SMOOTHING (points): ☐ None ☐ 5 ☐ 9 ☐ 19 ▶ 37

FIGURE 4

Element Parameter - C:\AA_USER\5100\ELEMENT\PB.GEL

Main | Calib | Inst | HGA | AS-60/1 | AS-60/2 | AS-60/3

Sample Volume: 20
Diluent Volume: _ Diluent Location: _

MATRIX MODIFIER PARAMETERS:
Modifier #1 Location: _ Modifier #1 Volume: _
☐ Pipet Before Sample ▶ Pipet With Sample ☐ Pipet After Sample

Modifier #2 Location: _ Modifier #2 Volume: _
☐ Pipet Before Sample ▶ Pipet With Sample ☐ Pipet After Sample

PIPET PARAMETERS:
Protocol: ▶ Pipet Together ☐ Pipet Separately
Number of Wash Steps: ▶ 1 ☐ 2 ☐ 3
Number of Injections: ▶ 1 ☐ 3 ☐ 5 ☐ _ (1-10)

FIGURE 5

Element Parameter - C:\AA_USER\5100\ELEMENT\PB.GEL

Main | Calib | Inst | HGA | AS-60/1 | AS-60/2 | AS-60/3

REFERENCE STANDARDS PARAMETERS:

Solutions	Location	Volume	Diluent Volume	Modifier #1 Volume	Modifier #2 Volume
Std. Blank	36	20			
Standard 1	37	20			
Standard 2	38	20			
Standard 3	39	20			
Standard 4					
Standard 5					
Standard 6					
Standard 7					
Standard 8					
Reslope					

Sample Blank Location: __

Additions Definition: ☑ Automated Additions ☐ Premixed Additions

FIGURE 6

```
Element Parameter - C:\AA_USER\5100\ELEMENT\PB.GEL

Main | Calib | Inst | HGA | AS-60/1 | AS-60/2 | AS-60/3

SAMPLE BEYOND CALIBRATION RANGE:
  Beyond Calibration Action:  [▶] Continue   [ ] Dilute & Reanalyze
  Alternate Sample Volume: __   Separate Overcal Blank:  [ ] Yes  [▶] No

RECOVERY MEASUREMENT PARAMETERS:
 Use __ uL of the _________ ug/L  standard at location __
 The net recovery concentration will be            ug/L

PERIODIC RECALIBRATION PARAMETERS:
  Recalibration Type:  [▶] Restandardize  [ ] Reslope
  Run Before Sample Locations:  __  __  __  __  __  __

PERIODIC CHECK SAMPLE PARAMETERS:
  Location: __      Lower Limit: ________      Upper Limit: ________
  Run Before Sample Locations:  __  __  __  __  __  __
  Out of Limit Action:  [▶] Continue   [ ] Recalibrate & Continue
                        [ ] Stop       [ ] Recalibrate & Rerun
```

FIGURE 7

ID/Weight Parameter - C:\AA_USER\5100\IDWT\INITSTD.A60

Analyst: _____ Samp. Vol.: 100_____ Nominal Wt.: 1.0____

Weight Units: [ ] g [ ] mg [▶] ug [ ] : _____ Factor: _____
Volume Units: [▶] L [ ] mL [ ] uL [ ] : _____ Factor: _____
Autosampler : [ ] Manual [ ] AS-51 [▶] AS-60

Position	Sample ID	Weight	Dilution
1	CRA	1	1
2	ICV	1	1
3	ICB	1	1
4	CCV 1	1	1
5	CCB 1	1	1
6			
7			

FIGURE 8

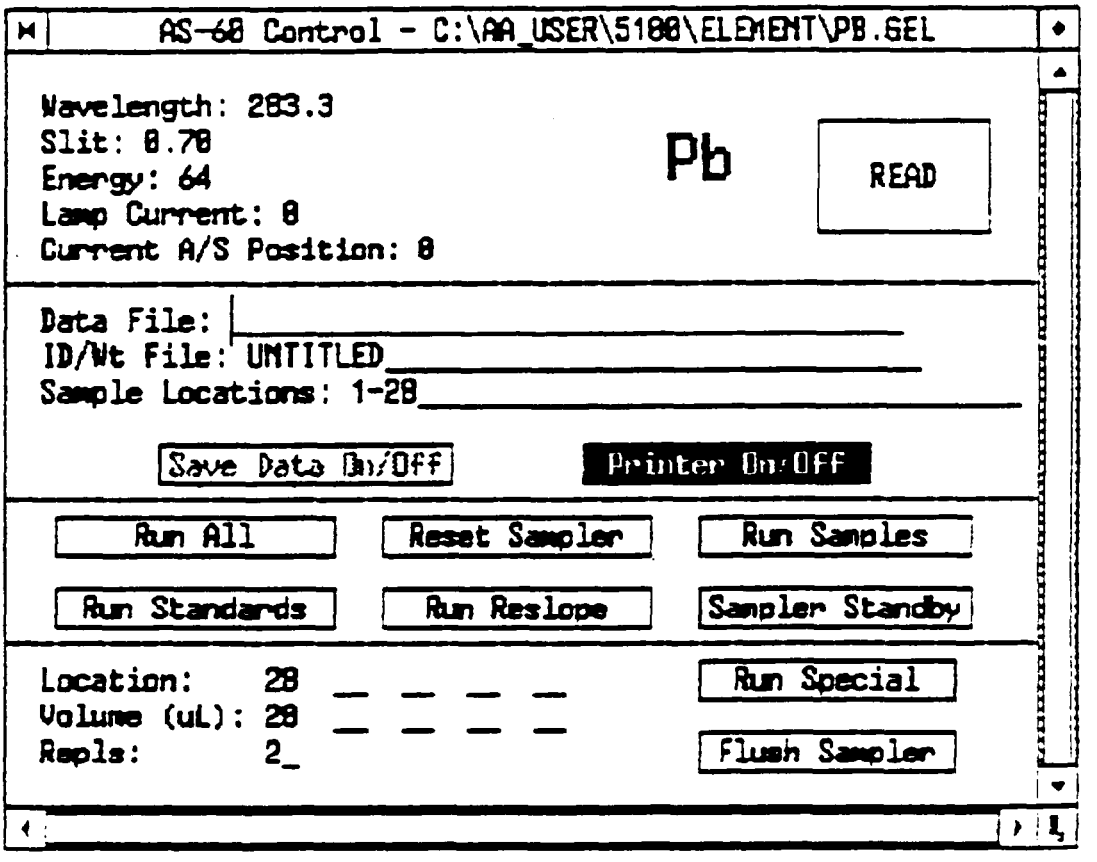

AS-68 Control - C:\AA_USER\5100\ELEMENT\PB.GEL
Wavelength: 283.3
Slit: 0.70
Energy: 64
Lamp Current: 0
Current A/S Position: 0
Pb
READ
Data File:
ID/Wt File: UNTITLED
Sample Locations: 1-20
Save Data On/Off
Printer On/Off
Run All
Reset Sampler
Run Samples
Run Standards
Run Reslope
Sampler Standby
Location: 20
Volume (uL): 20
Repls: 2
Run Special
Flush Sampler

FIGURE 9

Element Parameter - C:\AA_USER\5100\ELEMENT\AS.GEL

Main | Calib | Inst | HGA | AS-60/1 | AS-60/2 | AS-60/3

Step Number	Furnace Temp.	Time (sec) Ramp	Time (sec) Hold	Internal Flow	Gas Type	Read	Prelim/ Repeat
1	200	1	25	300	N A		P R
2	1300	10	10	300	N A		P R
3	20	1	5	300	N A		P R
4	2300	1	5	0	N A	▶	P R
5	2600	1	5	300	N A		P R
6		1	5	300	N A		P R
7		1	5	300	N A		P R
8		1	5	300	N A		P R
9		1	5	300	N A		P R
10		1	5	300	N A		P R
11		1	5	300	N A		P R
12		1	5	300	N A		P R

FIGURE 10

Element Parameter - C:\AA_USER\5100\ELEMENT\SE.GEL

Main | Calib | Inst | HGA | AS-60/1 | AS-60/2 | AS-60/3

Step Number	Furnace Temp.	Time (sec) Ramp	Time (sec) Hold	Internal Flow	Gas Type				Read	Prelim/ Repeat			
1	200	1	25	300	▶	M		A			P		R
2	900	5	15	300	▶	M		A			P		R
3	20	1	5	300	▶	M		A			P		R
4	2200	0	5	0	▶	M		A	▶		P		R
5	2600	1	5	300	▶	M		A			P		R
6		1	2	300	▶	M		A			P		R
7		1	5	300	▶	M		A			P		R
8		1	5	300	▶	M		A			P		R
9		1	5	300	▶	M		A			P		R
10		1	5	300	▶	M		A			P		R
11		1	5	300	▶	M		A			P		R
12		1	5	300	▶	M		A			P		R

FIGURE 11

Element Parameter - C:\AA_USER\5100\ELEMENT\TL.GEL

Main | Calib | Inst | HGA | AS-60/1 | AS-60/2 | AS-60/3

Step Number	Furnace Temp.	Time (sec) Ramp	Time (sec) Hold	Internal Flow	Gas Type	Read	Prelim/ Repeat
1	200	1	25	300	N A		P R
2	600	5	15	300	N A		P R
3	20	1	5	300	N A		P R
4	2200	0	5	0	N A	▶	P R
5	2600	1	5	300	N A		P R
6		1	5	300	N A		P R
7		1	5	300	N A		P R
8		1	5	300	N A		P R
9		1	5	300	N A		P R
10		1	5	300	N A		P R
11		1	5	300	N A		P R
12		1	5	300	N A		P R

FIGURE 12

Element Parameter - C:\AA_USER\5100\ELEMENT\PB.GEL

Main | Calib | Inst | HGA | AS-60/1 | AS-60/2 | AS-60/3

Step Number	Furnace Temp.	Time (sec) Ramp	Time (sec) Hold	Internal Flow	Gas Type				Read	Prelim/ Repeat			
1	200	1	25	300		N	▶	A			P		R
2	500	5	15	300		N	▶	A			P		R
3	20	1	5	300		N	▶	A			P		R
4	1800	8	5	0		N	▶	A	▶		P		R
5	2600	1	4	300		N	▶	A			P		R
6		1	2	300		N	▶	A			P		R
7		1	5	300	▶	N		A			P		R
8		1	5	300	▶	N		A			P		R
9		1	5	300	▶	N		A			P		R
10		1	5	300	▶	N		A			P		R
11		1	5	300	▶	N		A			P		R
12		1	5	300	▶	N		A			P		R

FIGURE 13

Element Parameter - C:\AA_USER\5100\ELEMENT\SB.GEL

Main | Calib | Inst | HGA | AS-60/1 | AS-60/2 | AS-60/3

Step Number	Furnace Temp.	Time (sec) Ramp	Time (sec) Hold	Internal Flow	Gas Type		Read	Prelin/ Repeat	
1	200	1	25	300	▶ N	A		P	R
2	1100	10	15	300	▶ N	A		P	R
3	20	1	5	300	▶ N	A		P	R
4	2300	0	5	0	▶ N	A	▶	P	R
5	2600	1	5	300	▶ N	A		P	R
6	20	1	2	300	▶ N	A		P	R
7		1	5	300	▶ N	A		P	R
8		1	5	300	▶ N	A		P	R
9		1	5	300	▶ N	A		P	R
10		1	5	300	▶ N	A		P	R
11		1	5	300	▶ N	A		P	R
12		1	5	300	▶ N	A		P	R

AA, FURNACE, AND AUTOSAMPLER SETTINGS
PE 2380

HGA APPLICATIONS DATA SHEET

June, 1993

Ensecc

Element: Arsenic Matrix: 100 ul 2% Ni

HGA-500 Program Location: NA

Instrumental Parameters

Inst. Model : 2380

Wavelength : 193.7

Spectral Bandwidth: 0.7 nm (alt)

Light Source : EDL

Current/Watts : 6 (W/D2)

Background Correction? D2

Readout Parameters

Absorbance/Conc: 150 abs for 50 ppb

[X] Peak Height; time 5.0

[ ] Peak Area; time

[X] Recorder: printer

HGA Parameters

HGA Model : 400

Graphite Tube : Uncoated

Sample Aliquot: 25 ul

Sample Introduction

Manual:

Automated: X

Replicates: 2

Purge Gas : Argon

Alternate Gas :

Keyboard Entries

Step	1	2	3	4	5	6	7	8	9
Temp °C	130	1100	2300	2600					
Ramp (s)	5	10	0	1					
Hold (s)	15	10	5	2					
Read			X						
Rec.									
Baseline									
Int.Flow ml/min			Stop						
Int.Alt. ml/min									
Ext.Alt. ml/min									

June, 1989 Ense

HGA APPLICATIONS DATA SHEET

Element: Selenium Matrix: 2% Ni in 5% HNO_3 (100 ul per 10 ml sample) as a matrix modifier

HGA-500 Program Location: _____

Instrumental Parameters

Inst. Model : 2380

Wavelength : 196.0 nm

Spectral Bandwidth: 0.7 nm ALT

Light Source : EDL

Current/Watts : 6

Background Correction? D_2 (Deuterium)

Readout Parameters

Absorbance/Conc: .160 for 50.0 ug/L std.

[X] Peak Height; time 5.0

[ ] Peak Area; time _____

[X] Recorder: printer

HGA Parameters

HGA Model : 400

Graphite Tube : pyro/pla

Sample Aliquot: 20 ul

Sample Introduction

Manual: _____

Automated: X

Replicates: 2

Purge Gas : Argon

Alternate Gas : _____

Keyboard Entries

Step	1	2	3	4	5	6	7	8	9
Temp °C	140	900	20	2200	2600	20			
Ramp (s)	1	5	1	0	1	1			
Hold (s)	20	10	5	5	3	2			
Read				X					
Rec.									
Baseline			0						
Int.Flow ml/min				Stop					
Int.Alt. ml/min									
Ext.Alt. ml/min									

HGA APPLICATIONS DATA SHEET

Element: Thallium Matrix: 50% ul 5% Ni in 10 ml aliquot

HGA-500 Program Location: _____

Instrumental Parameters

Inst. Model : 2380

Wavelength : 276.8 nm

Spectral Bandwidth: 0.7 nm (alt)

Light Source : EDL

Current/Watts : 6 watts (w/D2)

Background Correction? D2

Readout Parameters

Absorbance/Conc: .150 abs for 50 ppb

[X] Peak Height; time 5.0

[ ] Peak Area; time _____

[X] Recorder: printer

HGA Parameters

EGA Model : 400

Graphite Tube : Uncoated

Sample Aliquot: 20 ul

Sample Introduction

Manual: _____

Automated: X

Replicates: 2

Purge Gas : Argon

Alternate Gas : _____

Keyboard Entries

Step	1	2	3	4	5	6	7	8	9
Temp °C	120	500	2300	2500					
Ramp (s)	5	5	0	1					
Hold (s)	15	15	5	2					
Read			X						
Rec.									
Baseline									
Int.Flow ml/min			Stop						
Int.Alt. ml/min									
Ext.Alt. ml/min									

HGA APPLICATIONS DATA SHEET

Element: Lead Matrix: 50 ul 5% Ni

HGA-500 Program Location: _____

Instrumental Parameters

Inst. Model : 2380

Wavelength : 283.3 nm

Spectral Bandwidth: 0.7 nm

Light Source : EDL

Current/Watts : 8 (w/D2)

Background Correction? D2/yes

Readout Parameters

Absorbance/Conc: .150 abs for 50 ppb

[X] Peak Height; time 5.0

[ ] Peak Area; time _____

[X] Recorder: printer

HGA Parameters

HGA Model : 400

Graphite Tube : Uncoated

Sample Aliquot: 20 ul

Sample Introduction

Manual: _____

Automated: X

Replicates: 2

Purge Gas : Argon

Alternate Gas : _____

Keyboard Entries

Step	1	2	3	4	5	6	7	8	9
Temp °C	120	500	2300	2600					
Ramp (s)	5	5	0	1					
Hold (s)	15	15	5	2					
Read			X						
Rec.									
Baseline									
Int.Flow ml/min			Stop						
Int.Alt. ml/min									
Ext.Alt. ml/min									

June, 1988 Ensecc

HGA APPLICATIONS DATA SHEET

Element: Cadmium Matrix: 1% HNO_3

HGA-500 Program Location: _____

Instrumental Parameters

Inst. Model : 2380

Wavelength : 228.8 nm

Spectral Bandwidth: 0.7 nm Alt

Light Source : HCI / EDL

Current/Watts : 5 ma / 5 watts

Background Correction? D_2

Readout Parameters

Absorbance/Conc: 0.042/1.0 ug/L

[X] Peak Height; time 5 sec.

[ ] Peak Area; time _____

[X] Recorder: printer

HGA Parameters

HGA Model : 400

Graphite Tube : Uncoated

Sample Aliquot: 20 ul

Sample Introduction

Manual: _____

Automated: X

Replicates: _____

Purge Gas : Ar

Alternate Gas : None

Keyboard Entries

Step	1	2	3	4	5	6	7	8	9
Temp °C	130	500	1800	2400					
Ramp (s)	5	10	0	1					
Hold (s)	15	10	5	3					
Read			X						
Rec.									
Baseline									
Int.Flow ml/min			Stop						
Int.Alt. ml/min									
Ext.Alt. ml/min									

AA, FURNACE AND AUTOSAMPLER SETTINGS
PE 5000

HGA APPLICATIONS DATA SHEET

Element: Arsenic Matrix: 2% Ni, 5% HNO_3

HGA-500 Program Location: _____

Instrumental Parameters

Inst. Model : Z5000

Wavelength : 193.7 nm

Spectral Bandwidth: 0.7 nm

Light Source : EDL

Current/Watts : 6 watts

Background Correction? Zeeman

Readout Parameters

Absorbance/Conc: .150 for 50 ug/L

[ ] Peak Height; time _____

[X] Peak Area; time 5.0 sec

[ ] Recorder: _____

HGA Parameters

HGA Model : 500

Graphite Tube : pyro/plat

Sample Aliquot: 25 ul

Sample Introduction

Manual: _____

Automated: X

Replicates: 2

Purge Gas : Argon

Alternate Gas : _____

Keyboard Entries

Step	1	2	3	4	5	6	7	8	9
Temp °C	140	1100	20	2300	2600				
Ramp (s)	1	10	1	1	1				
Hold (s)	25	10	5	5	3				
Read				X					
Rec.				X					
Baseline			0						
Int.Flow ml/min				0					
Int.Alt. ml/min									
Ext.Alt. ml/min									

INTERFERENCES

REAGENTS

STANDARD SOLUTIONS

SAMPLE PREPARATION

ANALYSIS

CALCULATION

NOTES

Determination performed by: _____ Date: _____

HGA APPLICATIONS DATA SHEET

Element: Selenium Matrix: 2% Ni, 5% HNO_3

HGA-500 Program Location: _____

Instrumental Parameters

Inst. Model : Z5000

Wavelength : 196.0 nm

Spectral Bandwidth: 0.7 nm

Light Source : EDL

Current/Watts : 4-5 watts

Background Correction? Zeeman

Readout Parameters

Absorbance/Conc: .150 for ug/L std

[X] Peak Height; time 5.0 sec

[ ] Peak Area; time _____

[X] Recorder: printer

HGA Parameters

HGA Model : 500

Graphite Tube : pyro/plat

Sample Aliquot: 20 ul

Sample Introduction

Manual: _____

Automated: X

Replicates: 2

Purge Gas : Argon

Alternate Gas : _____

Keyboard Entries

Step	1	2	3	4	5	6	7	8	9
Temp °C	140	900	20	2200	2600				
Ramp (s)	1	5	1	0	1				
Hold (s)	20	10	5	5	3				
Read				X					
Rec.				X					
Baseline			0						
Int.Flow ml/min				0					
Int.Alt. ml/min									
Ext.Alt. ml/min									

INTERFERENCES

REAGENTS

STANDARD SOLUTIONS

SAMPLE PREPARATION

ANALYSIS

CALCULATION

NOTES

Determination performed by: _____ Date: _____

HGA APPLICATIONS DATA SHEET

Element: Thallium Matrix: 2% Ni, 5% HNO_3

HGA-500 Program Location: _____

Instrumental Parameters

Inst. Model : Z5000

Wavelength : 283.3 nm

Spectral Bandwidth: 0.7 nm

Light Source : EDL

Current/Watts : _____

Background Correction? _____

Readout Parameters

Absorbance/Conc: .150 50 ug/L

[X] Peak Height; time 5.0 sec

[ ] Peak Area; time _____

[X] Recorder: printer

HGA Parameters

HGA Model : 500

Graphite Tube : pyro/plat

Sample Aliquot: 25 ul

Sample Introduction

Manual: _____

Automated: X

Replicates: 2

Purge Gas : Argon

Alternate Gas : _____

Keyboard Entries

Step	1	2	3	4	5	6	7	8	9
Temp °C	140	500	2300	2600					
Ramp (s)	1	5	0	1					
Hold (s)	25	10	5	3					
Read			X						
Rec.			X						
Baseline		9							
Int.Flow ml/min			0						
Int.Alt. ml/min									
Ext.Alt. ml/min									

INTERFERENCES

REAGENTS

STANDARD SOLUTIONS

SAMPLE PREPARATION

ANALYSIS

CALCULATION

NOTES

Determination performed by: _____ Date: _____

July 1989

HGA APPLICATIONS DATA SHEET

Element: Lead Matrix: 2% Ni, 5% HNO3

HGA-500 Program Location: _____

Instrumental Parameters

Inst. Model : Z5000

Wavelength : 283.3 nm

Spectral Bandwidth: 0.7 nm

Light Source : EDL

Current/Watts : _____

Background Correction? _____

Readout Parameters

Absorbance/Conc: .150 for 50 ug/L

[X] Peak Height; time _____

[ ] Peak Area; time _____

[X] Recorder: printer

HGA Parameters

HGA Model : 500

Graphite Tube : pyro/plat

Sample Aliquot: 25 ul

Sample Introduction

Manual: _____

Automated: X

Replicates: 2

Purge Gas : Argon

Alternate Gas : _____

Keyboard Entries

Step	1	2	3	4	5	6	7	8	9
Temp °C	140	500	2300	2600					
Ramp (s)	1	5	0	1					
Hold (s)	25	10	5	3					
Read			X						
Rec.			X						
Baseline		9							
Int.Flow ml/min			0						
Int.Alt. ml/min									
Ext.Alt. ml/min									

INTERFERENCES

REAGENTS

STANDARD SOLUTIONS

SAMPLE PREPARATION

ANALYSIS

CALCULATION

NOTES

Determination performed by: _____ Date: _____

HGA APPLICATIONS DATA SHEET

Element: Antimony Matrix: 2% HNO_3

HGA-600 Program Location: _____

Instrumental Parameters

Inst. Model : 5000

Wavelength : 217.6

Spectral Bandwidth: 0.2 nm (Low)

Light Source : EDL

Current/Watts : 8 watts

Background Correction? Zeeman

Readout Parameters

Absorbance/Conc: .150/50 ug/L

[X] Peak Height; time 5 sec.

[ ] Peak Area; time _____

[X] Recorder: printer

HGA Parameters

HGA Model : 600

Graphite Tube : pyrolplat-form

Sample Aliquot: 25 ul

Sample Introduction

Manual: _____

Automated: X

Replicates: 2

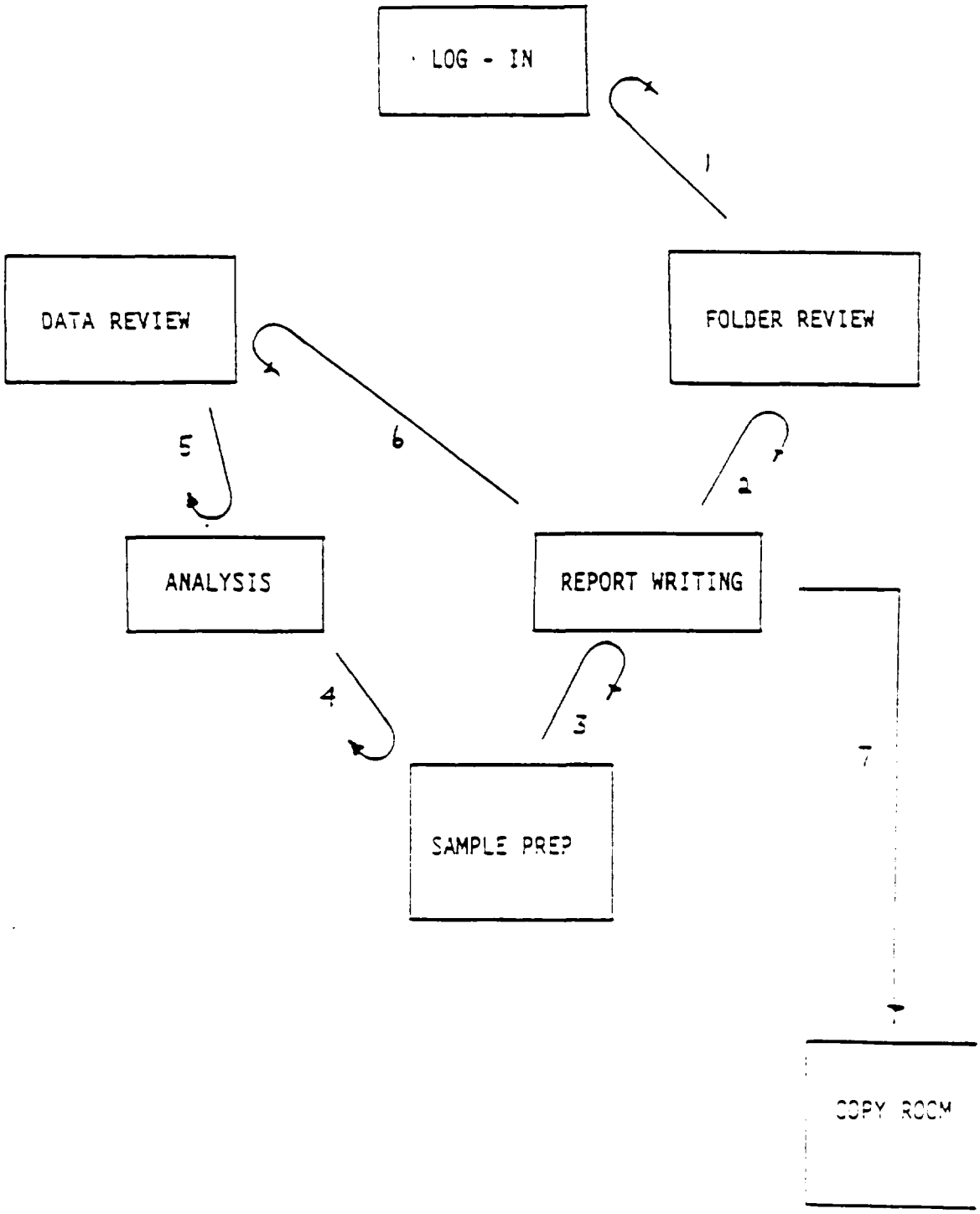
Purge Gas : Ar

Alternate Gas : NA

Keyboard Entries

Step	1	2	3	4	5	6	7	8	9
Temp °C	140	1200	20	2300	2600				
Ramp (s)	1	10	1	0	1				
Hold (s)	25	10	5	5	2				
Read				X					
Rec.				X					
Baseline			0						
Int.Flow ml/min				0					
Int.Alt. ml/min									
Ext.Alt. ml/min									

METALS/CN KANBAN



APPENDIX B-11

LANDFILL GAS VOCS (ENSECO)

Enseco - Air Toxics Laboratory
18501 East Gale Avenue, Suite 130
City of Industry, CA 91748-1321
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The above statement relates to the following attached confidential Enseco documents:

SOP NO. CRL-LM-7001
"The Determination of Volatile Organics (VOCs)
in Ambient Air by GC/MS - Scan Mode"

STANDARD
OPERATING
PROCEDURE

Subject or Title:

The Determination of Volatile Organics (VOCS)
in Ambient Air by GC/MS - Scan Mode

SOP NO: CRL-LM-7001 | Revision No.: 2.0 | Effective Date: March 1, 1990

Supercedes: Revision 1.0

1. Scope and Application

1.1 Analytes (See Table 1)

1.2 Detection limit (See Table 1)

1.3 Applicable matrices - air

1.4 Dynamic range (See Table 1)

1.5 Approximate analytical time

4 min. - cool down of cryo trap
2 min. - flush of inlet system on trap
10 min. - collection of 500 mL sample on trap
2 min. - flush of inlet system with internal std.
2 min. - collection of 100 mL of internal std on trap
2 min. - flush of trap with HP Helium
32 min. - GC run time

When running multiple samples, steps can be overlapped to reduce run time to 40 min.

Prepared by: Steve Harris — Date: 6/04/90

Management Approval: — Date: 6/04/90

QA Officer Approval: Paul Christie — Date: 6/5/90

TABLE 1. VOC Target Compounds

	Compound	R.T.	Detection Limits MDL (ppbv)	Dynamic Range (ppbv)
2)	Dichlorodifluoromethane (Freon 12)	1.49	0.87	0.87-300
3)	Chloromethane	2.48	1.2	1.2-300
4)	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	2.52	1.0	1.0-300
5)	Vinyl chloride	2.86	1.2	1.2-300
6)	Bromomethane	3.58	1.5	1.5-300
7)	Chloroethane	3.93	2.5	2.5-300
8)	Trichlorofluoromethane (11)	4.54	0.55	0.55-300
9)	cis-1,2-Dichloroethene	5.63	0.84	0.84-300
10)	Carbon disulfide	5.63	6.2	6.2-1200
11)	1,1,2-Trichloro- 1,2,2-trifluoroethane (Freon 113)	5.87	0.96	0.96-300
12)	Acetone	6.10	6.6	6.6-300
14)	Methylene chloride	6.89	1.9	1.9-300
15)	trans-1,2-Dichloroethene	7.36	1.9	1.9-300
16)	Hexane	7.98	4.0	4.0-300
17)	1,1-Dichloroethane	8.22	1.2	1.2-300
18)	Vinyl Acetate	8.71	1.3	1.3-300
19)	1,1-Dichloroethene	9.43	1.1	1.1-300
20)	2-Butanone	9.68	1.4	1.4-300
21)	Chloroform	10.27	1.1	1.1-300
22)	1,1,1-Trichloroethane	10.27	0.45	0.45-300
23)	Carbon tetrachloride	10.53	0.55	0.55-300
24)	Benzene	11.00	1.6	1.6-300
25)	1,2-Dichloroethane	11.19	0.53	0.53-300
26)	Trichloroethene	12.44	1.2	1.2-300
27)	1,2-Dichloropropane	12.87	3.9	3.9-300
28)	1,4-Dioxane	13.34	3.5	3.5-300
29)	Bromodichloromethane	13.68	0.90	0.90-300
30)	cis-1,3-Dichloropropene	14.61	1.5	1.5-300
31)	4-Methyl-2-pentanone	15.14	1.6	1.6-300
32)	Toluene	15.16	1.5	1.5-300
33)	trans-1,3-Dichloropropene	16.00	1.6	1.6-300
34)	1,1,2-Trichloroethane	16.34	1.4	1.4-300
35)	Tetrachloroethene	16.28	1.4	1.4-300
36)	2-Hexanone	17.10	3.0	3.0-300
37)	Dibromochloromethane	17.06	1.4	1.4-300
38)	1,2-Dibromoethane	17.08	1.0	1.0-300
39)	Chlorobenzene	18.28	1.3	1.3-300
40)	Ethylbenzene	18.73	1.3	1.3-300
41)	1,4-and 1,3-(p,m) Xylene	19.04	2.6	2.6-600

TABLE 1. VOC Target Compounds (Continued)

	Compound	R.T.	Detection Limits MDL (ppbv)	Dynamic Range
42)	1,2-(ortho) Xylene	19.94	1.1	1.1-300
43)	Styrene	20.02	3.5	3.5-300
44)	Bromoform	20.37	1.0	1.0-300
45)	1,1,2,2-Tetrachloroethane	21.99	1.9	1.9-300
46)	Benzyl chloride	21.90	1.0	1.0-300
47)	4-Ethyltoluene	22.31	2.0	2.0-300
48)	1,3,5-Trimethylbenzene	22.48	1.3	1.3-300
49)	1,2,4-Trimethylbenzene	23.37	1.5	1.5-300
50)	1,3-Dichlorobenzene	23.04	1.7	1.7-300
51)	1,4-Dichlorobenzene	24.13	2.2	2.2-300
52)	1,2-Dichlorobenzene	24.97	2.4	2.4-300
53)	1,2,4-Trichlorobenzene	29.28	3.6	3.6-300
54)	Hexachlorobutadiene	29.93	2.4	2.4-300

2. Summary of Method

2.1 A pressurized air sample is metered through a mass flow controller onto a cryogenically cooled trap. After 500 mL of the sample has been trapped, a valve is switched and the trap is heated to purge the trap's contents onto the gas chromatography column. The target compounds are analyzed with a mass spectrometer operated in the scan mode.

3. Comments

3.1 Interferences

3.1.1 Gas regulators are cleaned by the manufacturer using Freon 113, which is one of the target compounds. Before using ultra high purity (UHP) Nitrogen (N_2), Hydrocarbon (HC) free air, Internal Standard (I.S.), or a target compound standard mix, each regulator should be purged a minimum of three times with the appropriate gas.

3.1.2 Contamination may occur in the sampling system if canisters are not properly cleaned prior to use. Canisters used to collect source samples should not be used for the collection

of ambient air samples until a blank analysis indicates that no target compounds are present above the MDL. All other sampling equipment including pumps, flow controllers and filters must be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples.

3.2 Helpful Hints

None

4. Safety Issues

4.1 In order to prevent contamination of the lab air by the samples, the vent line must be connected to the system outlet and the fume hood must be on.

4.2 While making standards, the fume hood must be running. When finished valves must be closed and lines vented.

4.3 All compressed gas cylinders must be securely fastened to a bench or wall.

4.4 Normal precautions should be used in the handling of liquid nitrogen (LN_2) (do not touch transfer lines as burns can result).

4.5 Sampling canisters should never be pressurized over 40 psig.

5. Sample Collection, Preservation, Containers and Holding Times

5.1 Samples should be collected in precleaned and batch analyzed SUMMA passivated canisters. A 7 micron filter should be placed on the inlet of the can to protect the valve from particulates. Canisters should never be pressurized over 40 psig.

5.2 The absolute pressure of the canister must be recorded before and after sample collection.

5.3 Samples must be kept at <25°C.

5.4 Samples should be analyzed within 14 days of collection.

6. Apparatus and Materials

6.1 Gas chromatograph - capable of subambient temperature programming for the oven and with the jet separator option (Hewlett Packard 5890).

6.2 Mass-selective detector - equipped with computer and appropriate software (Hewlett Packard 5970B with HP-1000 RTE-A data system).

6.3 Cryogenic trap with temperature control assembly (Nutech 8533). See Figure 1.

6.4 Electronic mass flow controller - for maintaining constant sample flow (Unit Instruments)

6.5 Chromatographic grade stainless steel tubing and stainless steel plumbing fittings.

6.6 Chromatographic column - DB-624 0.53 ID, 30 meter length (J&W Scientific).

6.7 Stainless steel vacuum/pressure gauge capable of measuring from 30" of mercury (Hg) to 40 psig. (Span Instruments)

6.8 High precision vacuum gauge - for making daily standards. (Wallace & Tiernan Pennwalt)

6.9 Pressure regulators for carrier gas and standards - 2 stage, stainless steel diagram.

6.10 SUMMA passivated canisters 6 L (Scientific Instrumentation Specialists)

6.11 7 micron filters (Nupro), or equivalent.

7. Reagents and Standards

7.1 4-bromofluorobenzene, 50 ng/mL in methanol (for tuning of mass spectrometer).

7.2 High purity helium for carrier gas.

7.3 Standards at a nominal concentration of 1 ppmv (CS_2 is not as stable and so the concentration is 5 ppmv). Standards are prepared in a balance gas of nitrogen and are analytically certified by the supplier (Scott-Marrin and Scott Specialty). To facilitate certification by vendor, the standards were divided into 5 cylinders. (See Tables 2-7.)

7.4 Internal standard mix of bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d5 at 1000 ug/ml each in methanol (Supelco). (See Table 6.)

FIGURE 1. Nutech 8533 Flow Diagram

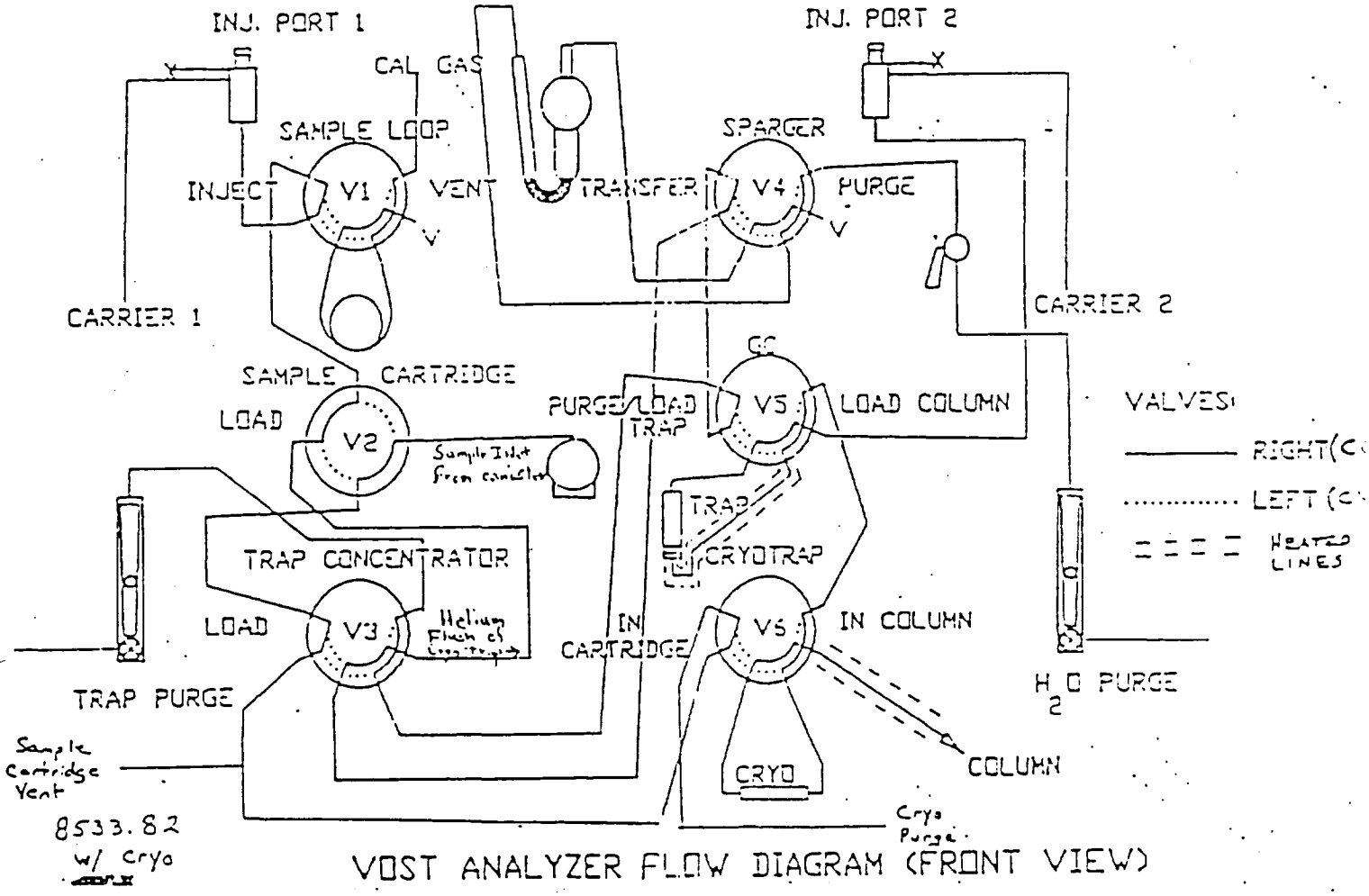

TABLE 2. Cylinder No. CC72069

Component	Concentration (v/v)
Chloromethane	0.98 ± 0.05 ppm
Bromomethane	1.00 ± 0.05 ppm
Chloroethane	0.96 ± 0.05 ppm
Dichloromethane	1.08 ± 0.05 ppm
trans-1,2-Dichloroethylene	1.08 ± 0.05 ppm
Trichloroethane	1.07 ± 0.05 ppm
1,2-Dichloroethane	1.10 ± 0.05 ppm
1,1,1-Trichloroethane	0.99 ± 0.05 ppm
Tetrachloromethane	1.01 ± 0.05 ppm
1,2-Dichloropropane	1.08 ± 0.05 ppm
cis-1,3-Dichloropropene	1.03 ± 0.05 ppm
trans-1,3-dichloropropene	1.20 ± 0.06 ppm
Dibromochloromethane	1.13 ± 0.05 ppm
Tetrachloroethylene	1.14 ± 0.05 ppm
Ethylbenzene	1.20 ± 0.06 ppm
p-Xylene	1.20 ± 0.06 ppm
Styrene	1.25 ± 0.06 ppm
1,1,2,2-Tetrachloroethane	1.24 ± 0.06 ppm
Bromodichloromethane	1.08 ± 0.05 ppm
Trichloroethene	0.82 ± 0.05 ppm
Acetonitrile	1.00 ± 0.05 ppm
Nitrogen	Balance

TABLE 3. Standard Cylinder No. CC72058

Component	Concentration (v/v)
Carbon Disulfide	4.86 ± 0.1 ppm
Nitrogen	Balance

TABLE 4. Standard Cylinder No. CC72063

Component	Concentration (v/v)
Vinyl Chloride	1.00 ± 0.05 ppm
1,1-Dichloroethene	1.08 ± 0.05 ppm
1,1-Dichloroethane	1.06 ± 0.05 ppm
2-Butanone	1.02 ± 0.05 ppm
cis-1,2-Dichloroethene	1.07 ± 0.05 ppm
Benzene	1.07 ± 0.05 ppm
4-Methyl-2-pentanone	1.09 ± 0.05 ppm
1,1,2-Trichloroethane	1.06 ± 0.05 ppm
Toluene	1.08 ± 0.05 ppm
2-Hexanone	1.18 ± 0.05 ppm
Chlorobenzene	1.08 ± 0.05 ppm
m-Xylene	1.11 ± 0.05 ppm
o-Xylene	1.12 ± 0.05 ppm
1,2-Dichlorobenzene	1.25 ± 0.05 ppm
Acetone	0.99 ± 0.05 ppm
1,4-Dichlorobenzene	1.04 ± 0.05 ppm
Nitrogen	Balance

TABLE 5. Standard Cylinder No. CC12390

Component	Concentration (v/v)
Freon-12	1.015 ± 0.05 ppm
Freon-114	0.95 ± 0.05 ppm
Freon-11	0.94 ± 0.05 ppm
Freon-113	0.99 ± 0.05 ppm
n-Hexane	1.02 ± 0.05 ppm
1,2-Dibromoethane	0.99 ± 0.05 ppm
4-Ethyltoluene	0.89 ± 0.05 ppm
1,3,5-Trimethylbenzene	0.95 ± 0.05 ppm
1,2,4-Trimethylbenzene	0.92 ± 0.05 ppm
Nitrogen	Balance

TABLE 6. Internal Standard Liquid Mix

Component	Concentration (ug/ml)
Bromochloromethane	1000
1,4-Difluorobenzene	1000
Chlorobenzene-d5	1000

TABLE 7. Standard Cylinder No. ALM 002636

Component	Concentration (v/v)
Benzyl chloride	0.737 ppm
1,3-Dichlorobenzene	0.768 ppm
1,4-Dioxane	0.895 ppm
Hexachloro-1,3-butadiene	0.804 ppm
Bromoform	0.84 ppm
1,2,4-Trichlorobenzene	0.898 ppm
Vinyl acetate	0.838 ppm
Nitrogen	

8. Procedure

8.1 Sample Preparation

8.1.1 The pressure of the sample canister is checked and recorded by attaching a vacuum/pressure gauge to the top valve of the canister (the gauge should be rinsed for few seconds with HC free air by physically holding against the air outlet and flushing). The canister valve is opened briefly and the pressure is recorded. If the pressure is less than 10 psig, pressurize the canister to 10 psig with HC free air.

8.1.2 If the canister pressure is increased, a dilution factor (DF) is calculated and recorded.

$$DF = \frac{Y_a}{X_a}$$

Where: X_a = absolute canister pressure absolute before dilution

Y_a = absolute canister pressure absolute after dilution

8.2 Daily GC/MS Tuning

8.2.1 At the beginning of each day or prior to a calibration, the GC/MS system must be tuned to verify that acceptable performance criteria are achieved. If any of the key ions fail the abundance criteria listed in Table 8, the system must be retuned using 4-Bromofluorobenzene (BFB).

8.2.2 For daily tuning, the relays on the Nutech controller (see Figure 1) should be in the right hand position, with the cryo trap at 150°C (alternatively valves 2 and 6 could be placed in the left hand position with the auxiliary He flow set at 10 ml/min or greater). The GC program is initiated by using the Datac command in file manager (FMGR). The GC program is named "GCBFB1." This downloads the program from the data system to the GC. Once the oven has stabilized, the remote start light will turn on and the system is ready for injection.

1 uL of a 50 ng/uL 4-bromofluorobenzene (BFB) standard is injected into injection port 2 of the Nutech 8533 and the remote start button is activated.

TABLE 8. 4-Bromofluorobenzene Key Ions and 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	<2% of mass 174
174	>50% of mass 96
175	5 to 9% of mass 174
176	>95% but <101% of mass 174
177	5 to 9% of mass 176

8.2.3 Once the tuning run is complete (~ 8 minutes), review a scan close to the center of the BFB peak. If it looks close to passing, type in the command TRF, TUNVOA, data file. This will start a program that will find a scan that will pass the tuning and print out the required information automatically. If the BFB tuning criteria cannot be met on 2-3 injections, retuning the instrument with PFTBA may be required.

TABLE 9. BFB Tuning Method

Enter the name of the method file: GCBFB1

M E T H O D F I L E L I S T

Method file: GCBFB1 | GC type: 5890 | Column: Cap | Run type: SCAN, GC, EI | Splitless: Yes

Temperature:	Inj.P	Intfc	Source
	90.0	250.0	0.0

GC/DIP		LEVEL A	LEVEL B	POST RUN
Temp 1	30.0	100.0	0.0	0.0
Time 1	1.0	15.0	0.0	0.0
Rate	35.0	0.0	0.0	
Temp 2	100.0	0.0	0.0	
Time 2	15.0	0.0	0.0	

Oven equilibration Time .10 min

Run time: 6.00
Scan Start time: 2.50
Splitless valve time: .80

	ON	OFF	ON	OFF
Relay #1:	327.0	327.0	327.0	327.0
Relay #2:	327.0	327.0	327.0	327.0
Triac #0:	327.0	327.0	327.0	327.0
Triac #1:	327.0	327.0	327.0	327.0

Scan Parameters: Mass Range 35 to 260
Multiplier voltage: 2244 Number of A/D samples: 8
GC Peak threshold: 20000 counts
Threshold: 100 counts

TABLE 10. Analytical Method

Enter the name of the method file: GCTO14

M E T H O D F I L E L I S T

Method file: GCTO14 | GC type: 5890 | Column: Cap | Run type: SCAN, GC, EI | Splitless: Yes

Temperature:	Inj.P	Intfc	Source
	90.0	250.0	0.0

GC/DIP		LEVEL A	LEVEL B	POST RUN
Temp 1	-50.0	-20.0	127.0	0.0
Time 1	2.0	0.0	20.0	0.0
Rate	70.0	5.0	0.0	
Temp 2	-20.0	127.0	0.0	
Time 2	0.0	20.0	0.0	

Oven equilibration Time .10 min

Run time: 32.10
Scan Start time: .10
Splitless valve time: 0.00

	ON	OFF	ON	OFF
Relay #1:	327.0	327.0	327.0	327.0
Relay #2:	327.0	327.0	327.0	327.0
Triac #0:	327.0	327.0	327.0	327.0
Triac #1:	327.0	327.0	327.0	327.0

Scan Parameters: Mass Range 35 to 260
Multiplier voltage: 2244 Number of A/D samples: 8
GC Peak threshold: 20000 counts
Threshold: 10 counts

8.3 Calibration

8.3.1 A static dilution of the stock standard gas mixtures is made in a 6 liter canister. The high precision vacuum gauge is flushed with HC free air and attached to the top valve of a clean, evacuated canister. After recording the absolute pressure, 2 psi of each of the 5 standard mixtures is added to the canister (each regulator and the transfer line must be

flushed several times before transfer of standard to the canister). Close the canister valves and replace the high precision gauge with a vacuum/pressure gauge. Pressurize the can with HC free air to 30 psig. This will yield a standard with a nominal concentration of 44 ppbv for most compounds (see Table 11).

8.3.2 An initial 5 point curve is run in the linear working range of the system. The nominal concentration of the 5 standards will be 18 ppbv, 67 ppbv, 90 ppbv, 224 ppbv and 287 ppbv.

8.3.3 On a daily basis, a one point midrange standard (500 ml of 44 ppbv) is run to verify the 5 point curve. 90% of the target compounds must be within 30% of the 5 point curve, or a new 5 point must be run. The daily, one point check standard is used to calculate the concentration of the samples.

8.3.4 After the calibration runs and the QA/QC sample runs, an HC free air blank is run. This must be < the MDL for each target compound.

8.4 Analysis

8.4.1 The daily check standard and the QA/QC samples are analyzed the same as samples. The HC free air blank is a system blank and differs only in that it is not transferred to a canister, but run directly from the cylinder regulator to the sample inlet system.

8.4.2 The sample canister is connected to the sample inlet system. The Nutech controller should have valves 2 and 6 in the left hand position, while valves 1 and 3-5 should be in the right hand position. The auxiliary He flow should be set at 40 ml/minute. The canister valve is opened and the pressurized sample is allowed to flow through the mass flow controller (set at 50 ml/min) and out the vent line.

8.4.3 The cryogenic trap is cooled to its lower set point of -170°C. When the cryo trap reaches -170°C, the Nutech valve #2 is switched to the right hand position and a timer is started. After 10 minutes (500 mL) valve #2 is switched back to the left hand position. Thus 500 ml of blank, standard or sample is concentrated on the cryo trap.

8.4.4 The valve on the sample canister is closed and the remaining line pressure is allowed to drop to ambient. The 3-way valve is then switched to the internal standard canister and the I.S. canister valve is opened and allowed to flush for at least 2 minutes. (The internal standard is made by injecting 20 ul of the liquid mix into an evacuated canister and pressurizing to 30 psi(44.6 psia). Valve is then switched to the right hand position and a timer is started After 2 minutes, Valve 2 is switched back to the left hand position Thus 100 ml of 200 ppbv nominal internal standard mix is injected into the cryotrap with each blank, standard or sample.

8.4.5 The GC is cooled to its' initial set point of -50°C by using Datac in file manager. The name of the GC program is "GCTO14." This takes about 2.5 minutes. During this time valves 2 and 6 remain in the left hand position, allowing He to sweep the trap and remove most oxygen, nitrogen and other permanent gases.

8.4.6 When the GC has reached equilibrium the red remote start light will turn on. Switch valve #6 to the right hand position. Wait at least 10 seconds to allow flow through the trap to equilibrate. The blue "cool" button on the Nutech controller and remote start button should be pressed simultaneously. This will heat the cryo trap to 150°C and start the GC program.

9. Data Interpretation

9.1 Qualitative Analyses

9.1.1 An analyte (e.g., those listed in Table 1) is identified 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 should be obtained on the user's GC/MS within the same 12 hours as the sample analysis. These standard reference spectra may be obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification. (1) elution of sample component at the same GC relative retention time (RRT) as those of the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.

9.1.1.1 The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within the same 12 hours as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

9.1.1.2 (1) All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. (2) The relative intensities of ions specified in (1) must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the correspondin sample abundance must be must be between 30 and 70 percent.

9.1.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Guidelines for making tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

9.2 Quantitative Analysis:

When a compound has been identified, the quantification of that compound will be based on the integrated abundance from the EICP of the primary charateristic ion. Quantification will take place using the internal standard technique.

10. QA/QC Requirements

10.1 The mass spectrometer must meet the tuning criteria described in Section 8.2.

10.2 After tuning, a single point check standard must be analyzed. Ninety percent of the target compound concentrations must be within ± 30% of the ~~three~~ five point calibration curve. If the check standard fails to meet this criterion, a new ~~three~~ five point calibration curve must be run.

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10.3 A laboratory control sample (LCS) must be analyzed after the check standard. This sample will consist of the target VOCs prepared in a separate canister at a concentration that differs from that of the check standard. Five compounds will be used to assess control for the LCS: methylene chloride, 1,1-dichloroethene, trichloroethene, toluene and 1,1,2,2-tetrachloroethane. The percent recovery for the five control compounds must be within a window of 80-115%.

10.4 For each lot of 20 samples analyzed, a duplicate control sample (DCS) must be analyzed after the LCS. The DCS sample is identical to the LCS in composition and source. The 80-115% recovery criterion must be met. In addition, the relative percent difference (RPD) for the LCS and DCS must be ≤ 20%.

10.5 A system blank of HC free air must be analyzed after the LCS or DCS. The blank results must indicate that there are no target compounds present above the MDL.

10.6 If any of the above criteria are not met, corrective actions must be implemented before analyses can proceed.

11. Calculations

11.1 The HP data system automatically quantitates the sample results based on a 500 mL sample size. The results are in ppbv. If the canister was pressurized before analysis, the results must be multiplied by the dilution factor DF (see Section 8.1.2).

11.2 If a sample size other than 500 mL was used, the result must be adjusted as shown below:

$$\text{result ppbv} \times \frac{\text{sample volume injected}}{500\ \text{mL}}$$

12. Reporting

12.1 Reporting units are ppbv. If results are to be reported in ng/L use the following equation:

$$\text{result ppbv} \times \frac{\text{Molecular weight of compound}}{24.5} = \text{ng/L}$$

Note: 24.5 is the standard volume of ideal gas at 25 degrees Centigrade and 1 atm.

12.2 Reporting limits

See Table 12

12.3 Significant figures

12.3.1 All results should be reported to two significant figures.

12.3.2 Only report results below detection limit as ND(DL).

12.4 No conversion of the analytical results to the standard conditions is made.

13. References

13.1 Method Source

"EPA Compendium Method TO-14. The Determination of Volatile Organic Compounds (VOCs) in Ambient Air using SUMMA Passivated Canister Sampling and Gas Chromatographic Analysis."

13.2 Deviations from Method

13.2.1 Dry HC free air is used for the daily blank and for dilution purposes.

13.2.2 TO-14 recommends the use of a .32 mm column coupled directly to the MSD. With the HP system, the MSD can only handle flow of 1 mL/min or less. The .32 mm column provides ~ 3 mL/min. Enseco uses a .53 mm column through a jet separator.

13.2.3 TO-14 describes an inlet system that uses a vacuum to pull a slip stream sample through the trap. Enseco uses the pressure of the sample canister to drive the sample through the trap.

TABLE 11. Concentration of Daily Check Standard

	Compound	Concentration (ppbv)
2)	Dichlorodifluoromethane (Freon 12)	45.42
3)	Chloromethane	43.84
4)	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	42.50
5)	Vinyl chloride	44.74
6)	Bromoethane	44.74
7)	Chloroethane	42.96
8)	Trichlorofluoromethane (11)	42.06
9)	cis-1,2-Dichloroethene	47.88
10)	Carbon disulfide	217.44
11)	1,1,2-Trichloro- 1,2,2-trifluoroethane (Freon 113)	44.30
12)	Acetone	44.30
14)	Methylene chloride	48.32
15)	trans-1,2-Dichloroethene	48.32
16)	Hexane	45.64
17)	1,1-Dichloroethane	47.42
18)	Vinyl Acetate	37.50
19)	1,1-Dichloroethene	48.32
20)	2-Butanone	45.64
21)	Chloroform	47.82
22)	1,1,1-Trichloroethane	44.30
23)	Carbon tetrachloride	45.20
24)	Benzene	47.82
25)	1,2-Dichloroethane	49.22
26)	Trichloroethene	36.68
27)	1,2-Dichloropropane	48.32
28)	1,4-Dioxane	40.04
29)	Bromodichloromethane	48.32
30)	cis-1,3-Dichloropropene	46.08
31)	4-Methyl-2-pentanone	48.76
32)	Toluene	48.32
33)	trans-1,3-Dichloropropene	53.70
34)	1,1,2-Trichloroethane	53.70
35)	Tetrachloroethene	51.00
36)	2-Hexanone	52.80
37)	Dibromochloromethane	50.56
38)	1,2-Dibromoethane	44.30
39)	Chlorobenzene	48.32
40)	Ethylbenzene	53.70
41)	1,4-and 1,3-(p,m) Xylene	103.36

TABLE 11. Concentration of Daily Check Standard

Compound	Concentration (ppbv)
42) 1,2-(ortho) Xylene	50.12
43) Styrene	55.92
44) Bromoform	37.58
45) 1,1,2,2-Tetrachloroethane	55.48
46) Benzyl chloride	32.98
47) 4-Ethyltoluene	39.82
48) 1,3,5-Trimethylbenzene	42.50
49) 1,2,4-Trimethylbenzene	41.16
50) 1,3-Dichlorobenzene	34.36
51) 1,4-Dichlorobenzene	46.54
52) 1,2-Dichlorobenzene	55.92
53) 1,2,4-Trichlorobenzene	40.18
54) Hexachlorobutadiene	35.98

Table 12. VOC Reporting Limits

	Compound	Reporting Limits (ppbv)
2)	Dichlorodifluoromethane (Freon 12)	2.0
3)	Chloromethane	2.5
4)	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	2.0
5)	Vinyl chloride	2.5
6)	Bromoethane	3.0
7)	Chloroethane	5.0
8)	Trichlorofluoromethane (11)	1.0
9)	cis-1,2-Dichloroethene	2.0
10)	Carbon disulfide	10
11)	1,1,2-Trichloro- 1,2,2-trifluoroethane (Freon 113)	2.0
12)	Acetone	10
14)	Methylene chloride	4.0
15)	trans-1,2-Dichloroethene	4.0
16)	Hexane	8.0
17)	1,1-Dichloroethane	2.5
18)	Vinyl Acetate	2.5
19)	1,1-Dichloroethene	2.0
20)	2-Butanone	3.0
21)	Chloroform	2.0
22)	1,1,1-Trichloroethane	2.0
23)	Carbon tetrachloride	2.0
24)	Benzene	3.0
25)	1,2-Dichloroethane	2.0
26)	Trichloroethene	2.5
27)	1,2-Dichloropropane	8.0
28)	1,4-Dioxane	7.0
29)	Bromodichloromethane	2.0
30)	cis-1,3-Dichloropropene	3.0
31)	4-Methyl-2-pentanone	3.0
32)	Toluene	3.0
33)	trans-1,3-Dichloropropene	3.0
34)	1,1,2-Trichloroethane	3.0
35)	Tetrachloroethene	3.0
36)	2-Hexanone	5.0
37)	Dibromochloromethane	3.0
38)	1,2-Dibromoethane	2.0
39)	Chlorobenzene	2.5
40)	Ethylbenzene	2.5
41)	1,4-and 1,3-(p,m) Xylene	5.0

Table 12. VOC Reporting Limits

	Compound	Reporting Limits (ppbv)
42)	1,2-(ortho) Xylene	2.0
43)	Styrene	7.0
44)	Bromoform	2.0
45)	1,1,2,2-Tetrachloroethane	4.0
46)	Benzyl chloride	2.0
47)	4-Ethyltoluene	4.0
48)	1,3,5-Trimethylbenzene	2.5
49)	1,2,4-Trimethylbenzene	3.0
50)	1,3-Dichlorobenzene	3.0
51)	1,4-Dichlorobenzene	4.0
52)	1,2-Dichlorobenzene	5.0
53)	1,2,4-Trichlorobenzene	7.0
54)	Hexachlorobutadiene	5.0

APPENDIX B-12

CLAY MINERALOGY BY X-RAY DIFFRACTION (CORE)

4.2.2 X-ray Diffraction Standard Operating Procedures

INTRODUCTION

Parameter to be measured

The parameter to be measured in an X-ray diffraction analysis is accurate mineral content of a selected sample in weight percent.

Range of measurement

Core Laboratories uses a Philips Electronic Instruments Model 3600 Automated Powder Diffractometer. The sample holder of a Philips goniometer is connected to a center shaft, called the "Theta Shaft" and rotates at a prescribed rate which is propelled by a computer-controlled stepper motor. Each angular increment is described as theta angle increment. The detector arm is coupled to the theta shaft in a 2 to 1 fashion. All measurements of a sample are expressed as "two-theta" measurements. The Philips goniometer has a working distance of 2 degrees two-theta to 140 degrees two-theta.

The scope of phases measured includes those that are crystalline or quasi-crystalline (certain clay minerals). Amorphous materials cannot be directly measured by X-ray diffraction. Two basic groups of phases, or minerals, are considered, namely rock-forming minerals (quartz, feldspars, carbonates, sulfides, etc.) and phyllosilicates (clay minerals). The diffraction peaks of these minerals are measured and stored for later analysis.
Certain procedures are available for detection and semi-quantitative analysis of amorphous phases; however, they are currently not implemented by Core Laboratories. X-ray diffraction techniques cannot supply any qualitative information about amorphous phases unless elemental data is supplied or prior knowledge of the phase leaves no doubt to its chemical composition.

Limit of Detection

The limit of detection of X-ray diffraction of a normally prepared sample is 1 weight percent of the fraction analyzed. Various methods for concentrating certain target groups of minerals are implemented by Core Laboratories, as necessary. Also, certain conditions within the diffractograms (overlaps of two phases' diffraction peaks) may raise the detection limit of a phase. Accuracy of the diffraction analysis is ± 5 weight percent.

Principle, Scope, and Application

X-ray diffraction analysis is a method used for determining the mineral content of a substance. Most materials consist of tiny crystals which are made of ordered arrays of atoms arranged in a periodic or repetitive way. When an incident X-ray beam impinges upon such an array, general scattering occurs, such that the scattered waves interfere with and destroy one another. In certain specific directions, however, the scattered waves are in phase with one another and combine to form new wave-fronts. This constructive interference is know as diffraction. The direction in which diffraction occurs depend upon the size and shape of the unit cell of the crystal, whereas the intensity of diffraction is determined

by the actual atomic array or the nature of the crystal structure. The condition for reflection of X-rays from a parallel arrangement of atoms within a crystal is called the Bragg Angle. Bragg's Law,

$$n\ l = 2d\sin 0,$$

where, n is the order of reflection, lambda is the wavelength of incident X-rays, d is the interplanar spacing between rows of atoms and theta is the angle of the incident X-rays. This is the basic principle behind X-ray diffraction. This principle applies to crystalline materials and does not apply on amorphous substances (glass, certain organics, certain clays, etc.). For a more detailed discussion, please refer to Cullity (1967).

Each crystalline substance, or phase, has a unique set of integral peaks that are like individual fingerprints of humans. For each diffractogram, these peaks are measured in the angular expression of two-theta space, identified and indexed for future reference. A very large database (> 100,000) of phases has been compiled by the Joint Committee of Powder Diffraction Standards (JCPDS) and is available in book form and magnetic media for computer-aided search and match algorithms and for comparison to the indexed pattern for identification of phases in the diffractogram. The intensity of a diffraction peak of a phase is indirectly proportional to the concentration of the phase. There are a number of phenomenon that need to be considered when doing a quantitative analysis on multiphase substances. Please refer Klug and L. E. Alexander for more detail on this matter.

SAMPLE PREP AND ANALYSIS

Sample Requirements

Sample supplied for X-ray diffraction must be a representative portion of the sample. Approximately 10 grams are required for a routine analysis. No preservation is necessary other than insuring the sample was maintained at ambient conditions after coring.

Sample Preparation

Sample selected for X-ray diffraction analysis is dried and cleaned of obvious contaminants. For hydrocarbon removal, the sample is placed in a low temperature, methylene chloride soxhlet for 24 hours. The sample is gently ground in a ceramic mortar and pestle and split into two equal portions using a riffle splitter. One split is saved for later milling for a bulk analysis pellet. This procedure is described later. The remaining split is tested for halides (silver nitrate test). If needed, the sample is placed in a low-temperature methanol soxhlet for up to 72 hours, or until sample is leached of all halides. The sample is weighed and placed in 150 milliliters of deionized water treated with 1 volume percent Calgon water softener and treated with a sonic cell disrupter for 5 minutes. This procedure breaks the clay from host grains and breaks down lithic fragments containing clay. The Calgon is added to aid in clay dispersion. The resultant slurry is placed in a

centrifuge in order to fractionate the sample at 2 microns. The suspended < 2 micron fraction is decanted and saved, and the > 2 micron fraction is washed and centrifuged again to remove any < 2 micron particles. The remaining < 2 micron particles are added to the saved < 2 micron portion. The > 2 micron fraction is dried and weighed to determine the percent of clay- and silt-sized material. This weight is used to determine the net clay of the sample. The suspended < 2 micron fraction is suctioned onto a pure silver substrate with an average pore diameter of 1.2 microns. The resulting aggregate of sample and substrate is dried and mounted on a glass slide. The > 2 micron fraction and the previously saved split of sample are milled in a McCrone micronizing mill with low-water isopropanol and then packed into sample holders for analysis. The > 2 micron fraction and the bulk split are scanned from 2 to 60 degrees two-theta at 1 degree per minute. The < 2 micron sample mount is run first in the natural (air-dried) state and then treated with ethylene glycol vapor for 24 hours and run again. Both clay runs are from 2 to 40 degrees at 1 degree per minute.

Apparatus

The sample is analyzed with a Philips APD 3600 X-ray diffractometer at a slow rate (1 degree/minute) using a copper tube (wavelength CuK alpha$_1$ = 1.54051 angstroms) with 35 kilovolt potential and 35 milliamp current. Scintillator voltage is 800 volts and time constant is 1. A Data General Nova 4X is used to the collect and store data while a Data General Eclipse S/140 is used for data reduction.

Analytical Measurements

The diffractograms for each run are first inspected for any data collection anomalies and the samples are scanned again if necessary. The bulk split is analyzed for any water-soluble minerals. The bulk split is also compared to the > 2 micron sample for consistency, clays, et cetera. If any inconsistencies are identified, the sample may be rerun. If any water-soluble minerals are detected, they are quantified and added to the total mineral analysis.

Minerals in the >2 micron powder mount and clay mount are identified by comparison of the diffractograms to standards, either prepared and run on Philips diffractometer or standards from the Joint Committee on Powder Diffraction Standards. A computer algorithm is available for complex pattern or for rare mineral phases which matches the unknown pattern to those of the JCPDS for accurate phase identification. Most of the clay mineral standards are patterns that were run on the Philips diffractometer. Certain clay minerals called "mixed-layer clays" are found in many sediments. Occasionally, it is not possible to collect standards for every mixed-layer clay that could be anticipated in a given basin. Therefore, Core Laboratories uses a computer algorithm developed by Reynolds that calculates a pattern of mixed-layer clays based on unit cell and chemical formulae restrictions. The resulting calculated pattern intensities are analyzed with the profile-fitting algorithm and then intensity relationships are

established. Compositions and species of the clay minerals are determined according to procedures outlined by Reynolds (1980, 1985), Grim (1962), Wilson (1987), Srodon (1980, 1984), Carroll (1970).

The integrated areas of the most intense reflection for each phase are obtained using a multiple Lorentzian profile-fitting algorithm (Schreiner and Jenkins, 1983) which is part of an analytical software package provided with the Philips diffractometer. Some modifications were made by Core Laboratories to the source code of the profile-fitting algorithm to make it more efficient. The integrated areas are corrected for various absorption and orientation phenomena with structure factors obtained from previous calibrations of each phase using a modified Chung method (1974a, b, and c). The corrected integrated areas are normalized to 100 percent. Any clay minerals trapped in the > 2 micron fraction are quantified and mathematically added to the clay category. Any rock-forming minerals trapped in the < 2 fraction are also mathematically removed and added to the rock-forming mineral category. The integrated area for each phase is entered into a spreadsheet that calculates the weight percent data. The results are then tabulated and sorted according to depth or any relevant sample identification.

Interferences and corrective actions

Particle size plays an important part of the diffraction intensity. Too small a particle size produces a small and broad diffraction peak, and in extreme cases, the peak of phase with too small a particle size can no longer diffract X-rays and completely disappear due to extensive lattice damage. Too large a particle size produces primary extinction effects and also can lead to preferred orientation. A completely random ordering of crystallites in a powder mount is most preferred because preferred cleavage planes are often the most intense diffraction planes which could lead to overestimation of the orienting phase. Conversely, clay minerals are often oriented by using a suction or settling method because they are not as efficient as diffracting X-rays along what are called their "hkl" planes. Clay minerals are typically flat, platey crystals with the edge surfaces being the hkl planes of the clay mineral crystallites. Basal planes of clay minerals are aligned with the long axis of the crystallite, consequently when the clay crystallite is aligned along the long axis, it diffracts much more efficiently due to an ideal arrangement of atoms along the basal or "001" plane and integrals.

Salts are a problem with respect to water separating the sample to < 2 and > 2 microns because as the halite goes into solution it provides a nucleating site for the strongly negatively charged clay particles. As the clays nucleate, or flocculate, onto cations in solution, the clays tend to fall out of suspension immediately, thus ruining the gravimetric portion of the experiment. That is why the salts are removed with methanol if their presence is detected. It does not take very much salt to ruin the procedure.

Hydrocarbons are also a problem with respect to preparation of samples because they tend to cause the sand to ball up and float on the surface. Consequently, if detected or suspected, the soluble hydrocarbons are removed with methylene chloride. Any insoluble hydrocarbons remain behind but cause no major sample prep problems.

A large source of error in diffraction is the overlap of two or more peaks at the same or proximal two-theta positions. This is minimized by performing profile-fitting (not deconvolution) of overlapped areas of the diffractograms. One particularly notorious overlap is that between two clay minerals, kaolinite and chlorite. If these two minerals (one occurring at 24.9 and 25.2 degrees two theta, respectively) were quantified by peak intensity only, errors as high as 100% may occur.

The silver substrate below the deposited clay minerals often is detected in a diffractogram as elemental silver. This is due to the finite thickness of the clay on the silver substrate. If not accounted for, errors will be generated by comparing intensities from low two theta angles to intensities from high two theta angles. The error occurs due to the path distance differential of the incident X-ray beam. At low two-thetas, the path distance will be much larger due to the larger incident angle, consequently giving a much larger intensity of a diffraction peak than that of a diffraction peak from a higher two-theta. At higher two-thetas, the path distance is much shorted due to the higher incident angle. This must be corrected by applying the formula

$$I_{inf} = I_{thin}/[1-\exp(-2ut/\sin 0)]$$

that corrects all two-theta position intensities to those of an infinitely thick mount. By measuring the intensity of the silver peak on the unknown sample versus that of an external standard of pure silver, the exact thickness of the sample substrate can be calculated and all intensities can be converted to that of infinitely thick "powder mount".

Poor alignment of the X-ray diffractometer can also lead to serious errors and is remedied by frequently observing the most intense peak (101 - hkl index) of alpha quartz which occurs at approximately 26.66 degrees two theta and making adjustments to goniometer position as needed. Any time a diffractometer is moved, it is necessary to do a "ground up" alignment.

Calibration

Calibration of the rock-forming minerals was accomplished by running a series of external standard mineral phases, tested for purity and known for their abundance of source, against alpha quartz as a flushing agent (Chung, 1974). Ten mixtures of each phase with quartz, in graduated amounts from 0 to 100 percent, were analyzed and the ratio of observed versus actual concentrations were plotted. A non-weighted, least-squares regression analysis was performed on each data set to evaluate the best fit line

through the data. The slope and y-intercept of each phase were used to calculate a normalized intensity correction coefficient for each standard. This correction coefficient is free of matrix absorption effects and is also free of orientation effects since the standards and the unknowns are prepared in the same fashion. The clay minerals were calibrated in a similar fashion with the exception of chlorite (CCa-1, ripidolite) replacing quartz. This was repeated for each phase. Up to 70 minerals have now been calibrated.

Data Deliverables

As requested, the following will be provided

a) Case narrative as outlined.

b) Summary of initial calibration as detailed above. This calibration is performed only once until a major change is effected.

c) Summary of sample analysis as outlined.

d) QC analyses are not routinely performed. Can be effected upon request.

e) Diffractograms will be provided.

f) Instrument logbook will be provided as requested.

Safety Precautions

Core Laboratories is fully licenced facility registered with the Texas Department of Health for operating radiation generating devices. A site survey was performed in August, 1990 by Henken Industries for site leakage of X-rays and were deemed operating at a safe (minimal) exposure level. All employees wear both deep and shallow dose radiation detection devices that are monitored on a monthly basis. All employees are sufficiently trained in the safe operation of the X-ray diffractometer and appropriate warnings and advisements are posted in proximity to the machine and at all doors entering into the laboratory.

Our laboratory staff practice all safety procedures for handling hazardous materials and wear all accoutrements necessary for safe laboratory operation.

Preventative Maintenance

No preventative maintenance is required for the X-ray diffractometer other than checking cooling water levels in the heat exchanger and checking the overall appearance and insuring that all safety interlocks are functioning properly.

Quality Control

Besides data quality checks by the X-ray analyst, quality control as a strict regimen is not observed; however numerous cross checks are performed when thin section or FTIR analyses are performed.

Any discrepancies observed are promptly investigated and, if necessary, samples are re-analyzed to check results.

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APPENDIX B-13

NATURAL MOISTURE CONTENT (WARZYN)

Standard Method for LABORATORY DETERMINATION OF WATER (MOISTURE) CONTENT OF SOIL, ROCK, AND SOIL-AGGREGATE MIXTURES[1]

This standard is issued under the fixed designation D 2216; 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 method covers the laboratory determination of the water (moisture) content of soil, rock, and soil-aggregate mixtures by weight. For simplicity, the word "material" hereinafter refers to either soil, rock, or soil-aggregate mixtures, whichever is most applicable.

1.2 The water content of a material is defined as the ratio, expressed as a percentage, of the mass of "pore" or "free" water in a given mass of material to the mass of the solid material particles.

1.3 This method does not give true representative results for: materials containing significant amounts of halloysite, montmorillonite, or gypsum minerals; highly organic soils; or, materials in which the pore water contains dissolved solids (such as salt in the case of marine deposits). For a material of the previously mentioned types, a modified method of testing or data calculation may be established to give results consistent with the purpose of the test.

2. Summary of Method

2.1 The practical application in determining the water content of a material is to determine the mass of water removed by drying the moist material (test specimen) to a constant mass in a drying oven controlled at 110 ± 5°C and to use this value as the mass of water in the test specimen. The mass of material remaining after oven-drying is used as the mass of the solid particles.

3. Significance and Use

3.1 For many soil types, the water content is one of the most significant index properties used in establishing a correlation between soil behavior and an index property.

3.2 The water content of a soil is used in almost every equation expressing the phase relationships of air, water, and solids in a given volume of material.

3.3 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 limit, is used to express its relative consistency or liquidity index.

3.4 The term "water" as used in geotechnical engineering, is typically assumed to be "pore" or "free" water and not that which is hydrated to the mineral surfaces. Therefore, the water content of materials containing significant amounts of hydrated water at in-situ temperatures or less than 110°C can be misleading.

3.5 The term "solid particles" as used in geotechnical engineering, is typically assumed to mean naturally occurring mineral particles that are not readily soluble in water. Therefore, the water content of materials containing extraneous matter (such as cement, etc), water-soluble matter (such as salt) and highly organic

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[1] This method is under the jurisdiction of ASTM Committee D-18 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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matter typically require special treatment or a qualified definition of water content.

4. Apparatus

4.1 *Drying Oven*, thermostatically-controlled, preferably of the forced-draft type, and maintaining a uniform temperature of 110 ± 5°C throughout the drying chamber.

4.2 *Balances*, having a precision (repeatability) of ±0.01 g for specimens having a mass of 200 g or less, ±0.1 g for specimens having a mass of between 200 and 1000 g, or ± 1 g for specimens having a mass greater than 1000 g.

4.3 *Specimen Containers*—Suitable containers made of material resistant to corrosion and a change in mass upon repeated heating, cooling, and cleaning. 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 (Note 1). One container is needed for each water content determination.

NOTE 1—The purpose of close-fitting lids is to prevent loss of moisture from specimens before initial weighing and to prevent absorption of moisture from the atmosphere following drying and before final weighing.

4.4 *Desiccator*—A desiccator of suitable size (a convenient size is 200 to 250-mm diameter) containing a hydrous silica gel. This equipment is only recommended for use when containers having close-fitting lids are not used. See 7.4.1.

5. Samples

5.1 Keep the samples that are stored prior to testing in noncorrodible airtight containers at a temperature between approximately 3 and 30°C and in an area that prevents direct contact with sunlight.

5.2 The water content determination should be done as soon as practicable after sampling, especially if potentially corrodible containers (such as steel thin-walled tubes, paint cans, etc.) or sample bags are used.

6. Test Specimen

6.1 For water contents being determined in conjunction with another ASTM method, the method of specimen selection specified in that method controls.

6.2 The manner in which the test specimen is selected and its required mass is basically dependent on the purpose (application) of the test, type of material being tested, and the type of sample (specimen from another test, bag, tube, split-barrel, etc.). In all cases, however, a representative portion of the total sample shall be selected. If a layered soil or more than one soil type is encountered, select an average portion or individual portions or both, and note which portion(s) was tested in the report of the results.

6.2.1 For bulk-samples, select the test specimen from the material after it has been thoroughly mixed. The mass of moist material selected shall be in accordance with the following table:

Sieve Retaining More Than About 10 % of Sample	Recommended Minimum Mass of Moist Specimen, g
2.0 mm (No. 10) sieve	100 to 200
4.75 mm (No. 4) sieve	300 to 500
19 mm	500 to 1000
38 mm	1500 to 3000
76 mm	5000 to 10 000

6.2.2 For small (jar) samples, select a representative portion in accordance with the following procedure:

6.2.2.1 For cohesionless soils, thoroughly mix the material, then select a test specimen having a mass of moist material in accordance with the table in 6.2.1. See Note 2.

6.2.2.2 For cohesive soils, remove about 3 mm of material from the exposed periphery of the sample and slice it in half (to check if the material is layered) prior to selecting the test specimen. If the soil is layered see 6.2. The mass of moist material selected should not be less than 25 g or should be in accordance with the table in 6.2.1 if coarse-grained particles are noted. (Note 2).

6.3 Using a test specimen smaller than the minimum mass indicated previously requires discretion, though it may be adequate for the purpose of the test. A specimen having a mass less than the previously indicated value shall be noted in the report of the results.

NOTE 2—In many cases, when working with a small sample containing a relatively large coarse-grained particle, it is appropriate not to include this particle in the test specimen. If this occurs, it should be noted in the report of the results.

7. Procedure

7.1 Select representative test specimens in accordance with Section 6.

7.2 Place the moist specimen in a clean, dry container of known mass (Note 3), set the lid securely in position, and determine the mass of the container and moist material using an appropriate balance (4.2). Record these values.

7.3 Remove the lid and place the container with moist material in a drying oven maintained at 110 ± 5°C and dry to a constant mass (Notes 4, 5, and 6).

NOTE 3—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.

NOTE 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. In most cases, drying a test specimen over night (about 16 h) is sufficient. In cases where there is doubt concerning the adequacy of drying, drying should be continued until the mass after two successive periods (greater than ½ h) of drying indicate an insignificant change (less than about 0.1 %). Specimens of sand may often be dried to constant mass in a period of about 4 h, when a forced-draft oven is used.

NOTE 5—Oven-drying at 110 ± 5°C does not always result in water content values related to the intended use or the basic definition especially for materials containing gypsum or other minerals having significant amounts of hydrated water or for soil containing a significant amount of organic material. In many cases, and depending on the intended use for these types of materials, it might be more applicable to maintain the drying oven at 60 ± 5°C or use a vacuum desiccator at a vacuum of approximately 133 Pa (10 mm Hg) and at a temperature ranging between 23 and 60°C for drying. If either of these drying methods are used, it should be noted in the report of the results.

NOTE 6—Since some dry materials may absorb moisture from moist specimens, dried specimens should be removed before placing moist specimens in the oven. However, this requirement is not applicable if the previously dried specimens will remain in the drying oven for an additional time period of about 16 h.

7.4 After the material has dried to constant mass remove the container from the oven and replace the lid. Allow the material 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. Determine the mass of the container and oven-dried material using the same balance as used in 7.2. Record this value.

7.4.1 If the container does not have a lid, weigh the container and material right after their temperatures are such that the operation of the balance will not be affected by convection currents or after cooling in a desiccator.

NOTE 7—Cooling in a desiccator is recommended since it prevents absorption of moisture from the atmosphere during cooling.

8. Calculation

8.1 Calculate the water content of the material as follows:

$$w = [(W_1 - W_2)/(W_2 - W_c)] \times 100 = \frac{W_w}{W_s} \times 100$$

where:

w = water content, %,
W_1 = mass of container and moist specimen, g,
W_2 = mass of container and oven-dried specimen, g,
W_c = mass of container, g,
W_w = mass of water, g, and
W_s = mass of solid particles, g.

9. Report

9.1 The report (data sheet) shall include the following:

9.1.1 Identification of the sample (material) being tested, by boring number, sample number, test number, etc.

9.1.2 Water content of the specimen to the nearest 0.1 % or 1 %, depending on the purpose of the test.

9.1.3 Indication of test specimen having a mass less than the minimum indicated in Section 6.

9.1.4 Indication of test specimen containing more than one soil type (layered, etc).

9.1.5 Indication of the method of drying if different from oven-drying at 110 ± 5°C.

9.1.6 Indication of any material (size and amount) excluded from the test specimen.

10. Precision and Accuracy

10.1 Requirements for the precision and accuracy of this test method have not yet been developed.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103.

BY _____ DATE _____ JOB NAME _____ JOB NO. _____

CHECKED BY _____ _____ SHEET _____ OF _____

	SAMPLE NO.									
	MATERIAL.									
UNCONFINED COMP.	DIA, in.									
	INITIAL HT, in.									
	FINAL HT, in.									
	MAX LOAD, lbs.									
	Qa, tsf.									
	Qu, tsf.									
UNIT WEIGHT	DIA, in.									
	LENGTH, in.									
	WEIGHT, gms.									
	1 + NM									
	DRY UNIT WEIGHT pcf.									
NATURAL MOISTURE	CAN NO.									
	WET WT, gms.									
	DRY WT, gms.									
	TARE, gms.									
	% MOISTURE.									
LIQUID LIMIT	CAN NO.									
	WET WT, gms.									
	DRY WT, gms.									
	TARE, gms.									
	BLOW MULT									
	% MOISTURE.									
PLASTIC LIMIT	CAN NO.									
	WET WT, gms.									
	DRY WT, gms.									
	TARE. gms.									
	% MOISTURE									
IGNITION LOSS	CUP NO.									
	START WT, gms.									
	END WT, gms.									
	TARE, gms.									
	% LOSS.									

REMARKS:

APPENDIX B-14

ATTERBERG LIMITS (WARZYN)

Standard Test Method for LIQUID LIMIT, PLASTIC LIMIT, AND PLASTICITY INDEX OF SOILS[1]

This standard is issued under the fixed designation D 4318; 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 test method has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This test method covers the determination of the liquid limit, plastic limit, and the plasticity index of soils as defined in Section 3.

1.1.1 Two procedures for preparing test specimens and two procedures for performing the liquid limit are provided as follows:

A Multipoint test using a wet preparation procedure, described in Sections 10.1, 11, and 12.

B Multipoint test using a dry preparation procedure, described in Sections 10.2, 11, and 12.

C One-point test using a wet preparation procedure, described in Sections 13, 14, and 15.

D One-point test using a dry preparation procedure, described in Sections 13, 14, and 15.

The procedure to be used shall be specified by the requesting authority. If no procedure is specified, Procedure A shall be used.

NOTE 1—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.

1.1.2 The plastic limit test procedure is described in Sections 16, 17, and 18. The plastic limit test is performed on material prepared for the liquid limit test. In effect, there are two procedures for preparing test specimens for the plastic limit test.

1.1.3 The procedure for calculating the plasticity index is given in Section 19.

1.2 The liquid limit and plastic limit of soils (along with the shrinkage limit) are often collectively referred to as the Atterberg limits in recognition of their formation by Swedish soil scientist, A. Atterberg. These limits distinguish the boundaries of the several consistency states of plastic soils.

1.3 As used in this test method, soil is any natural aggregation of mineral or organic materials, mixtures of such materials, or artificial mixtures of aggregates and natural mineral and organic particles.

1.4 The multipoint liquid limit procedure is somewhat more time consuming than the one-point procedure when both are performed by experienced operators. However, the one-point procedure requires the operator to judge when the test specimen is approximately at its liquid limit. In cases where this is not done reliably, the multipoint procedure is as fast as the one-point procedure and provides additional precision due to the information obtained from additional trials. It is particularly recommended that the multipoint procedure be used by inexperienced operators.

1.5 The correlations on which the calculations of the one-point procedure are based may not be valid for certain soils, such as organic soils or

[1] This test method is under the jurisdiction of ASTM Committee D-18 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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soils from a marine environment. The liquid limit of these soils should therefore be determined by the multipoint procedure (Procedure A).

1.6 The liquid and plastic limits of many soils that have been allowed to dry before testing may be considerably different from values obtained on undried 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.

1.7 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 D 2216). 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 consideration if meaningful results are to be obtained.

1.8 Since the tests described herein are performed only on that portion of a soil which passes the 425-µm (No. 40) sieve, 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 the properties of a soil.

1.9 The values stated in acceptable metric units are to be regarded as the standard. The values given in parentheses are for information only.

1.10 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Documents

2.1 *ASTM Standards:*

C 702 Methods for Reducing Field Samples of Aggregate to Testing Size[2]

D 75 Practice for Sampling Aggregates[3]

D 420 Recommended Practice for Investigating and Sampling Soil and Rock for Engineering Purposes[4]

D 653 Terms and Symbols Relating to Soil and Rock[4]

D 1241 Specification for Materials for Soil-Aggregate Subbase, Base, and Surface Courses[4]

D 2216 Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures[4]

D 2240 Test Method for Rubber Property—Durometer Hardness[5]

D 2487 Test Method for Classification of Soils for Engineering Purposes[4]

D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)[4]

D 3282 Practice for Classification of Soils and Soil-Aggregate Mixtures for Highway Construction Purposes[4]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[6]

E 319 Methods of Testing Single-Arm Balances[6]

E 898 Method of Testing Top-Loading, Direct-Reading Laboratory Scales and Balances[6]

3. Definitions

3.1 *Atterberg limits*—originally, seven "limits of consistency" of fine-grained soils were defined by Albert Atterberg. In current engineering usage, the term usually refers only to the liquid limit, plastic limit, and in some references, the shrinkage limit.

3.2 *consistency*—the relative ease with which a soil can be deformed.

3.3 *liquid limit (LL)*—the water content, in percent, of a soil at the arbitrarily defined boundary between the liquid and plastic states. This water content is defined as the water content at which a pat of soil placed in a standard cup and cut by a groove of standard dimensions will flow together at the base of the groove for a distance of 13 mm (½ in.) when subjected to 25 shocks from the cup being dropped 10 mm in a standard liquid limit apparatus operated at a rate of 2 shocks per second.

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[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vols 04.02, 04.03, and 04.08.

[4] *Annual Book of ASTM Standards*, Vol 04.08.

[5] *Annual Book of ASTM Standards*, Vol 09.01.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

NOTE 2—The undrained shear strength of soil at the liquid limit is considered to be 2 ±0.2 kPa (0.28 psi).

3.4 *plastic limit (PL)*—the water content, in percent, of a soil at the boundary between the plastic and brittle states. The water content at this boundary is the water content at which a soil can no longer be deformed by rolling into 3.2 mm (⅛ in.) in diameter threads without crumbling.

3.5 *plastic soil*—a soil which has a range of water content over which it exhibits plasticity and which will retain its shape on drying.

3.6 *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.

3.7 *liquidity index*—the ratio, expressed as a percentage, of (*1*) the natural water content of a soil minus its plastic limit, to (*2*) its plasticity index.

3.8 *activity number (A)*—the ratio of (*1*) the plasticity index of a soil to (*2*) the percent by weight of particles having an equivalent diameter smaller than 0.002 mm.

4. Summary of Method

4.1 The sample 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 sample 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, Procedures A and B, 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, Procedures C and D, 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 is no longer able to be pressed together and rerolled. The water content of the soil at this stage 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 This test method is used as an integral part of several engineering classification systems to characterize the fine-grained fractions of soils (see Test Method D 2487 and Practice D 3282) and to specify the fine-grained fraction of construction materials (see Specification D 1241). The liquid limit, plastic limit, and plasticity index of soils are also used extensively, either individually or together with other soil properties to correlate with engineering behavior such as compressibility, permeability, compactibility, shrink-swell, and shear strength.

5.2 The liquid and plastic limits of a soil can be used with the natural water content of the soil to express its relative consistency or liquidity index and can be used with the percentage finer than 2-µm size to determine its activity number.

5.3 The one-point liquid limit procedure is frequently used for routine classification purposes. When greater precision is required, as when used for the acceptance of a material or for correlation with other test data, the multipoint procedure should be used.

5.4 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.

5.5 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.

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 a hard rubber base. A drawing showing the essential features of the device and the critical dimensions is given in Fig. 1. The design of the device may vary provided that the essential functions are

preserved. The device may be operated either by a hand crank or by an electric motor.

6.1.1 *Base*—The base shall be hard rubber having a D Durometer hardness of 80 to 90, and a resilience such that an 8-mm (5/16-in.) diameter polished steel ball, when dropped from a height of 25 cm (9.84 in.) will have an average rebound of at least 80 % but no more than 90 %. The tests shall be conducted on the finished base with feet attached.

6.1.2 *Feet*—The base shall be supported by rubber feet designed to provide isolation of the base from the work surface and having an A Durometer hardness no greater than 60 as measured on the finished feet attached to the base.

6.1.3 *Cup*—The cup shall be brass and have a weight, including cup hanger, of 185 to 215 g.

6.1.4 *Cam*—The cam shall raise the cup smoothly and continuously to its maximum height, over a distance of at least 180° of cam rotation. The preferred cam motion is a uniformly accelerated lift curve. The design of the cam and follower combination shall be such that there is no upward or downward velocity of the cup when the cam follower leaves the cam.

NOTE 3—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*—The cup carriage shall be constructed in a way that allows convenient but secure adjustment of the height of drop of the cup to 10 mm (0.394 in.). The cup hanger shall be attached to the carriage by means of a pin which allows removal of the cup and cup hanger for cleaning and inspection.

6.1.6 *Optional Motor Drive*—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 grooving tool having dimensions shown in Fig. 2. The tool shall be made of plastic or noncorroding metal. The design of the tool may vary as long as the essential dimensions are maintained. The tool may, but need not, incorporate the gage for adjusting the height of drop of the liquid limit device.

6.3 *Gage*—A metal gage block for adjusting the height of drop of the cup, having the dimensions shown in Fig. 3. The design of the tool may vary provided the gage 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 (3/8 in.) wide, and without bevel or radius.

6.4 *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*—A balance readable to at least 0.01 g and having an accuracy of 0.03 g within three standard deviations within the range of use. Within any 15-g range, a difference between readings shall be accurate within 0.01 g (Notes 4 and 5).

NOTE 4—See Methods E 898 and E 319 for an explanation of terms relating to balance performance.

NOTE 5—For frequent use, a top-loading type balance with automatic load indication, readable to 0.01 g, and having an index of precision (standard deviation) of 0.003 or better is most suitable for this method. However, nonautomatic indicating equal-arm analytical balances and some small equal arm top pan balances having readabilities and sensitivities of 0.002 g or better provide the required accuracy when used with a weight set of ASTM Class 4 (National Bureau of Standards Class P) or better. Ordinary commercial and classroom type balances such as beam balances are not suitable for this method.

6.6 *Storage Container*—A container in which to store the prepared soil specimen that will not contaminate the specimen in any way, and which prevents moisture loss. 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 *Ground Glass Plate*—A ground glass plate at least 30 cm (12 in.) square by 1 cm (3/8 in.) thick for mixing soil and rolling plastic limit threads.

6.8 *Spatula*—A spatula or pill knife having a

blade about 2 cm (¾ in.) wide by about 10 cm (4 in.) long. In addition, a spatula having a blade about 2.5 cm (1 in.) wide and 15 cm (6 in.) long has been found useful for initial mixing of samples.

6.9 *Sieve*—A 20.3 cm (8 in.) diameter, 425-µm (No. 40) sieve conforming to the requirements of Specification E 11 and having a rim at least 5 cm (2 in.) above the mesh. A 2-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*—A thermostatically controlled oven, preferably of the forced-draft type, capable of continuously maintaining a temperature of 110 ±5°C throughout the drying chamber. The oven shall be equipped with a thermometer of suitable range and accuracy for monitoring oven temperature.

6.12 *Washing Pan*—A round, flat-bottomed pan at least 7.6 cm (3 in.) deep, slightly larger at the bottom than a 20.3-cm (8-in.) diameter sieve.

6.13 *Rod* (optional)—A metal or plastic rod or tube 3.2 mm (⅛ in.) in diameter and about 10 cm (4 in.) long for judging the size of plastic limit threads.

7. Materials

7.1 A supply of distilled or demineralized water.

8. Sampling

8.1 Samples may be taken from any location that satisfies testing needs. However, Methods C 702, and Practice D 75, and Recommended Practice D 420 should be used as guides for selecting and preserving samples from various types of sampling operations. Samples which will be prepared using the wet preparation procedure, 10.1, must be kept at their natural water content prior to preparation.

8.2 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.

8.3 Where data from this test method are to be used for correlation with other laboratory or field test data, use the same material as used for these tests where possible.

8.4 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 may be reduced by the methods of quartering or splitting. Cohesive samples 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. The following specific points should be checked:

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.

9.1.1.2 *Wear of Cup*—The cup must be replaced when the grooving tool has worn a depression in the cup 0.1 mm (0.004 in.) deep or when the edge 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.

9.1.2 *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. Sandy soils cause rapid wear of grooving tools; therefore, when testing these materials, tools should be inspected more frequently than for other soils. Any tool with a tip width greater than 2.1 mm must not be used. The depth

of the tip of the grooving tool must be 7.9 to 8.1 mm.

NOTE 6—The width of the tip of grooving tools is conveniently checked using a pocket-sized measuring magnifier equipped with a millimetre 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. 4 for proper location of the gage relative to the cup during adjustment.

NOTE 7—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 gage under the cup from the front, and observe whether the gage contacts the cup or the tape. See Fig. 4. If the tape and cup are both contacted, the height of drop is approximately correct. If not, adjust the cup until simultaneous contact is made. Check adjustment by turning the crank at 2 revolutions per second while holding the gage in position against the tape and cup. If a ringing or clicking sound is heard without the cup rising from the gage, the adjustment is correct. If no ringing is heard or if the cup rises from the gage, readjust the height of drop. If the cup rocks on the gage 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.

MULTIPOINT LIQUID LIMIT—PROCEDURES A AND B

10. Preparation of Test Specimens

10.1 *Wet Preparation*—Except where the dry method of specimen preparation is specified (10.2), prepare specimens for test as described in the following sections.

10.1.1 *Samples Passing the 425-µm (No. 40) Sieve*—When by visual and manual procedures it is determined that the sample has little or no material retained on a 425-µm (No. 40) sieve, prepare a specimen of 150 to 200 g by mixing thoroughly with distilled or demineralized water on the glass plate using the spatula. If desired, soak soil in a storage dish with small amount of water to soften the soil before the start of mixing. Adjust the water content of the soil to bring it to a consistency that would require 25 to 35 blows of the liquid limit device to close the groove (Note 8). 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 specimen through a 425-µm (No. 40) sieve using a piece of rubber sheeting, rubber stopper, or other convenient device provided the operation 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 methods 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 (No. 40) sieve, but remove by hand or by washing. Place the mixed soil in the storage dish, cover to prevent loss of moisture, and allow to stand for at least 16 h (overnight). After the standing period and immediately before starting the test, thoroughly remix the soil.

NOTE 8—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 — in may be needed for stiff, fat clays.

10.1.2 *Samples Containing Material Retained on a 425-µm (No. 40) Sieve:*

10.1.2.1 Select a sufficient quantity of soil at natural water content to provide 150 to 200 g of material passing the 425-µm (No. 40) sieve. Place in a pan or dish and add sufficient water to cover the soil. Allow to soak until all lumps have softened and the fines no longer adhere to the surfaces of the corase particles (Note 9).

NOTE 9—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 should tap water be 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 washing operations.

10.1.2.2 When the sample contains a large percentage of material 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 (No. 40) sieve in the bottom of the clean pan. Pour 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, pour the soil water mixture over a 2-mm (No. 10) sieve nested atop the 425-µm (No. 40) sieve, rinse the fine material through and remove the 2-mm (No. 10) 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 (No. 40) 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 (No. 40) 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 the air currents at ordinary room temperature, (*b*) exposing to warm air currents from a source such as an electric hair dryer, (*c*) filtering in a Buckner funnel or using filter candles, (*d*) decanting clear water from surface of suspension, or (*e*) draining in a colander or plaster of paris dish lined with high retentivity, high wet-strength filter paper.[7] If a plaster of paris dish is used, take care that the dish never becomes sufficiently saturated that it fails to actively absorb water into its surface. Thoroughly dry dishes between uses. During evaporation and cooling, stir the sample often enough to prevent overdrying of the fringes and soil pinnacles on the surface of the mixture. For soil samples containing soluble salts, use a method of water reduction such as *a* or *b* that will not eliminate the soluble salts from the test specimen.

10.1.2.4 Thoroughly mix the material passing the 425-µm (No. 40) sieve on the glass plate 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. The soil should be at a water content that will result in closure of the groove in 25 to 35 blows. Return the mixed soil to the mixing dish, cover to prevent loss of moisture, and allow to stand for at least 16 h. After the standing period, and immediately before starting the test, remix the soil thoroughly.

10.2 *Dry Preparation:*

10.2.1 Select sufficient soil to provide 150 to 200 g of material passing the 425-µm (No. 40) sieve after processing. Dry the sample 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 sample is not allowed to completely dry. However, the soil should have a dry appearance when pulverized. Pulverize the sample in a mortar with a rubber tipped pestal or in some other way that does not cause breakdown of individual grains. 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.

10.2.2 Separate the sample 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 (No. 40) sieve to the pulverizing apparatus and repeat the pulverizing and sieving operations as many times as necessary to assure that all finer material has been disaggregated and material retained on the 425-µm (No. 40) sieve consists only of individual sand or gravel grains.

10.2.3 Place material remaining on the 425-µm (No. 40) sieve after the final pulverizing operations in a dish and soak in a small amount of water. Stir the soil water mixture and pour over the 425-µm (No. 40) 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 (No. 40) sieve. Discard material retained on the 425-µm (No. 40) sieve.

10.2.4 Adjust the water content as necessary by drying as described in 10.1.2.3 or by mixing on the glass plate, using the spatula while adding increments of distilled or demineralized water,

[7] S and S 595 filter paper, available in 32-cm circles, has proven satisfactory.

until the soil is at a water content that will result in closure of the groove in 25 to 35 blows.

10.2.5 Put soil in the storage dish, cover to prevent loss of moisture and allow to stand for at least 16 h. After the standing period, and immediately before starting the test, thoroughly remix the soil (Note 8).

11. Procedure

11.1 Place a portion 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. Heap the unused soil on the glass plate and cover with the inverted storage dish or a wet towel.

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. 5. 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. 6.

NOTE 10—Use the end of the grooving tool, Fig. 2, or a scale 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 to 11.3. If the soil slides on the surface of the cup, repeat 11.1 through 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 angles to the groove and including that portion of the groove in which the soil flowed together, place in a weighed container, and cover.

11.6 Return the soil remaining in the cup to the glass plate. 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 on the glass plate 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 through 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 Method D 2216. Make all weighings on the same balance. Initial weighings should be performed immediately after completion of the test. If the test is to be interrupted for more than about 15 min, the specimens already obtained should be weighed at the time of the interruption.

12. Calculations

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 the 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. Computa-

tional 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—PROCEDURES C AND D

13. Preparation of Test Specimens

13.1 Prepare the specimen in the same manner as described in Section 10, except that at mixing, adjust the water content to a consistency requiring 20 to 30 drops of the liquid limit cup to close the groove.

14. Procedure

14.1 Proceed as described in 11.1 through 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.

14.2 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 operations. Repeat 11.2 through 11.5, and, 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. Otherwise, remix the entire specimen and repeat.

NOTE 11—Excessive drying or inadequate mixing will cause the number of blows to vary.

14.3 Determine water contents of specimens as described in 11.8.

15. Calculations

15.1 Determine the liquid limit for each water content specimen using one of the following equations:

$$LL = W_N\left(\frac{N}{25}\right)^{0.121} \quad \text{or}$$

$$LL = K(W_N)$$

where:

N = the number of blows causing closure of the groove at water content,

W_N = water content, and

K = a factor given in Table 1.

The liquid limit is the average of the two trial liquid limit values.

15.2 If the difference between the two trial liquid limit values is greater than one percentage point, repeat the test.

PLASTIC LIMIT

16. Preparation of Test Specimen

16.1 Select a 20-g 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 test. Reduce the water content of the soil to a consistency at which it can be rolled without sticking to the hands by spreading and mixing continuously on the glass plate. 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, such as hard surface paper toweling or high wet strength filter paper.

17. Procedure

17.1 From the 20-g mass, select a portion of 1.5 to 2.0 g. Form the test specimen into an ellipsoidal mass. Roll this 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 (Note 12). The thread shall be further deformed on each stroke so that its diameter is continuously reduced and its length extended until the diameter reaches 3.2 ±0.5 mm (0.125 ±.020 in.), taking no more than 2 min (Note 13). The amount of hand or finger pressure required will vary greatly, according to the soil. Fragile soils of low plasticity are best rolled under the outer edge of the palm or at the base of the thumb.

NOTE 12—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 13—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, especially for inexperienced operators.

17.1.1 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 reroll. Continue this alternate rolling to a thread 3.2 mm in diameter, gathering together, kneading and rerolling, until the thread crumbles under the pres-

sure required for rolling and the soil can no longer be rolled into a 3.2-mm diameter thread (See Fig. 7). 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 they are able to 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.

17.2 Gather the portions of the crumbled thread together and place in a weighed container. Immediately cover the container.

17.3 Select another 1.5 to 2.0 g portion of soil from the original 20-g specimen and repeat the operations described in 17.1 and 17.2 until the container has at least 6 g of soil.

17.4 Repeat 17.1 through 17.3 to make another container holding at least 6 g of soil. Determine the water content, in percent, of the soil contained in the containers in accordance with Method D 2216. Make all weighings on the same balance.

NOTE 14—The intent of performing two plastic limit trials is to verify the consistency of the test results. It is acceptable practice to perform only one plastic limit trial when the consistency in the test results can be confirmed by other means.

18. Calculations

18.1 Compute the average of the two water contents. If the difference between the two water contents is greater than two percentage points, repeat the test. The plastic limit is the average of the two water contents.

PLASTICITY INDEX

19. Calculations

19.1 Calculate the plasticity index as follows:

$$PI = LL - PL$$

where:

LL = the liquid limit,

PL = the plastic limit.

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.

20. Report

20.1 Report the following information:

20.1.1 Sample identifying information,

20.1.2 Any special specimen selection process used, such as removal of sand lenses from undisturbed sample,

20.1.3 Report sample as airdried if the sample was airdried before or during preparation,

20.1.4 Liquid limit, plastic limit, and plasticity index to the nearest whole number and 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.

20.1.5 An estimate of the percentage of sample retained on the 425-µm (No. 40) sieve, and

20.1.6 Procedure by which liquid limit was performed, if it differs from the multipoint method.

21. Precision and Bias

21.1 No interlaboratory testing program has as yet been conducted using this test method to determine multilaboratory precision.

21.2 The within laboratory precision of the results of tests performed by different operators at one laboratory on two soils using Procedure A for the liquid limit is shown in Table 2.

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	0.974
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

TABLE 2 Within Laboratory Precision for Liquid Limit

	Average Value, $\bar{x}$	Standard Deviation, s
Soil A:		
PL	21.9	1.07
LL	27.9	1.07
Soil B:		
PL	20.1	1.21
LL	32.6	0.98

DIMENSIONS

LETTER	A Δ	B Δ	C Δ	E Δ	F	G	H	J Δ	K Δ	L Δ	M Δ
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 Δ	V	W	Z			
MM	24	28	24	45	47 ± 1.0	3.8	13	6.5			

Δ 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	0.945 R
300	0.974 R
330	0.995 R
360	1.000 R

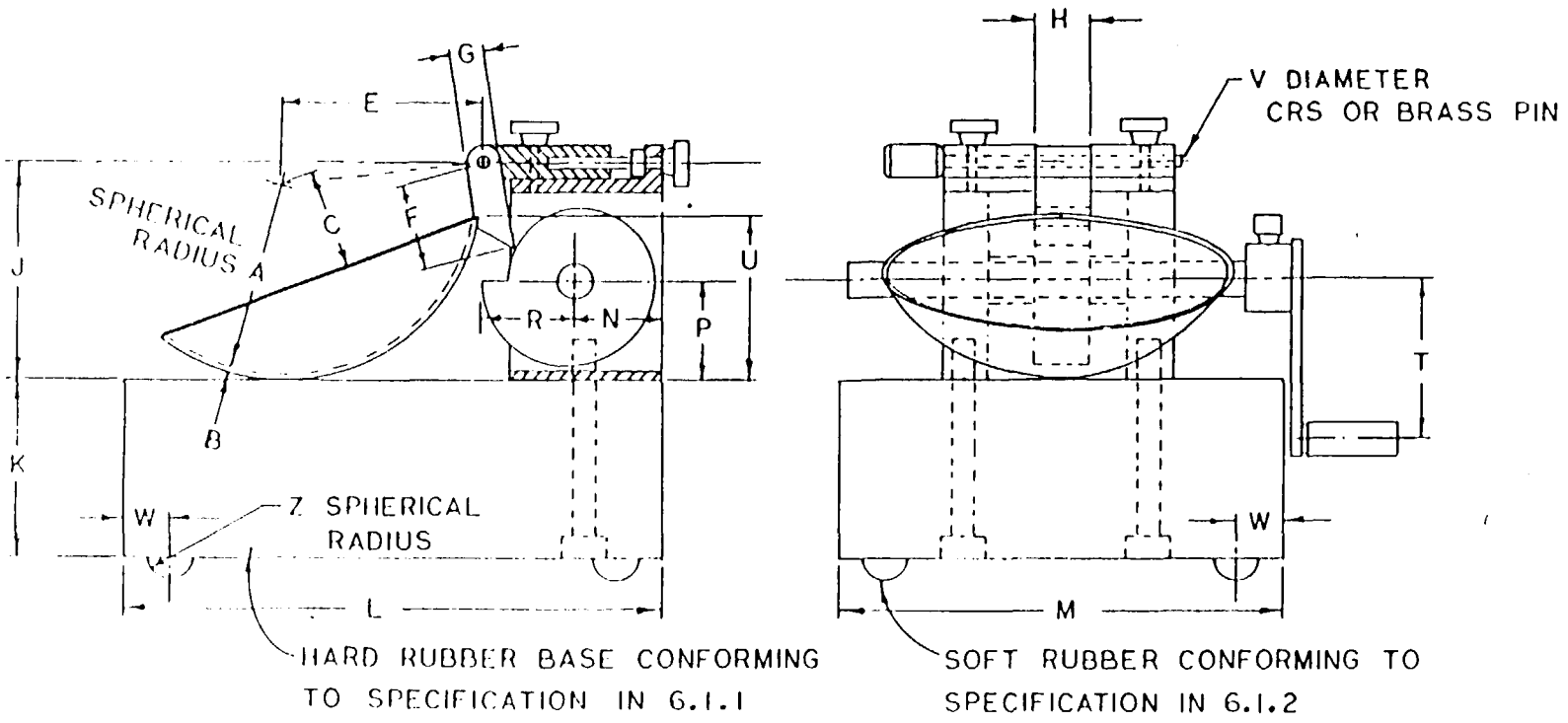


FIG. 1 Hand-Operated Liquid Limit Device

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

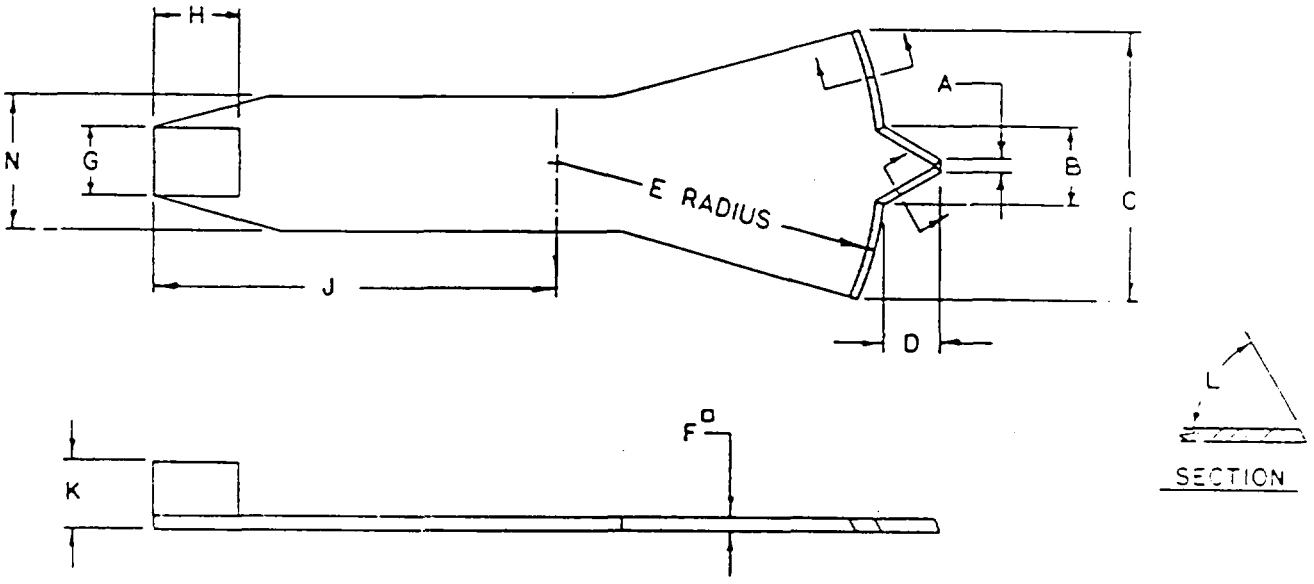
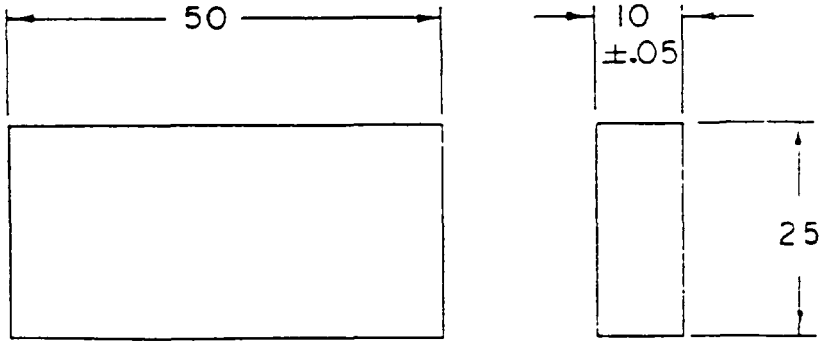


FIG. 2 Grooving Tool (Optional Height-of-Drop Gage Attached)



DIMENSIONS IN MILLIMETRES

FIG. 3 Height of Drop Gage

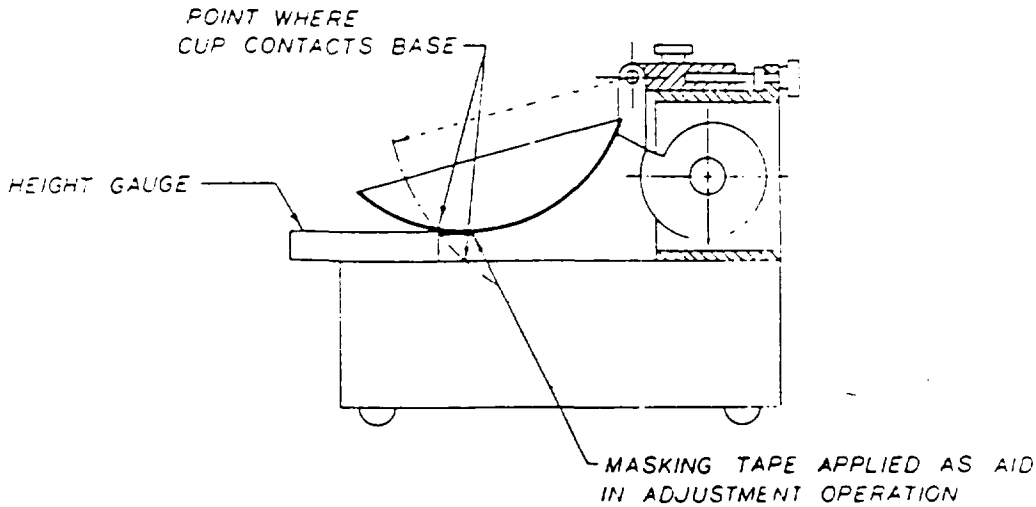


FIG. 4 Calibration for Height of Drop

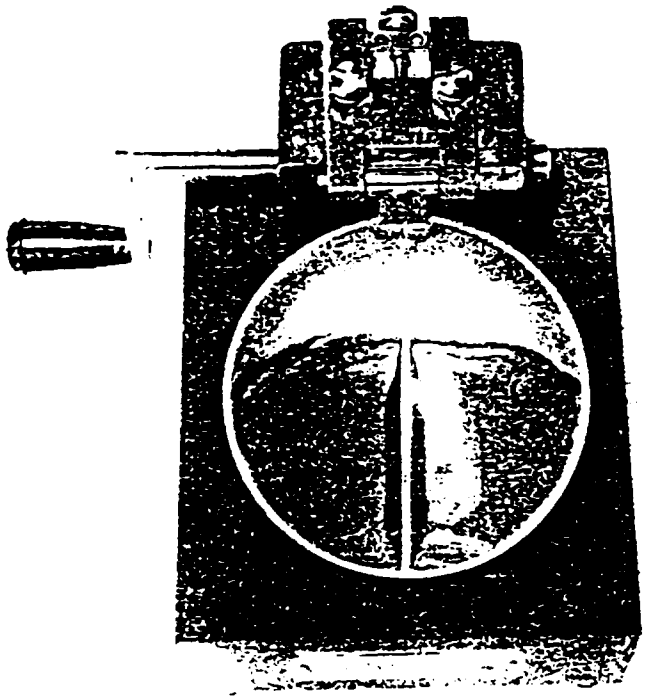


FIG. 5 Grooved Soil Pat in Liquid Limit Device

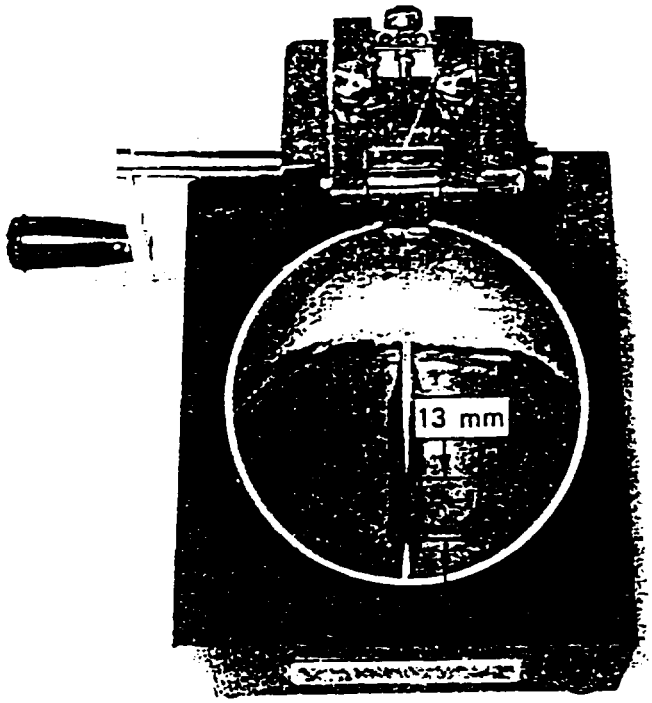


FIG. 6 Soil Pat After Groove Has Closed

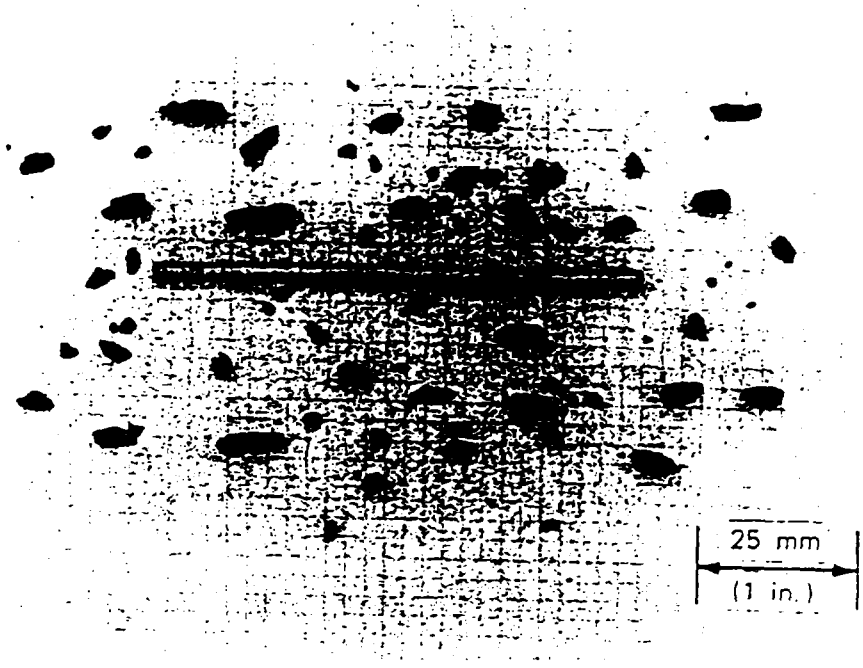


FIG. 7 Lean Clay Soil at the Plastic Limit

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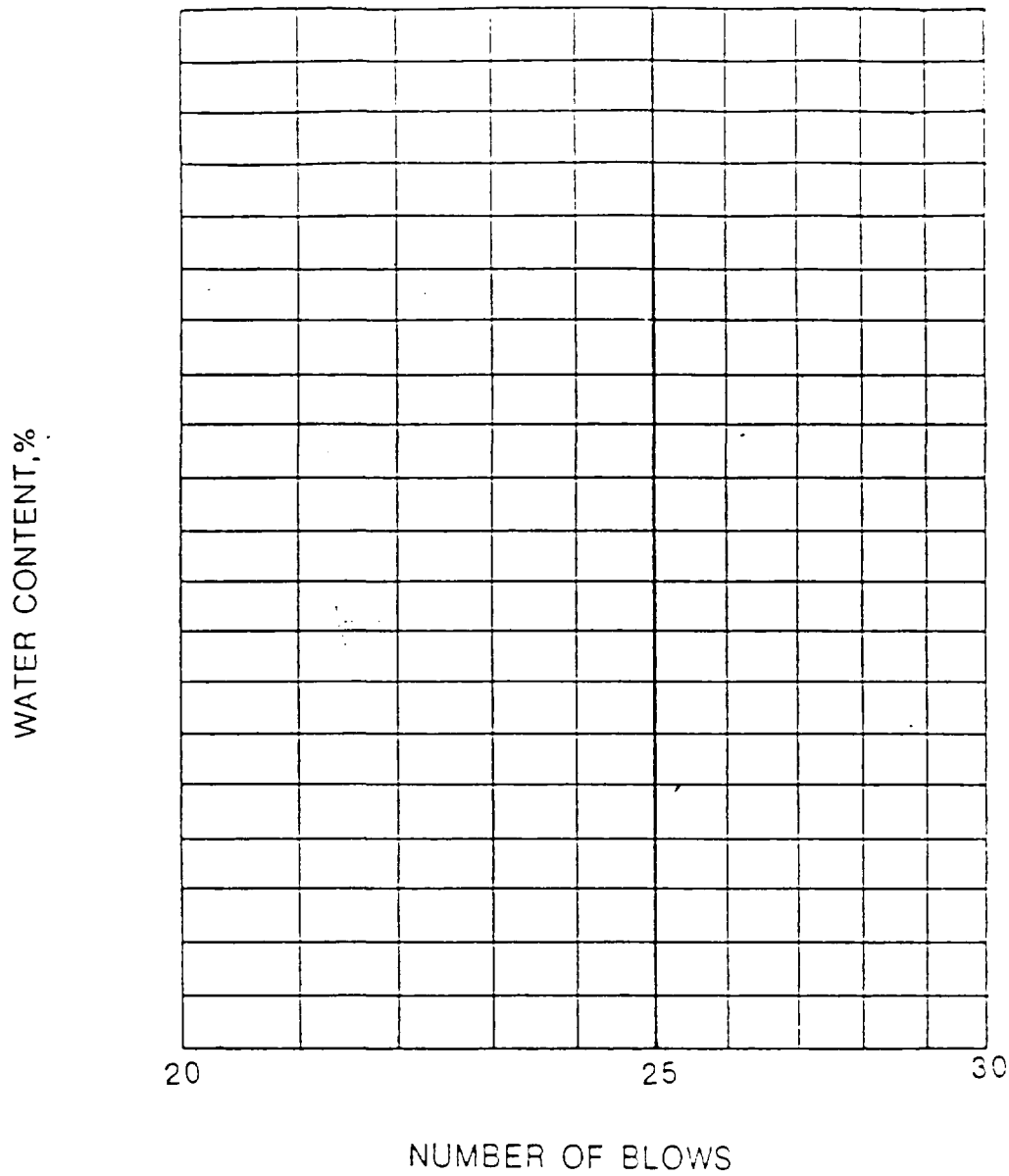
Y _____ DATE _____ JOB NAME _____ JOB NO. _____

CHECKED BY _____ _____ SHEET _____ OF _____

	SAMPLE NO.								
	MATERIAL.								
UNCONFINED COMPR.	DIA, in.								
	INITIAL HT, in.								
	FINAL HT, in.								
	MAX LOAD, lbs.								
	Qa, tsf.								
	Qu, tsf.								
UNIT WEIGHT	DIA, in.								
	LENGTH, in.								
	WEIGHT, gms.								
	1 + NM								
	DRY UNIT WEIGHT pcf.								
NATURAL MOIST.	CAN NO.								
	WET WT, gms.								
	DRY WT, gms.								
	TARE, gms.								
	% MOISTURE.								
LIQUID LIMIT	CAN NO.								
	WET WT, gms.								
	DRY WT, gms.								
	TARE, gms.								
	BLOW MULT								
	% MOISTURE.								
PLASTIC LIMIT	CAN NO.								
	WET WT, gms.								
	DRY WT, gms.								
	TARE. gms.								
	% MOISTURE								
LOSS ON IGNITION	CUP NO.								
	START WT, gms.								
	END WT, gms.								
	TARE, gms.								
	% LOSS.								

REMARKS:

LIQUID LIMIT TEST



WARZYN

DWN	CHK'D	APP'D	DATE

Standard Test Method for SHRINKAGE FACTORS OF SOILS[1]

This standard is issued under the fixed designation D 427; 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.

[ε1] NOTE—Paragraph 6.3.1 was editorially added in April 1984.

1. Scope

1.1 This test method covers tests for obtaining the data from which the following subgrade soil constants may be calculated: shrinkage limit, shrinkage ratio, volumetric shrinkage, linear shrinkage, and specific gravity (approximate).

1.2 The values stated in inch-pound units are to be regarded as the standard.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precaution statements, see 6.3.1.

2. Applicable Document

2.1 *ASTM Standard:*

D421 Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[2]

3. Significance and Use

3.1 The shrinkage factors covered in this test method can only be determined on basically fine-grained (cohesive) soils which exhibit a dry strength when oven dried.

3.2 The term "shrinkage limit", expressed as a water content in percent, is typically assumed to represent the amount of water required to fill the voids of a given cohesive soil at its minimum void ratio obtained by drying (usually oven). Thus, the concept shrinkage limit can be used to evaluate the shrinkage potential, or possibility of development, or both, of cracks in earthworks involving cohesive soils.

4. Apparatus

4.1 *Evaporating Dish*, porcelain, about 5½ in. (139.7 mm) in diameter.

4.2 *Spatula*, or pill knife having a blade about 3 in. (76.2 mm) in length and about ¾ in (19.0 mm) in width.

4.3 *Shrinkage Dish*—A circular porcelain or monel metal milk dish having a flat bottom and being about 1¾ in. (44.4 mm) in diameter and about ½ in. (12.7 mm) in height.

4.4 *Straightedge*, steel, about 6 in. (150 mm) in length.

4.5 *Glass Cup*, about 2¼ in. (57.2 mm) in diameter and about 1¼ in. (31.8 mm) in height, the top rim of which is ground smooth and is in a plane essentially parallel with the bottom of the cup.

4.6 *Glass Plate*, with three metal prongs for immersing the soil pat in mercury, as shown in Fig. 1.

4.7 *Graduate*, glass, having a capacity of 25 mL and graduated to 0.2 mL.

4.8 *Balance*, sensitive to 0.1 g.

4.9 *Mercury*, sufficient to fill the glass cup to overflowing.

5. Sampling

5.1 Take a sample weighing about 30 g from the thoroughly mixed portion of the material passing the No. 40 (425-μm) sieve which has been obtained in accordance with Practice D 421.

[1] This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D 18.03 on Texture, Plasticity, and Density Characteristics of Soils.

Current edition approved Nov. 28, 1983. Published January 1984. Originally issued as D 427 – 35. Last previous edition D 427 – 61 (1974).

[2] *Annual Book of ASTM Standards*, Vol 04.08.

6. Procedure

6.1 Place the sample in the evaporating dish and thoroughly mix with distilled water in an amount sufficient to fill the soil voids completely and to make the soil pasty enough to be readily worked into the shrinkage dish without the inclusion of air bubbles. The amount of water required to furnish friable soils with the desired consistency is equal to or slightly greater than the liquid limit, and the amount necessary to furnish plastic soils with the desired consistency may exceed the liquid limit by as much as 10 %.

6.2 Coat the inside of the shrinkage dish with a thin layer of petroleum jelly or some other heavy grease to prevent the adhesion of the soil to the dish. Place an amount of the wetted soil equal to about one third the volume of the dish in the center of the dish, and cause the soil to flow to the edges by taping the dish on a firm surface cushioned by several layers of blotting paper or similar material. Add an amount of soil approximately equal to the first portion, and tap the dish until the soil is thoroughly compacted and all included air has been brought to the surface. Add more soil and continue the tapping until the dish is completely filled and excess soil stands out about its edge. Strike off the excess soil with a straightedge, and wipe off all soil adhering to the outside of the dish.

6.3 Weigh the dish immediately after it is filled and struck off and record the mass as the mass of dish and wet soil. Allow the soil pat to dry in air until the color of the pat turns from dark to light. Oven-dry the pat to constant mass at 230 ±9°F (110 ±5°C), the mass being recorded as the mass of dish and dry soil. Determine and record the mass of the empty dish. Determine the capacity of the dish in cubic centimetres, which is also the volume of the wet soil pat, by filling the dish to overflowing with mercury, removing the excess by pressing a glass plate firmly over the top of the dish, and measuring by means of a glass graduate the volume of mercury held in the dish (see 6.3.1). Record this volume as the volume of the wet soil pat, V.

6.3.1 CAUTION: Mercury is a hazardous substance which can cause serious health effects from prolonged inhalation of the vapor or contact with the skin. The following precautions should be followed whenever mercury is used in the laboratory: (1) Always store mercury in a shatterproof, sealed container, (2) Always work with mercury in a well ventilated area, preferably under a fume hood, (3) Avoid direct contact with mercury liquid; wear rubber gloves if direct contact with mercury is unavoidable, (4) Prevent uncontrolled spills by performing that part of the procedure requiring the use of mercury in a large pan which can act as a catchment should mercury be spilled during the procedure, and (5) Uncontrolled spills must be cleaned up as thoroughly as possible; mercury spill clean-up materials may be necessary and can be obtained from laboratory supply companies.

6.4 Determine the volume of the dry soil pat by removing the pat from the shrinkage dish and immersing it in the glass cup full of mercury in the following manner: Fill the glass cup to overflowing with mercury and remove the excess mercury by pressing the glass plate with the three prongs (Fig. 1) firmly over the top of the cup. Carefully wipe off any mercury that may be adhering to the outside of the cup. Place the cup, filled with mercury, in the evaporating dish and place the soil pat on the surface of the mercury. Carefully force the pat under the mercury by means of the glass plate with the three prongs (Fig. 1) and press the plate firmly over the top of the cup. It is essential that no air be trapped under the soil pat. Measure the volume of the mercury so displaced in the glass graduate and record as the volume of the dry soil pat, V_0.

7. Calculation of Moisture Content

7.1 Calculate the moisture content of the soil at the time it was placed in the dish expressed as a percentage of the dry mass of the soil as follows:

$$w = [(W - W_0)/W_0] \times 100$$

where:

w = moisture content of the soil when placed in the dish,

W = mass of wet soil pat obtained by subtracting the mass of the shrinkage dish from the mass of the dish and wet pat, and

W_0 = mass of dry soil pat obtained by subtracting the mass of the shrinkage dish from the mass of the dish and dry pat.

8. Shrinkage Limit

8.1 The shrinkage limit of a soil is the maximum water content at which a reduction in water content will not cause a decrease in the volume of the soil mass.

8.2 Calculate the shrinkage limit, SL, from the data obtained in the volumetric shrinkage determination, as follows:

$$SL = w - [(V - V_0)\rho_w / W_0] \times 100]$$

where:

SL = shrinkage limit,

w = moisture content of wet soil, in percentage of the mass of oven-dried soil,

V = volume of wet soil pat, ft^3 (cm^3),

V_0 = volume of dry soil pat, ft^3 (cm^3),

W_0 = mass of oven-dried soil pat, lb (g), and

ρ_w = density of water, 62.4 lb/ft^3 or 1.0 g/cm^3.

8.3 *Optional Method*—When both the specific gravity, G_s, and the shrinkage ratio, R, are known, calculate the shrinkage limit as follows:

$$SL = [(1/R) - (1/G_s)] \times 100$$

9. Shrinkage Ratio

9.1 The shrinkage ratio of a soil is the ratio of a given volume change, expressed as a percentage of the dry volume, to the corresponding change in water content above the shrinkage limit, expressed as a percentage of the mass of oven-dried soil.

9.2 Calculate the shrinkage ratio, R, from the data obtained in the volumetric shrinkage determination by the following equation:

$$R = W_0/V_0 \times \rho_w$$

10. Volumetric Shrinkage

10.1 The volumetric shrinkage of the soil is the decrease in volume, expressed as a percentage of the soil mass when dried, of a soil mass when the water content is reduced from a given percentage to the shrinkage limit.

10.2 The volumetric shrinkage, V_s, shall be calculated from the data obtained in the volumetric shrinkage determination by the following equation:

$$V_s = (w_1 - SL)R$$

where:

w_1 = given percentage of water content,

SL = shrinkage limit, and

R = shrinkage ratio.

11. Linear Shrinkage

11.1 The linear shrinkage of a soil is the decrease in one dimension of a soil mass expressed as a percentage of the original dimension, when the water content is reduced from a given value to the shrinkage limit.

11.2 The linear shrinkage, L_s, shall be obtained by means of the following equation:

$$L_s = 100\,[1 - \sqrt[3]{100/(V_s + 100)}]$$

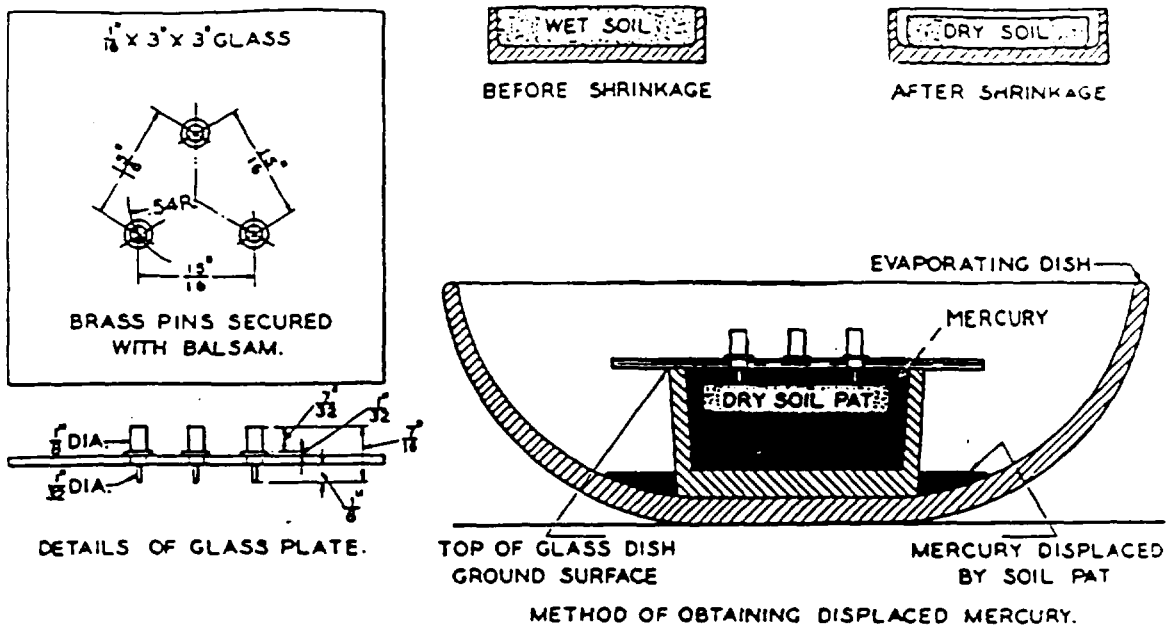
12. Specific Gravity

12.1 The specific gravity of the soil solids, G_s, may be calculated from the data obtained in the volumetric shrinkage test by the following equation:

$$G_s = 1/[(1/R) - (SL/100)]$$

13. Precision and Bias

13.1 Requirements for the precision and bias of this test method have not yet been developed.



Metric Equivalents

in.	1/32	1/16	1/8	7/32	7/16	15/16	3
mm	0.8	1.6	3.2	5.6	11.1	23.8	76.2

FIG. 1 Apparatus for Determining the Volumetric Change of Subgrade Soils

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103.

SHRINKAGE LIMIT TEST

Project _____ Job No. _____

Location of Project _____ Boring No. _____ Sample No. _____

Description of Soil _____ Depth of Sample _____

Tested By _____ Date of Testing _____

Wt. of coated dish + wet soil = _____ g

Wt. of coated dish + dry soil = _____ g

Wt. of coated dish = _____ g

Wt. of soil, W_s = _____ g

Wt. of Water, W_w = _____ g

Water content, w_o% = _____ g

Vol. of Wet Soil, V_o = _____ cu cm (Step 5 of procedure: Vol. of shrinkage dish = V_o)

Vol. of Dry Soil, V_f = _____ cu cm (Step 7 of procedure)

Shrinkage limit, $w_s = w_o - \frac{(V_o - V_f)\,\gamma_w}{W_s} \times 100 =$ _____

Shrinkage ratio, $SR = W_s / V_f$ = _____

WARZYN

APPENDIX B-15

GRAIN SIZE (WARZYN)

Standard Test Method for Particle-Size Analysis of Soils[1]

This standard is issued under the fixed designation D 422; 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.

[ε1] NOTE—Section 19 was added editorially in September 1990.

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 (Notes 1 and 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:*

D 421 Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

E 100 Specification for ASTM Hydrometers[4]

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[5] (Note 3) conforming to the general details shown in Fig. 3 (Notes 4 and 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 E 100. 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).

3.6 *Sieves*—A series of sieves, of square-mesh woven-wire cloth, conforming to the requirements of Specification E 11. A full set of sieves includes the following (Note 6):

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

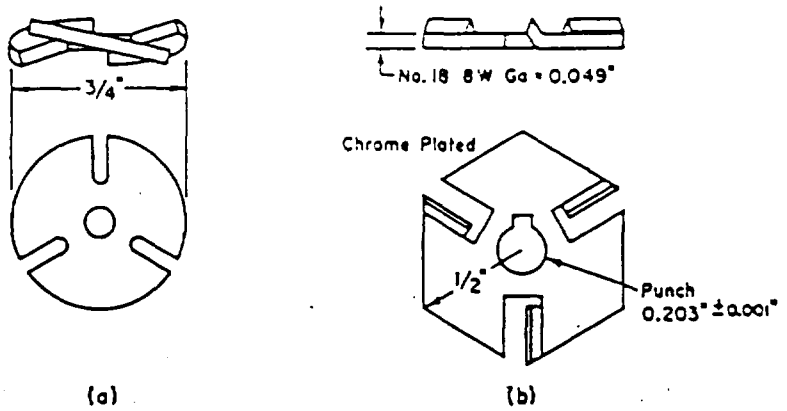
Current edition approved Nov. 21, 1963. Originally published 1935. Replaces D 422 - 62.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] Detailed working drawings for this cup are available at a nominal cost from the American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103. Order Adjunct No. 12-404220-00.



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

3-in. (75-mm)	No. 10 (2.00-mm)
2-in. (50-mm)	No. 20 (850-μm)
1½-in. (37.5-mm)	No. 40 (425-μm)
1-in. (25.0-mm)	No. 60 (250-μm)
¾-in. (19.0-mm)	No. 140 (106-μm)
⅜-in. (9.5-mm)	No. 200 (75-μm)
No. 4 (4.75-mm)	

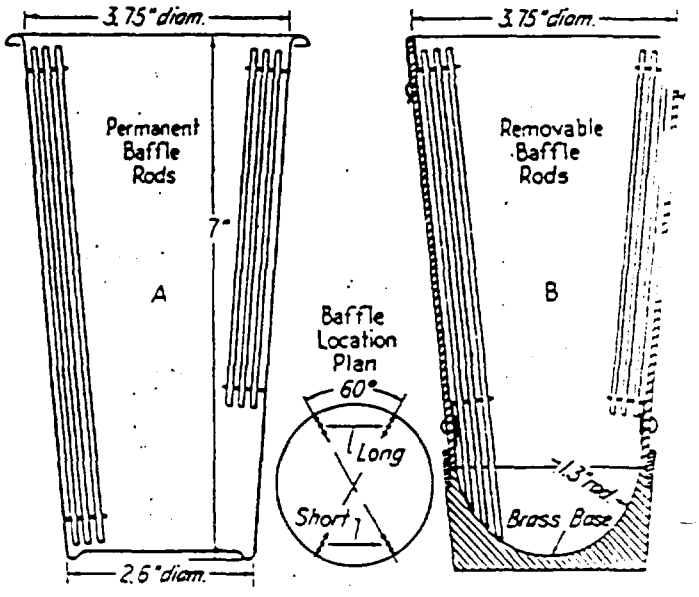
NOTE 6—A set of sieves giving uniform spacing of points for the graph, as required in Section 17, may be used if desired. This set consists of the following sieves:

3-in. (75-mm)	No. 16 (1.18-mm)
1½-in. (37.5-mm)	No. 30 (600-μm)
¾-in. (19.0-mm)	No. 50 (300-μm)
⅜-in. (9.5-mm)	No. 100 (150-μm)
No. 4 (4.75-mm)	No. 200 (75-μm)
No. 8 (2.36-mm)	

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.



Metric Equivalents

in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus

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 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.

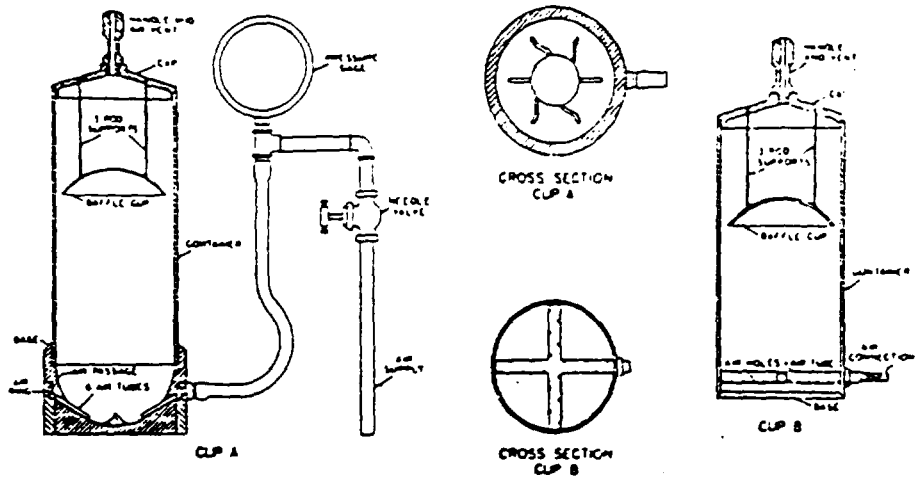


FIG. 3 Air-Jet Dispersion Cups of Apparatus B

5. Test Sample

5.1 Prepare the test sample for mechanical analysis as outlined in Practice D 421. 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 D 421, 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.

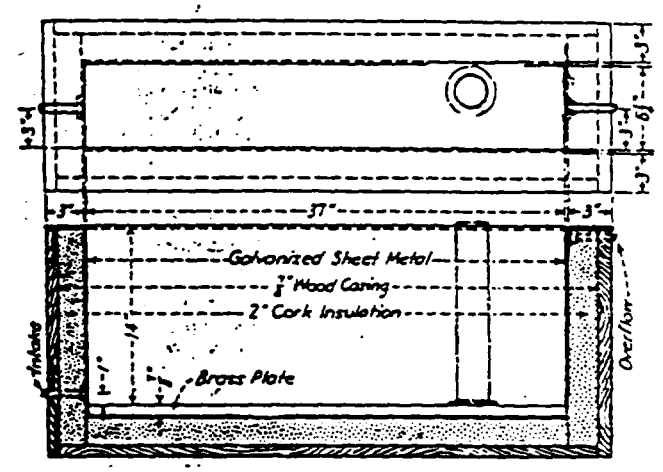
5.2 Provision is made in Section 5 of Practice D 421 for weighing of the air-dry soil selected for purpose of tests, the separation of the soil on the No. 10 sieve by dry-sieving and washing, and the weighing of the washed and dried fraction retained on the No. 10 sieve. From these two masses the percentages 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.



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

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 cyclinder 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 wi 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 samp further, using either stirring apparatus A or B. If stirring apparatus A is used, transfer the soil - water slurry from t beaker into the special dispersion cup shown in Fig 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 mo than half full. Stir for a period of 1 min.

NOTE 9—A large size syringe is a convenient device for handling th water in the washing operation. Other devices include the wash-wa bottle and a hose with nozzle connected to a pressurized distilled wa tank.

9.4 If stirring apparatus B (Fig. 3) is used, remove th cover cap and connect the cup to a compressed air supply means of a rubber hose. A air gage must be on the lin between the cup and the control valve. Open the contro valve so that the gage indicates 1 psi (7 kPa) pressure (No 10). Transfer the soil - water slurry from the beaker to t 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, b 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 mixtu 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 dispers for only 1 min. After the dispersion period, reduce the ga pressure to 1 psi preparatory to transfer of soil - water slur to the sedimentation cylinder.

10. Hydrometer Test

10.1 Immediately after dispersion, transfer the soil - wate slurry to the glass sedimentation cylinder, and add distil or demineralized water until the total volume is 1000 mL

10.2 Using the palm of the hand over the open end of t cylinder (or a rubber stopper in the open end), turn th cylinder upside down and back for a period of 1 min complete the agitation of the slurry (Note 11). At the end 1 min set the cylinder in a convenient location and ta hydrometer readings at the following intervals of ti (measured from the beginning of sedimentation), or as mo as may be needed, depending on the sample or the specifi tion for the material under test: 2, 5, 15, 30, 60, 250, an 1440 min. If the controlled water bath is used, the sedime tation cylinder should be placed in the bath between the and 5-min readings.

NOTE 11—The number of turns during this minute should approximately 60, counting the turn upside down and back as two tu

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.

TABLE 1 Values of Correction Factor, a, 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.

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:

$$P = [(100\,000/W) \times G/(G - G_1)](R - G_1)$$

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:

$$P = (Ra/W) \times 100$$

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),

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 P 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:

$$D = \sqrt{[30n/980(G - G_1)] \times L/T}$$

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 (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_l = specific gravity (relative density) of suspending medium (value may be used as 1.000 for all practical purposes).

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:

$$D = K\sqrt{L/T}$$

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 a

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.[illegible]
1.001	16.0	1	16.1	32	11.[illegible]
1.002	15.8	2	16.0	33	10.[illegible]
1.003	15.5	3	15.8	34	10.[illegible]
1.004	15.2	4	15.6	35	10.[illegible]
1.005	15.0	5	15.5		
1.006	14.7	6	15.3	36	10.[illegible]
1.007	14.4	7	15.2	37	10.[illegible]
1.008	14.2	8	15.0	38	10.[illegible]
1.009	13.9	9	14.8	39	9.[illegible]
1.010	13.7	10	14.7	40	9.[illegible]
1.011	13.4	11	14.5	41	9.[illegible]
1.012	13.1	12	14.3	42	9.[illegible]
1.013	12.9	13	14.2	43	9.[illegible]
1.014	12.6	14	14.0	44	9.[illegible]
1.015	12.3	15	13.8	45	8.9
1.016	12.1	16	13.7	46	8.9
1.017	11.8	17	13.5	47	8.9
1.018	11.5	18	13.3	48	8.[illegible]
1.019	11.3	19	13.2	49	8.[illegible]
1.020	11.0	20	13.0	50	8.[illegible]
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	7.5
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	6.9
1.029	8.6	29	11.5	59	6.5
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:

$$L = L_1 + \tfrac{1}{2}[L_2 - (V_B/A)]$$

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, cm³, and

A = cross-sectional area of sedimentation cylinder, cm².

Values used in calculating the values in Table 2 are as follows:

For both hydrometers, 151H and 152H:

L_2 = 14.0 cm

V_B = 67.0 cm³

A = 27.8 cm²

For hydrometer 151H:

L_1 = 10.5 cm for a reading of 1.000

= 2.3 cm for a reading of 1.031

For hydrometer 152H:

L_1 = 10.5 cm for a reading of 0 g/litre

= 2.3 cm for a reading of 50 g/litre

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	0.01510	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

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 ..... %

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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Standard Test Method for Amount of Material in Soils Finer Than the No. 200 (75-µm) Sieve[1]

This standard is issued under the fixed designation D 1140; 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. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

[ε1] NOTE—Editorial changes were made throughout in September 1990.

1. Scope

1.1 This test method covers determination of the total amount of material in soils finer than the No. 200 (75-µm) sieve.

2. Referenced Documents

2.1 *ASTM Standards:*

D 422 Method for Particle-Size Analysis of Soils[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

3. Apparatus

3.1 *Sieves*—A nest of two sieves, the lower being a No. 200 (75-µm) sieve and the upper a No. 40 (425-µm) sieve, both conforming to ASTM Specification E 11.

3.2 *Containers*—A pan or vessel of sufficient size to contain the test sample covered with water and to permit vigorous agitation without advertent loss of any part of the sample, and a second pan or container for use in drying the test sample after washing.

4. Test Sample

4.1 The test sample shall be selected from material that has been thoroughly mixed. A representative sample, sufficient to yield not less than the approximate weight of dried material shown in the following table, shall be selected using a sample splitter or by the method of quartering:

Nominal Diameter of Largest Particle, in.	Approximate Minimum Weight of Sample, g
0.0787 (No. 10 sieve) (2.0 mm)	200
0.187 (No. 4 sieve) (4.75 mm)	500
¾ (19.0 mm)	1500
1 (25.0 mm)	2000
1½ or over (37.5 mm)	2500

5. Procedure

5.1 Dry the test sample to a constant weight at a temperature not exceeding 230 ± 9 F (110 ± 5 C) and weigh to the nearest 0.05 percent, or alternatively, weigh the test sample moist and use an auxiliary moisture content sample to determine the moisture content of the sample. The weight of the moisture content sample shall be between 20 and 30 percent of the weight of the test sample. Calculate the oven-dry weight of the test sample from the moist weight and the moisture content.

5.2 Place the test sample in the container, add sufficient clean water to cover it, and allow to soak a minimum of 2 h (preferably overnight).

5.3 Agitate the contents of the container vigorously and pour the wash water immediately over the nested sieves, arranged with the coarser sieve on top. Repeat the process of adding clear water to the container to cover the sample, agitating the contents of the container, and pouring the wash water over the nested sieves until the wash water is clear. When the total sample is small, the entire contents of the soaking container may be transferred to the nested sieves after the first washing and the washing operation completed in accordance with 5.4. The wash water need not be saved.

NOTE 1—The percentage value secured at the end of the test may not be correct (being too low) for soils containing relatively high percentages of the minus 200 fraction. This appears to be due chiefly to inadequate agitation. When it is desired to secure the exact percentage for the minus 200 fraction for such a soil, the portion of the sample passing the No. 40 sieve and retained on the No. 200 sieve secured in the washing operation, shall be transferred to the dispersion cup of the stirring apparatus used in Method D 422. The cup filled half full with water and the contents agitated for 1 min. After this agitation the contents of the cup shall be transferred to the nested sieves and washing continued.

If the stirring apparatus has not been used prior to the drying of the portion of the sample larger than the No. 200 (75-µm) sieve, and it is desired to do so after drying, the dried material shall be separated on the No. 40 (425-µm) sieve; the portion retained shall be saved; and the portion passing shall be placed in the dispersion cup with water and agitated for 1 min with the stirring apparatus as previously described. The contents of the cup shall be transferred to the No. 200 sieve, washed, and dried. The revised total weight retained on the No. 200 sieve shall be secured by combining and weighing the two fractions.

5.4 Transfer the sample to the nested sieves and wash with running water (Note 2). When the sample is larger than can be handled at one time on the nested sieves, wash a portion of the sample and transfer to the container in which it is to be dried.

NOTE 2—Tapping of sieves has been found to expedite the washing operations.

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity and Density Characteristics of Soils.

Current edition approved Sept. 15, 1954. Originally issued 1950. Replaces D 1140 - 50 T.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vols 04.01, 04.02, 04.06, 05.05, and 14.02.

5.5 Dry the washed material retained on the nested sieves in a container to a constant weight at a temperature not exceeding 230 ± 9 F (110 ± 5 C) and dry-sieve it on the nested sieves (Note 3). Weigh the dry material retained on the nested sieves to the nearest 0.05 percent.

NOTE 3—Some material passes the No. 200 (75-μm) sieve on dry sieving that did not pass during the washing operation. When desired, a sieve analysis may be made on the portion of the sample retained on the No. 200 sieve, in accordance with Method D 422.

6. Calculation

6.1 Calculate the results as follows:

$$P = [(W_o - W_1)/W_o] \times 100$$

where:

P = percentage of material finer than No. 200 (75-μm) sieve,

W_o = weight of original sample on an oven-dry basis, g and

W_1 = oven-dry weight of sample after washing and dry-sieving, g.

7. Keywords

7.1 grain-size; No. 200 sieve; particle-size; sieve analysis

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Test Sieving: Principles and Procedures

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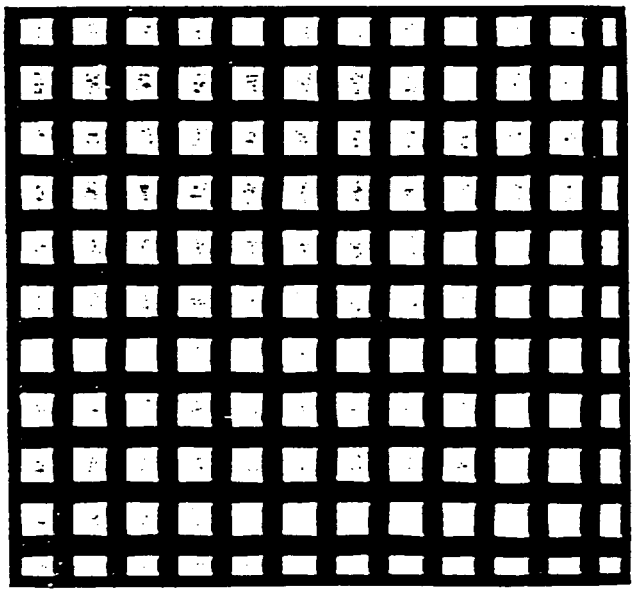
WHAT IS SIEVING?

A simplistic definition of sieving is the removal of fine material from coarse material by means of a meshed or perforated vessel. Professor Terence Allen characterizes sieving as "The aperture of a sieve may be regarded as a series of gauges which reject or pass particles as they are presented to the aperture." (1) This theory was actually in practice during the early Egyptian era as grains were sized with 'sieves' of woven reeds and grasses.

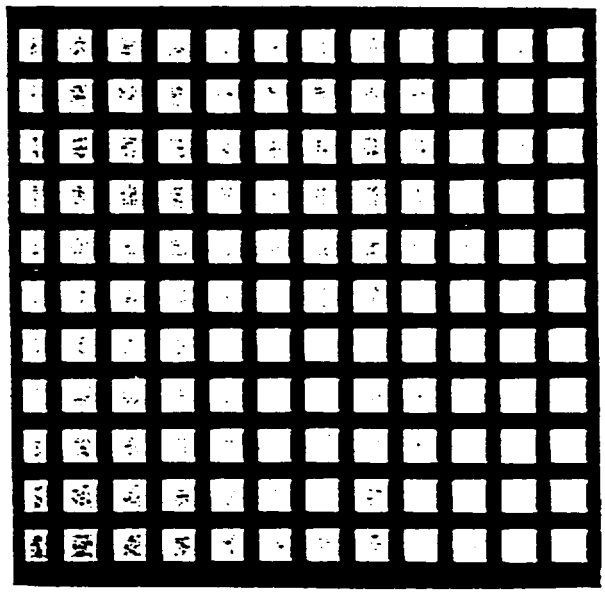
The level of sophistication increased with the rise of the industrial society. As requirements for sized material rose, technology in producing uniform sieving media increased. Woven wire cloth was introduced as an alternative providing greater accuracy and durability. At present, this woven cloth is available in a range of sizes from 5" openings to 20 micrometer openings. All mesh sizes are covered by an array of both national and international standards of tolerance.

The need for particle size anaylsis in the finer size ranges (i.e. 38 micrometers and less) prompted the development of the electrodeposited sieve. These sieves, sometimes called electroformed or micromesh, are currently being produced with openings as fine as 5 micrometers. The mesh openings are extremely uniform in both size and shape and maintain a tolerance of a maximum of 2 micrometer variation.

While the area of sieve analysis has come a long way since the reed sieves of ancient Egypt, few new developments have come along since the 1940's. Professor Kurt Leschonski wrote "Sieve analysis is one of the few methods of particle size analysis which has escaped modernisation (sic)." (2) While the modernization has not come in the actual hardware of sieving, refinements in the application and utilization of existing equipment has proceeded.



WOVEN SIEVE CLOTH
Variations in opening size and shape are common.



ELECTROFORMED SIEVE CLOTH
Electrodeposited material showing uniformity in opening size and shape.

CHAPTER 2

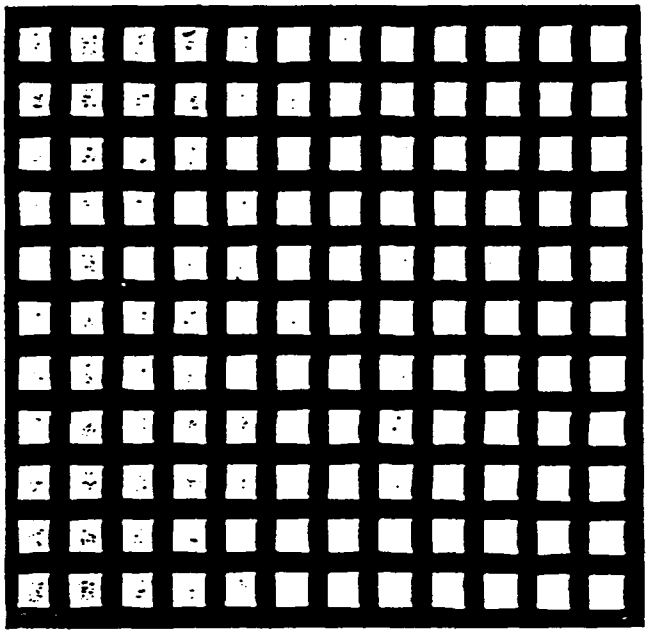
USES, LIMITATIONS AND ADVANTAGES

Harold Heywood wrote "I often refer to sieving as the 'Cinderella' of particle size analysis methods; it does most of the hard work and gets little consideration."(3)

There are numerous reasons for the selection of testing sieves as a first choice in particle size anaylsis work. Leschonski put it "...because of its simplicity — everyone immediately understands the purpose of a stack of sieves and its operation — and its inexpensiveness." (4)Standard sieve analysis is probably the fastest and most widely used quality control procedure in any powder process control industry. Used frequently as a mediating device between the production and sales divisions of a process corporation or between the sales force and the customer, test sieve analysis work enjoys the universality of being the best 'quick and dirty' test procedure for rapid particle size distribution data. The outcome of the analysis is easily calculated and interpreted for comparison between laboratories. Start-up cost to institute a basic sieving quality control program is minimal, and operators of most levels of training are capable of performing a successful sieve analysis. With these factors in mind, it is easy to see why testing sieves are as ubiquitous as they are in industry. Materials from crushed ore chunks of over 4½" in diameter to slurried alumina and porcelain powders of less than 20 micrometers are all analyzed with test sieves on a regular basis.

Whether hand or machine sieving, wet or dry preparations, analysis or production work, testing sieves have found a niche in the quality control laboratory. Given this overall acceptance of test sieves as a viable analytical device and the widespread presence of the sieve in laboratories in all industries, any shortcomings of such an analysis device would be magnified. For all of the advantages available to the test sieve user, limitations must be recognized and accounted for in data presentation and analysis.

Test sieves are individuals. Being fabricated of a woven mesh material, variations in the weave are common. The chances of locating two sieves of identical distribution of opening sizes are extremely remote. Because of these variations, reproducibility of test results between sieves is adversely affected. The stringent standards imposed by ASTM, ISO or other regulating bodies have built tolerance factors which allow for the variations in the weave while striving to maintain a level of uniformity in 'test grade' sieve cloth. (See Table 1.)



CLOTH WITH WIDE MESH VARIATIONS

Alternating areas of narrow and wide mesh openings can significantly change sieve analysis results.

With this distribution of opening sizes present, some smaller than the nominal and some larger, the time interval of the sieve analysis becomes extremely important. If, for example, a sieve has several openings far above the tolerance for the particular mesh size, and the test is run for 30 minutes, the probability of larger-than-nominal particles finding those oversize openings is much greater than if the test was run for only 15 minutes. Similarly, if the sample of powder contains a large percentage of elongated or needlelike particles, the longer the test interval, the greater the likelihood that the elongated particles will orient themselves 'on end' and pass through the openings. If the sieve cloth has a wide range of opening sizes, the sieving of such a material has a compounded error.

Another factor which must be carefully analyzed is the reaction of the material to ambient conditions. The most accurate test sieve available would be of

USES, LIMITATIONS AND ADVANTAGES (cont.)

minimal use if the relative humidity in the test lab was 99%. Likewise, extremely dry conditions can cause fine powders to adhere to the sieve parts and each other with strong electrostatic charges. Other types of sieving problems will be discussed in the glossary section.

To minimize the error unavoidably introduced by the wire cloth weavers, steps must be taken at every step of fabrication that assure the uniformity of the woven mesh as well as the compliance with the applicable standards. Both the weaver and the test sieve manfacturer must keep up a constant monitoring program of actual opening sizes of the wire cloth as well as the uniformity of those openings. The loss to the manufacturers in rejected sieve cloth is a gain to the end-user in uniformity and compliance.

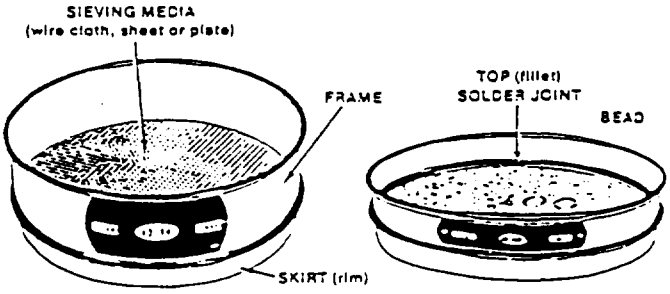
COMPARATOR
Profile projector specially designed and built for wire cloth and sieve inspection.

CHAPTER 3

WORKING GLOSSARY OF SIEVING TERMS

Sieving terminology is frequently used and abused in writing up specifications for materials. Listed below are some of the most frequently used terms and a general discussion of their meaning:

Agglomerate: natural tendency of materials to clump or ball together. This condition is very common in materials with high fat or oil content or those with fibrous or extremely irregular topography.



FULL HEIGHT
Height 2⅞", depth to cloth 2"

HALF HEIGHT
Height 1⅞", depth to cloth 1"

Blinding: plugging of the screen openings with particles either exactly the same size as the sieve opening or by fine particles which build up on the meshes and eventually close off the openings. Frequently referred to as pegging. (Photo Page 4)

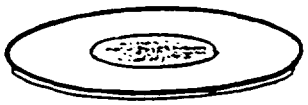
Cover: stamped or spun lid which tightly covers the top of a sieve to prevent the loss of the material sample during sifting.

Electrostatic charges: accumulation of electrical charges on the particles and sieve parts causing clinging, agglomeration or blinding. This condition is frequently seen in hydrocarbon based materials, plastics, reactive metals, paint pigments and powders with a large fraction below 20 micrometers.

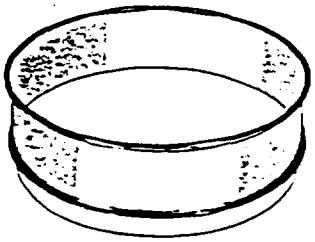
Extended rim pan: a sieving pan with a skirt designed to nest within a sieve stack, allowing multiple tests to be performed simultaneously. Frequently called a nesting pan or spacer.

WORKING GLOSSARY OF SIEVING TERMS (cont.)

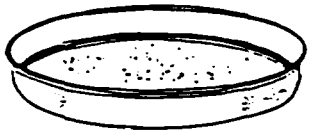
COVER WITH LIFTING RING



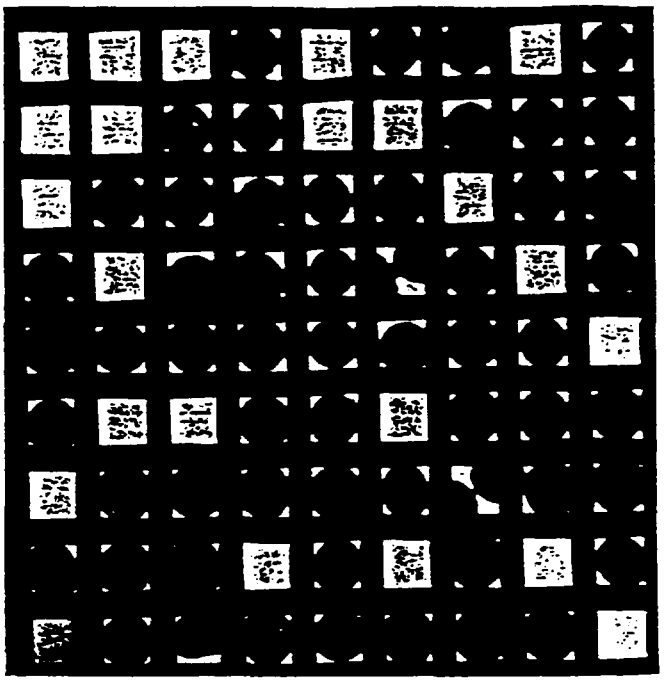
COVER WITHOUT LIFTING RING



EXTENDED RIM PAN
(Spacer)



PAN
(Receiver)



BLINDED SIEVE
Spherical and near-size particles can blind or peg in the sieve openings.

Flow additive: powdered substance added to the sample to reduce agglomeration, neutralize static charges and improve the flow characteristics of the sample. Common additives are fine silica, activated charcoal, talc, and other commercially produced synthetic substances. Generally, the additive is prescreened to a known average particle size, blended with the sample (approximately 1% additive by weight) and then screened.

Frame: rigid sidewall used to form body of the testing sieve. Common depths are 2" (full height) and 1" (half height). Special application sieves of other depths are also in use.

Mesh: screening medium with openings of uniform size and shape fabricated of woven, punched or electrodeposited material.

Pan: stamped or spun receiver of materials passing through the finest sieve.

Skirt: section of test sieve below the sieve mesh which allows for mating or nesting of the sieves in a test stack.

Support mesh: coarse sieve cloth mounted under fine sieve cloth in a test sieve to provide extra strength. This is widely used in wet sieving operations to protect the fragile fine sieve cloth. Frequently called backing cloth or rolled backing cloth.

Test Sieve: screening medium (mesh) with openings of uniform size and shape mounted on a rigid frame, usually for laboratory testing or small scale production applications. The frames can be made of various materials, the most common of which are brass and stainless steel in a cylindrical configuration having a diameter of 3", 5", 6", 8", 10", 12" or larger.

Wet sieving: the separation of fines from the coarse portion of a sample while suspended in an aqueous solution introduced to a testing sieve. The liquid medium is used to negate static charges, break down agglomerates and lubricate near-size particles. After the fines have been washed through the sieve, the residue is oven-dried and reweighed.

CHAPTER 4

SIEVE SPECIFICATIONS — Domestic and International

The U.S. Sieve Series is a metric system based series first suggested by the American Society of Testing and Materials in 1913. The opening sizes in this sieve series are in the ratio of the fourth root of two. This numerical relationship was first suggested by Professor P.R. Rittinger, a German researcher, in 1867.

In the fourth root of two series, every opening size is 1.189 times the opening size of the next smaller sieve. This relationship continues into sieve opening area measurement. The U.S. Sieve Series provides that the area of each sieve opening size is 1½ times the area of the preceding sieve size.

By using every other sieve in this number series, the relationship becomes based on the square root of two (1.414), with the area of the opening being twice that of the preceding sieve size. Thus, by skipping two sizes, you create an area ratio of 3 to 1, or by skipping three sizes, you create a ratio of 4 to 1.

When selecting sieves from this series, any number of sieves can be used for an analysis. Care must be taken in selecting every sieve between two points, every other sieve, every fourth sieve, etc., to keep within the mathematical progression of the series.

After World War II, the International Standards Organization (ISO) was formed in an attempt to establish world standards. Though the U.S. Sieve Series had proven to be effective and was in use throughout the world, members of the ISO would not accept the U.S. Sieve Series as a world standard. Rather, they chose to adopt the Preferred Number Series based on the roots of ten. The Preferred Number Series was suggested by Charles Renard of France in 1879. His system is based on the tenth, twentieth and fortieth roots of ten (designated R-10, R-20 and R-40). See Table 2.

Compromise was reached between the ISO and the proponents of the U.S. Sieve Series when it was discovered that every third value in the R-40/3 table is in a step ratio of 1.885, sufficiently close to the fourth root of two (1.1892) used by the U.S. Sieve Series. In 1970, slight adjustments were made in the U.S. Sieve Series to align the series perfectly with the ISO specifications.

Copies of these tables of specifications can be found in Table 3.

CHAPTER 5

SIEVE CALIBRATION PROCEDURES

Quantifying and accounting for variations in test sieve results have become two of the most important topics in particle technology today. Once again, the ubiquitous nature of stacks of test sieves in powder labs around the world has contributed to scope of the dilemma in sieve standardization and calibration. Kaye states "The inaccuracies and the uncertainties of characterization by sieve fractionation arise from the experimental problems of determining the sieve residues and from the nonideal nature of the sieving surfaces." Further, "The presence of a range of aperture sizes in any real sieving surface is a source of error in sieve based characterization studies since the theoretical or nominal size of the sieve is taken to be the boundary limit for the sieve residue." (5)

Not only is the test sieve user plagued with variations in the weave of the cloth, but confronted with the effects of particle shape on sieving results. Nearly 50 years ago, A.M. Gaudin wrote "Powders with identical size distributions, densities and chemical composition may behave quite differently as a result of variations in particle shape between samples. For example, powders consisting solely of spherical particles are likely to have good flow properties, while powders containing needlelike particles will not." Further, "In addition, it is impossible to isolate the concepts of particle size

SIEVE CALIBRATION PROCEDURES (cont.)

and shape, since the method of size measurement will influence the particle size which is determined."[6]

Numerous approaches have been tried to compensate for the effects of variations in wire cloth and particle shape. The methods have fallen into 3 basic categories: 1) inspection of the mesh to determine opening size, 2) material testing of the sieves to determine if sieves fall within performance specifications, and 3) a combination of methods 1 and 2, assuring compliance with both opening size and performance specifications.

Probably the most elementary of the inspection methods is the use of the etched glass slide. This procedure relies on what is referred to as the 'Moire Effect' which compares the number of meshes per inch in the wire cloth sample to the number of lines per inch etched on the glass slide. By microscopically measuring the wire diameters, a rough estimate of the opening size can be approximated. One major shortcoming of this procedure is the assumption that all wire diameters within the sample are the same. A slight variation in wire diameter can translate to a significant change in opening size.

An alternative to this measurement approach is the use of a high-powered optical comparator or profile projector. In this method, powerful light sources illuminate the mesh from both above and below and project the image onto a glass screen. Calibrated micrometer stages move the mesh sample in relation to a reference point allowing measurements with accuracy of 1 micrometer to be made on both the opening and wire diameter. The results are displayed on a numerical readout. The broad field of view of the comparator allows for a scan of a large number of sieve openings, facilitating a more comprehensive picture of the nature of the sieve cloth.

In material testing of sieves, powder samples are run on subject sieves and the residue calculated. These values are then compared with other sieves in selecting what some refer to as 'matched' sieves. There are a number of shortcomings in this procedure also. The first and foremost problem encountered is that of compliance. Conceivably, it is possible to find hundreds of sieves that will provide the same performance data when tested with a reference material and still not meet ASTM standards. While the sieves perform comparably, the fact that they do not meet the basic criteria of ASTM specifications should remove them from use as a U.S. Standard sieve. Another problem encountered with material matching is the use of reference samples which are different in shape, size or density than the users' products. For example, a manufacturer of spherical steel shot would yield significantly different results on a sieve that had been matched with an angular ground silica material. In this case, both shape and density are considerably different. The key to proper matching is the use of the user's own product or a material which approximates the product most closely.

The final approach is a combination of the first two methods. First, the sieve is checked optically for compliance with all applicable standards. Openings and wire diameters are physically measured, not averaged. After the sieve opening distribution has been characterized and evaluated, actual material testing can begin. During the material testing, samples of the user's own product are used for the standardization procedure. All tests are run for repeatability and the variation between test results calculated. This procedure yields a testing sieve with known values in the two most essential parameters — compliance with specifications and performance in an 'in vivo' setting.

An alternative that has been used with some success is the use of correction factors between sieves. Once a 'master set' of sieves has been established, a reference sample is tested on the stack. The values are calculated and retained. As new sieves are purchased, the original reference sample is tested on the new set and the values calculated. Any variations between the sieve stacks can be compensated for with correction factors or multipliers. For example, a sieve in stack 3 may retain more or less than the comparable sieve in the master set. A multiplier of magnitude greater than or less than 1 is necessary to calculate the comparable retention value on that sieve when compared to the master set. In this way, every sieve in use can be compared to the master set to standardize sieving results. Whatever method you use, it is essential that your starting point is grounded on the firm foundation of ASTM specifications. The compliance is necessary to assure uniformity between and within industries.

PERFORMING THE SIEVE ANALYSIS

In obtaining meaningful sieve analysis data, three major steps must take place. The first step is the preparation of the sample material, the second step is the performance of the actual sieving technique and the final step is the computation, presentation and analysis of the data.

Sample extraction and preparation is the most commonly overlooked variable in sieve standardization programs. Testing bias can be added at many places along the progression from material production to data analysis. The way the samples are extracted from the original bulk volume varies with the way the materials are produced or stored. The ideal sampling method is one which produces the most representative sample with the least amount of material required.

The following paragraphs were first published in the ASTM technical publication STP 447A. The collaborative efforts of the authors have produced a section on sampling technique which cannot be improved upon. (7)

Sampling from a chute or belt

Good accuracy in sampling is obtained where material is flowing from a chute or belt conveyor. The ideal place to take the sample is just where the material drops from the chute or belt. When taking the sample, if the stream is small enough, use a pail or other suitable receptacle which can be swung completely across the flowing stream in a brief interval of time and with a uniform movement. Under no circumstances should the sampling receptacle be allowed to overflow, because the overflow would tend to reject a higher proportion of the larger particles that exist in a representative sample. Mechanical sampling devices are available for selecting samples automatically from a stream at uniform spaced intervals of time.

Sampling from carload shipments of course bulk material

For coarse materials, such as crushed stone and gravel, shipped in railroad cars, a recommended method is to dig three or more trenches at least 1 foot (30.48 cm) deep and approximately 1 foot (30.48 cm) wide at the bottom. Equal portions are taken at seven equally spaced points along the bottom of the trench by pushing a shovel downward into the material and not by scraping horizontally. Samples from trucks, barges, or boats should be taken in the same manner as from railroad cars, except that the number of trenches should be adjusted to size of the transportation unit and tonnage involved.

Sampling from carload shipments of fine bulk materials

One established method for sampling a carload of bulk granular material is to take eight equal samples, (approximately 700 to 1000 grams each) from the bottom of a 1 foot (30.48 cm) conical excavation. Samples should be suitably spaced to represent the length and width of the car and then combined into a single gross sample.

Sampling bulk shipments of fine material with a sampling tube

An alternate and simpler method of sampling a carload, or other bulk quantity of fine or granular material is by use of a sampling tube which, for this purpose, should be 1½ inches (38.1 mm) by approximately 6 feet (1.829 m). Five or six insertions of the tube will produce approximately, a 2 pound (907-g) sample.

Sampling from a carload of bagged material

One method of sampling a carload of material shipped in bags is to select, at random, a number of bags equal to the cube root of the total number of bags in the car and to take suitable portions (800 to 1000 grams for minus 6 mm material) from each of the selected bags for a combined gross sample.

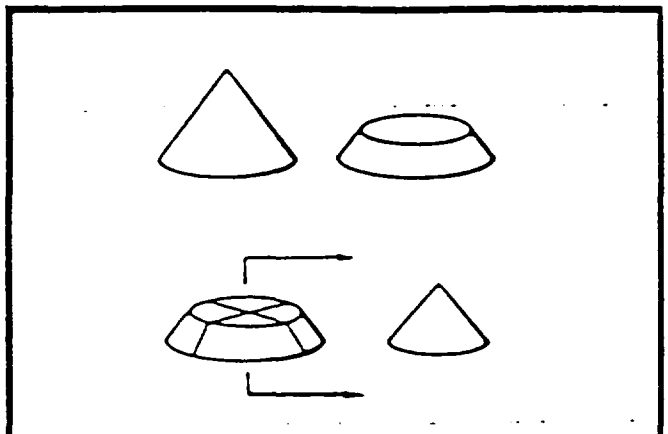
Sampling from a pile

In sampling from a pile, particularly material like crushed stone or coal containing large particles, it is extremely difficult to secure samples that are truly representative. At the apex of a conical pile, the proportion of fines will be greater, while at the base, the percentage of coarse particles will be greater. Therefore, neither location will be representative of the whole. In a shoveling process, every fifth or tenth shovel, etc., should be taken depending on the amount of the sample desired. The sample should consist of small quantities taken at random from as many parts of the pile as are accessible and taken in a manner that the composite will have the same grading as the larger amount.

Reduction of gross sample to test size for sieve analysis

After the gross sample has been properly taken, the next step is to reduce it to a suitable size for the sieve analysis test without impairing in any way the particle size distribution

PERFORMING THE SIEVE ANALYSIS (cont.)



CONING AND QUARTERING TECHNIQUE

characteristics of the original sample. This phase of the operation should follow the applicable procedures described in the succeeding sections, and should be performed with as much care as was used in the collection of the gross sample and in making the sieve test.

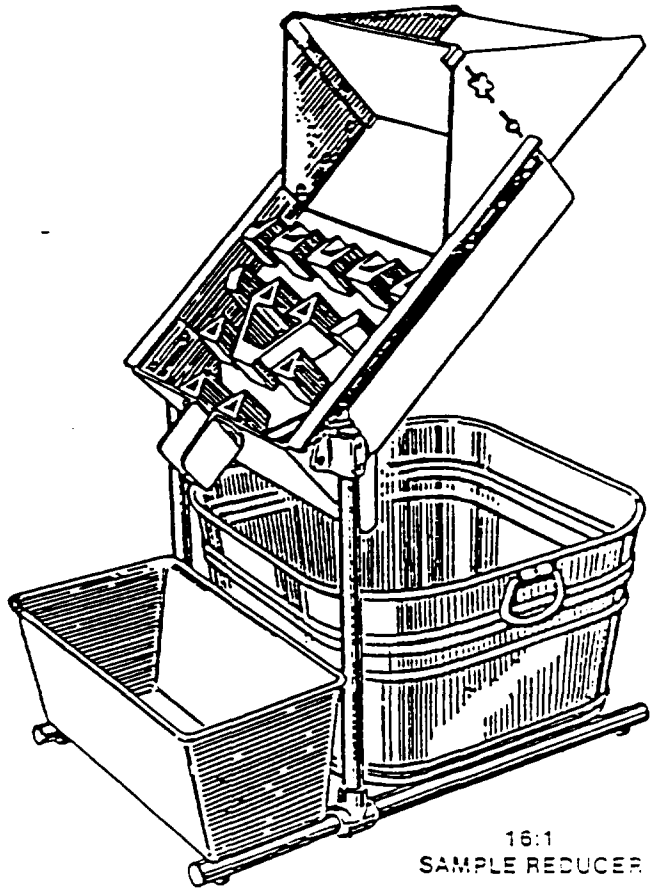
Coning and quartering

Pile the gross sample in a cone, place each shovelful at the apex of the cone, and allow it to run down equally in all directions. This will mix the sample. Then spread the sample in a circle and walk around the pile, gradually widening the circle with a shovel until the material is spread to a uniform thickness.

Mark the flat pile into quarters, and reject two opposite quarters. Mix again into a conical pile, taking alternate shovelfuls from the two quarters saved. Continue the process of piling, flattening, and rejecting two quarters until the sample is reduced to the required size.

Sample splitters and reducers

Gross samples, if not too large, may be reduced to test sample size by one or more passes through a sample splitter or Jones type riffler, which will divide a sample in half while maintaining the particle size distribution of the original sample. By repeated passes, the sample can be split into quarters, eighths, etc., until the size of the sample desired is obtained. For larger gross samples, sample reducers are available which will select a representative 16 part with a single pass. By just two passes through such a unit, a representative 1 pound sample can be obtained from an original 256 pounds. Three passes will give a 1 pound sample from two tons of material. Always make sure that the passages in the splitter or reducer are at least three times the size of the largest particle in the sample. Do not attempt to arrive at exactly the amount of material specified for the test. If a 50 gram sample is desired, arrive as near to this amount as practicable, because it will make no difference in the test percentage results whether the sample is slightly larger or smaller. In attempting to arrive at an exact weight, the tendency is to discriminate by the removal of sizes which are not representative of the whole, thus destroying the representative quality of the sample.



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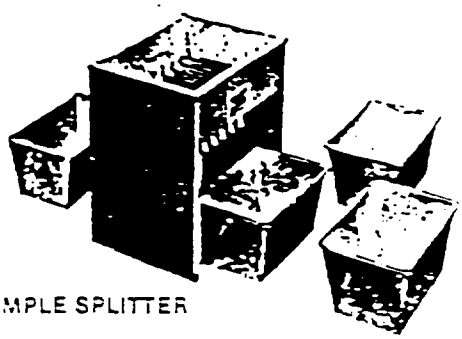
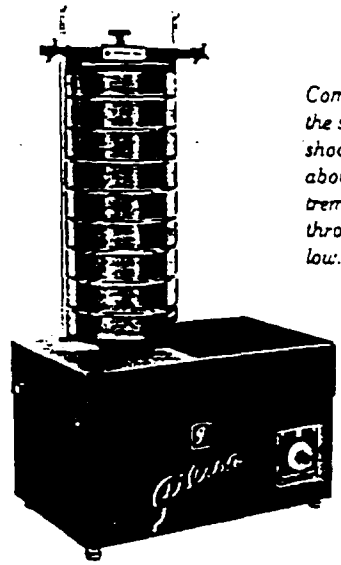
SAMPLE REDUCER



SAMPLE SPLITTER

PERFORMING THE SIEVE ANALYSIS (cont.)



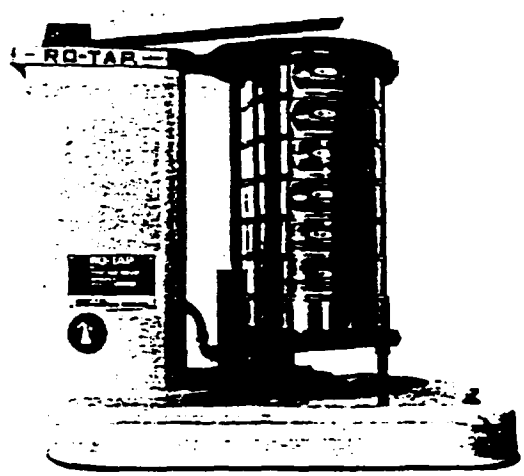
SIEVE SHAKER
Combined circular motion of the sieve stack with alternating shock from both below and above makes this shaker extremely effective from #4 mesh through #200 mesh and below.

Size of Sample in the Test

There is a natural tendency, although incorrect, to use too large a sample in the test. In most cases, the smaller the sample the more accurate the analysis. Testing sieves are the same as a go or no go gauge and too large a sample does not permit all the particles to present themselves to the screen surface.

Generally speaking a 25 to 100 gram sample should be used, however, if it is necessary to establish the correct sample size follow the following procedure. With a sample splitter accurately split, select samples of various weights such as 25, 50, 100 and 200 grams. Run these various sample sizes on a selected nest of sieves for a period of five minutes preferably on a mechanical sieve shaker. If the test with the 100 gram sample shows approximately the same percentage passing the finest sieve as the 50 gram sample, whereas the 200 gram sample shows a lower percentage, this would indicate that the 200 gram sample is too large and the 100 gram sample would be satisfactory. Then run the 100 gram sample on the same set of sieves for the same sieving time to see if you get repetitive results. Once the correct size sample has been determined use this on all sieve tests.

A useful table of recommended sample sizes for tests with 200 mm or 8" diameter sieves is Table 4. Note that the table gives sample sizes listed by volume. Recommended sample weights in grams can be determined by multiplying the values in Column 3 and 4 by the bulk density (grams per cubic centimeter) of the material to be tested rounded out within a reasonable tolerance. If the actual bulk density of a certain material is not known, the typical density factor for the most nearly similar material listed in Table 5, may be used.



RO-TAP SIEVE SHAKER
Combined circular motion of the sieve stack with shock from above the sieve stack mixes and redistributes powder on the sieves. Applicable over the ASTM E-11 sieve range.

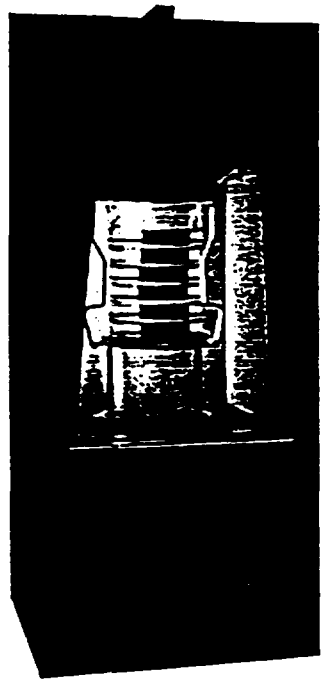
PERFORMING THE SIEVE ANALYSIS (cont.)

To perform the actual sieve analysis, screens should be chosen in a sequence as described earlier. Use either every sieve, every other sieve, every third sieve, etc. between the desired size parameters. The use of screens in this sequential order will allow for better data presentation and a more meaningful analysis of the test results. Care should also be taken in selecting the proper sieves to avoid overloading any sieve with an especially large material peak. For example, a specification may require 96% of the sample be retained above a #50 mesh sieve. The proper way to perform an analysis of this nature is to use 'relief screens', that is, sieves in the 30, 35, 40 and 45 mesh ranges to remove some of the burden from the critical cut point of 50 mesh. If the relief screens are not used, the particles of exactly 50 mesh size or slightly larger will be driven through by the mass of material resting above them. The screen cut point would be inaccurate and sample would not meet the specifications for the test.

The selected sieves should be assembled with the coarsest sieve at the top of the stack, and the balance of the stack in increasing magnitude of fineness (increasing sieve numbers). The stack should be finished off with a cover on the top sieve and a pan below the finest sieve. At this point, the sieve stack is either shaken and rapped by hand, or mounted in a sieve shaker with a motorized or electrostatic drive mechanism.

While many applications still use the hand-shaken method for sieving, motor driven shakers have proven to be much more consistent, not having to account for differences in operators. In the case of powder analysis below the 100 mesh range, the sieve shaker should be equipped with a device to impart a shock wave to the sieve stack at regular intervals. This hammer or rapping device is necessary to reorient the particles on the screen and impart some shear forces to near-size particles blocking the sieve openings.

The duration of the sieving interval is usually regulated by industry standards, or by in-house control specifications. Commonly, 10, 15 or 20 minute tests are used as arbitrary sieving intervals. To determine the best interval for a new material, or to double check the accuracy of existing specifications, the following procedure can be used. Select the desired sieves for the analysis. Weigh up a sample of the material to be tested and introduce it to the completed sieve stack. Shake the sieve stack for a period of 5 minutes. Weigh the residue in the pan and calculate the percentage in relation to the starting weight. Reassemble the stack and shake for one additional minute. Repeat the weigh-up procedure and calculate the percentage. If the percentage of fines increased more than 1% between 5 minutes and 6 minutes, reassemble the stack and shake for an additional minute. The data can be plotted as percentage throughput vs. time for each data point you calculate. When the change in the percentage of fines passing in the 1 minute period drops below 1%, the test can be considered complete. Record the total testing time for subsequent analysis.



SONIC SIFTER

A viable alternative for fine powder sieving using a unique oscillating air column. The effective size range is 5660 µm (3.5 mesh) through 5 µm using 3" diameter acrylic frame sieves. This method has the advantages of high accuracy with a low test time, while showing no signs of particle attrition or screen wear.

Another type of sieve analysis is the wet sieve test. In this method, the sample is weighed and then washed through the finest sieve in the stack with water, water with a wetting agent or some other compatible solvent. After thoroughly washing the fines from the raw sample, the residue is dried either over a hot plate or in an oven. Care must be taken to keep the temperature of the sieve below 300°F (149°C) to avoid loosening of the sieve cloth or failure of the solder joint. After drying, the residue is then sieved normally on the balance of the sieve stack. The loss in weight not accounted for on the coarse screens is assumed to be fines.

Wet sieve analysis is especially helpful when working with naturally agglomerated materials, ultrafine powders with severe static changes, and in samples where fine particles tend to cling to the coarse fractions in the blend. The disadvantages associated with wet sieving are primarily the time period required to perform the analysis due to drying time and the possible damage to the sieve mesh by overloading. A common practice with wet sieving operations is brushing or forcing the

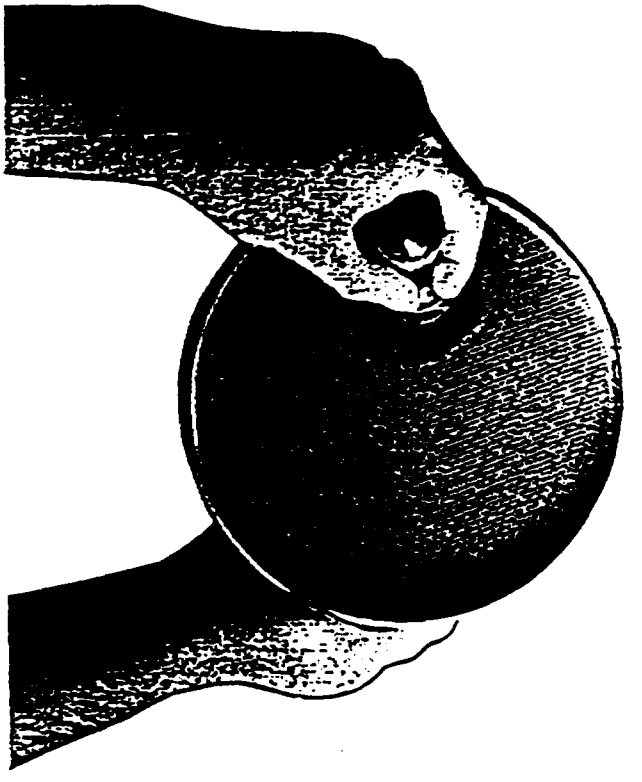
PERFORMING THE SIEVE ANALYSIS (cont.)

sample through the mesh while the liquid medium is directed on the sieve. This pressure can distort the sieve openings or tear the mesh at the solder joint through stress.

Once the sieving interval is complete, whether dry or wet sieving is used, the residue on each sieve is removed by pouring the residue into a suitable weighing vessel. To remove material wedged in the sieving openings, the sieve is inverted over a sheet of paper or suitable collector and the underside of the wire cloth brushed **gently** with a nylon paint brush with bristles cut to a 1" length. The side of the sieve frame may be tapped gently with the handle of the brush to dislodge the particles between brush strokes. At no time should a needle or other sharp object be used to remove the particles lodged in the wire cloth. Special care should be taken when brushing sieves finer than 80 mesh. Brushing can cause distortions and irregularities in the mesh. The procedure is repeated for each sieve in the stack and contents of the pan.

The individual weights retained on the sieves should be added and compared to the starting sample weight. Wide variations or sample losses should be determined immediately. Compare the weights on each sieve with the total sample weight and calculate the percentage of the total retained at that point.

Presentation and analysis of the resulting data is frequently made easier by plotting on one of a number of graph formats. The most common graphic presentation is the plot of the cumulative percentage of material retained on a screen (plotted on a logarithmic scale) versus percentage (plotted on a linear scale). The resulting curve allows a quick approximation of the sieve size at the fifty percentile point of accumulation. The curve also shows the smoothness of the distribution by revealing the presence of bimodal blends in the sample. Other plotting techniques include log-log and direct plotting of micron size versus percentage retained.



COMMONLY USED BRUSHING TECHNIQUE

Care should be exercised in the analysis of the data in relation to the length of time the test was run. If your sample contains a large amount of elongated or near-size particles, the test results can be misleading. The longer the sieving interval, the greater the percentage of these problem particles passing through the screens. Ideally each fraction should be inspected microscopically after sieving to determine the integrity of the sieve cut point.

Table 6 lists the ASTM published standards on sieve analysis procedures for specific materials or industries.

SIEVE CARE AND CLEANING

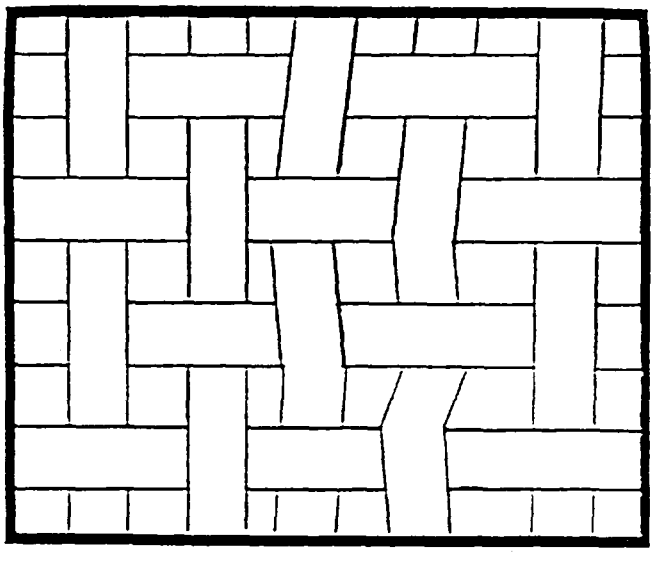
Test sieves, like any other piece of analytical laboratory equipment, require regular care to maintain their performance standards. The sieves should be kept clean and dry at all times, and stored either in the cardboard carton provided or in a suitable cabinet. In order for the test sieve to perform properly, the wire cloth must be taut and free from variations in opening size. For this reason, cleaning procedures must be clearly delineated as part of a comprehensive sieving program.

Test sieves should be cleaned ultrasonically on a regular basis. For some installations, this may be done at the end of a shift or at the end of a week, but must be done regularly to assure good sieving results. The sieves should be immersed in an ultrasonic cleaner filled with a solution of a mild detergent and water. The sieves should be cleaned for a suitable time interval (follow manufacturer's instructions). Before using, allow the test sieves to dry thoroughly. Regular ultrasonic cleaning will prevent the buildup of particles trapped in the mesh and prolong the useful life of the sieve. For between test clean-up, brushing of the mesh, sizes 100 and coarser, is allowable. For best results, use a nylon bristle paint brush with the bristles cut to a length of approximately 1". The mesh should be brushed from the underside only with a gentle circular motion. Vigorous brushing will distort the sieve openings and reduce the effective life of the sieve. Under no circumstances should particles lodged in the sieve openings be removed with a sharp object. These particles should be removed in an ultrasonic cleaner only. Brushing should be avoided on sieves finer than 100 mesh, as the fine wires are more likely to bend, distort or even break. Brushing has a tendency to loosen any wire cloth, but the fine ranges are even more susceptible to this damage.

Similarly, cleaning sieves with a compressed air jet is common, but can be disastrous to the 100 mesh and finer range. The concentrated jet of air can do severe 'local' damage to the wire cloth, and significantly loosen the wire cloth.

Generally, sieves can have an effective life of many years with proper care. The wear that occurs over time does not change the opening sizes, but does abrade the 'knuckles' or crimps of the mesh. A sieve with a noticeable sag to the mesh should be replaced. Fine mesh sieves that have torn should not be resoldered, as the localized heat of the soldering iron can distort the openings. Epoxies have been used for repairs, but usually tend to block a large percentage of the openings. The typical epoxies also become too brittle for the flexing of the wire cloth and can fracture with use.

Good general laboratory procedure should be observed with testing sieves as with any other piece of test equipment. Testing should be performed with clean, uncontaminated sieves, especially when using a sieve for the first time. With proper care and cleaning coupled with a good calibration procedure, any test sieve should provide many years of consistent service.



WIRE CLOTH DAMAGED BY IMPROPER BRUSHING

Note the irregularities in both opening size and shape.

EPILOG

We hope that the characterization of testing sieves and their uses presented in this manual will serve as an enhancement to your current particle size analysis program. By maximizing the analytical advantage potential of testing sieves while minimizing and compensating for shortcomings and inaccuracies, the testing sieve can be a viable and precise testing tool. Care, maintenance and proper test procedure are as critical with a testing sieve as they are with other, more sophisticated particle size analyzers.

Compliance with applicable industry, National and International specifications is essential. The intent of these regulating bodies is the formulation of general standards to assure uniformity in testing standards observed by both buyer and producer. The accepted specification should be the foundation for the in-house testing procedure.

Testing accuracy is highly dependent on the technique of the operators. Interpretation of data should be neither overstated nor understated in terms of importance. The effects of variables must be understood, accepted and factored into final data analysis to avoid these shortcomings.

By analyzing the total test sieve analysis program as we suggest from sample preparation to data presentation, variations can be reduced, accuracy improved, and productivity increased.

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U. S. A. STANDARD SIEVES ASTM SPECIFICATION E-11

Nominal Dimensions, Permissible Variations for Wire Cloth of Standard Test Sieves (U.S.A.) Standard Series

Sieve Designation (W)		Nominal Sieve Opening, in.[c]	Permissible Variation of Average Opening from the Standard Sieve Designation (y)	Intermediate Tolerance (z)[d]	Maximum Individual Opening (x)	Nominal Wire Diameter, mm[a]
Standard[b]	Alternative					
(1)	(2)	(3)	(4)	(5)	(6)	(7)
125 mm	5 in.	5	±3.7 mm	130.0 mm	130.9 mm	8.00
106 mm	4.24 in.	4.24	±3.2 mm	110.2 mm	111.1 mm	6.40
100 mm[d]	4 in.[d]	4	±3.0 mm	104.0 mm	104.8 mm	6.30
90 mm	3½ in.	3.5	±2.7 mm	93.6 mm	94.4 mm	6.08
75 mm	3 in.	3	±2.2 mm	78.1 mm	78.7 mm	5.80
63 mm	2½ in.	2.5	±1.9 mm	65.6 mm	66.2 mm	5.50
53 mm	2.12 in.	2.12	±1.6 mm	55.2 mm	55.7 mm	5.15
50 mm[d]	2 in.[d]	2	±1.5 mm	52.1 mm	52.6 mm	5.05
45 mm	1¾ in.	1.75	±1.4 mm	46.9 mm	47.4 mm	4.85
37.5 mm	1½ in.	1.5	±1.1 mm	39.1 mm	39.5 mm	4.59
31.5 mm	1¼ in.	1.25	±1.0 mm	32.9 mm	33.2 mm	4.23
26.5 mm	1.06 in.	1.06	±0.8 mm	27.7 mm	28.0 mm	3.90
25.0 mm[d]	1 in.[d]	1	±0.8 mm	26.1 mm	26.4 mm	3.80
22.4 mm	⅞ in.	0.875	±0.7 mm	23.4 mm	23.7 mm	3.50
19.0 mm	¾ in.	0.750	±0.6 mm	19.9 mm	20.1 mm	3.30
16.0 mm	⅝ in.	0.625	±0.5 mm	16.7 mm	17.0 mm	3.00
13.2 mm	0.530 in.	0.530	±0.41 mm	13.83 mm	14.05 mm	2.75
12.5 mm[d]	½ in.[d]	0.500	±0.39 mm	13.10 mm	13.31 mm	2.67
11.2 mm	7/16 in.	0.438	±0.35 mm	11.75 mm	11.94 mm	2.45
9.5 mm	⅜ in.	0.375	±0.30 mm	9.97 mm	10.16 mm	2.27
8.0 mm	5/16 in.	0.312	±0.25 mm	8.41 mm	8.58 mm	2.07
6.7 mm	0.265 in.	0.265	±0.21 mm	7.05 mm	7.20 mm	1.87
6.3 mm[d]	¼ in.[d]	0.250	±0.20 mm	6.64 mm	6.78 mm	1.82
5.6 mm	No. 3½[e]	0.223	±0.18 mm	5.90 mm	6.04 mm	1.68
4.75 mm	No. 4	0.187	±0.15 mm	5.02 mm	5.14 mm	1.54
4.00 mm	No. 5	0.157	±0.13 mm	4.23 mm	4.35 mm	1.37
3.35 mm	No. 6	0.132	±0.11 mm	3.55 mm	3.66 mm	1.23
2.80 mm	No. 7	0.11	±0.095 mm	2.975 mm	3.070 mm	1.10
2.36 mm	No. 8	0.0937	±0.080 mm	2.515 mm	2.600 mm	1.00
2.00 mm	No. 10	0.0787	±0.070 mm	2.135 mm	2.215 mm	0.900
1.70 mm	No. 12	0.0661	±0.060 mm	1.820 mm	1.890 mm	0.810
1.40 mm	No. 14	0.0555	±0.050 mm	1.505 mm	1.565 mm	0.725
1.18 mm	No. 16	0.0469	±0.045 mm	1.270 mm	1.330 mm	0.650
1.00 mm	No. 18	0.0394	±0.040 mm	1.080 mm	1.135 mm	0.580
850 μm[f]	No. 20	0.0331	±35 μm	925 μm	970 μm	0.510
710 μm	No. 25	0.0278	±30 μm	775 μm	815 μm	0.450
600 μm	No. 30	0.0234	±25 μm	660 μm	695 μm	0.390
500 μm	No. 35	0.0197	±20 μm	550 μm	585 μm	0.340
425 μm	No. 40	0.0165	±19 μm	471 μm	502 μm	0.290
355 μm	No. 45	0.0139	±16 μm	396 μm	425 μm	0.247
300 μm	No. 50	0.0117	±14 μm	337 μm	363 μm	0.215
250 μm	No. 60	0.0098	±12 μm	283 μm	306 μm	0.180
212 μm	No. 70	0.0083	±10 μm	242 μm	263 μm	0.152
180 μm	No. 80	0.0070	±9 μm	207 μm	227 μm	0.131
150 μm	No. 100	0.0059	±8 μm	174 μm	192 μm	0.110
125 μm	No. 120	0.0049	±7 μm	147 μm	163 μm	0.091
106 μm	No. 140	0.0041	±6 μm	126 μm	141 μm	0.076
90 μm	No. 170	0.0035	±5 μm	108 μm	122 μm	0.064
75 μm	No. 200	0.0029	±5 μm	91 μm	103 μm	0.053
63 μm	No. 230	0.0025	±4 μm	77 μm	89 μm	0.044
53 μm	No. 270	0.0021	±4 μm	66 μm	76 μm	0.037
45 μm	No. 325	0.0017	±3 μm	57 μm	66 μm	0.030
38 μm	No. 400	0.0015	±3 μm	48 μm	57 μm	0.025
32 μm	No. 450	0.0012	±3 μm	42 μm	50 μm	0.025
25 μm	No. 500	0.0010	±3 μm	34 μm	41 μm	0.025
20 μm	No. 635	0.0008	±3 μm	29 μm	35 μm	0.020

[a] The average diameter of the warp and of the shoot wires, taken separately, of the cloth of any sieve shall not deviate from the nominal values by more than the following:

Sieves coarser than 600 μm	5 percent
Sieves 600 to 125 μm	7½ percent
Sieves finer than 125 μm	10 percent

[b] These standard designations correspond to the values for test sieve apertures recommended by the International Standards Organization, Geneva, Switzerland.

[c] Only approximately equivalent to the metric values in Column 1.

[d] These sieves are not in the standard series but they have been included because they are in common usage.

[e] These numbers (3½ to 635) are the approximate number of openings per linear inch but it is preferred that the sieve be identified by the standard designation in millimeters or micrometers.

[f] 1000 μm = 1 mm.

[g] Not more than 5% of the openings may fall between the limits set by the values in Column 5 and Column 6.

Table 1

INTERNATIONAL STANDARDS ORGANIZATION
PREFERRED NUMBER SERIES

R 10	Basic sizes millimeters R 20	R 40	Equivalent sizes (for information) inches
1.00	1.00	*1.00	0.039 4
		1.06	0.041 7
	1.12	1.12	0.044 1
		*1.18	0.046 5
1.25	1.25	1.25	0.049 2
		1.32	0.052 0
	1.40	*1.40	0.055 2
		1.50	0.059 1
1.60	1.60	1.60	0.063 0
		*1.70	0.066 9
	1.80	1.80	0.070 9
		1.90	0.074 8
2.00	2.00	*2.00	0.078 7
		2.12	0.083 5
	2.24	2.24	0.088 2
		*2.36	0.092 9
2.50	2.50	2.50	0.098 4
		2.65	0.104 3
	2.80	*2.80	0.110 2
		3.00	0.118 1
3.15	3.15	3.15	0.124 0
		*3.35	0.131 9
	3.55	3.55	0.139 8
		3.75	0.147 6
4.00	4.00	*4.00	0.157 5
		4.25	0.167 3
	4.50	4.50	0.177 2
		4.75	0.187 0
5.00	5.00	5.00	0.196 9
		5.30	0.208 7
	5.60	*5.60	0.220 5
		6.00	0.236 2
6.30	6.30	6.30	0.248 0
		*6.70	0.263 8
	7.10	7.10	0.279 5
8.00	8.00	*8.00	0.315 0
		8.50	0.334 6
	9.00	9.00	0.354 3
		*9.50	0.374 0
10.00	10.00	10.00	0.393 7
		10.6	0.417 3
	11.2	*11.2	0.440 9
			0.464 6
		11.8	
12.5	12.5	12.5	0.492 1
		*13.2	0.519 7
	14.0	14.0	0.551 8
		15.0	0.590 6
16.0	16.0	*16.0	0.629 9
		17.0	0.669 3
	18.0	18.0	0.708 7
		*19.0	0.748 0
20.0	20.0	20.0	0.787 4
		21.2	0.834 6
	22.4	*22.4	0.881 9
		23.6	0.929 1
25.0	25.0	25.0	0.984 3

Table 2

* — R-40/3 Series - Every third number of R-40 Series
Same as ASTM E-11 USA Standard Sieve Series

R-10 — Tenth Root of ten ratio

R-20 — Twentieth root of ten

R-40 — Fortieth root of ten

COMPARISON TABLE
INTERNATIONAL TEST SIEVE SERIES

INTERNATIONAL ISO 565 (TBL2): 1983	AMERICAN ANSI/ASTM E-11-81		BRITISH BS 410: 1976		CANADA B-GP-2M: 1976	FRENCH AFNOR NEX 11-501: 1970	GERMAN DIN 4188: 1977
Aperture mm	Opening mm	Equivalent inch/Number	Aperture mm	Equivalent BS Mesh	Aperture mm	Aperture mm	Aperture mm
	106.00	4.24"	106.00				
100.00	100.00	4.0"	100.00		100.00	100.00	100.00
90.00	90.00	3 1/2"	90.00		90.00	90.00	90.00
71.00	75.00	3"	75.00		71.00	71.00	71.00
63.00	63.00	2 1/2"	63.00		63.00	63.00	63.00
	53.00	2.12"	53.00		53.00	53.00	53.00
50.00	50.00	2.0"	50.00		50.00	50.00	50.00
45.00	45.00	1 3/4"	45.00		45.00	45.00	45.00
	37.50	1 1/2"	37.50				
31.50	31.50	1 1/4"	31.50		31.50	31.50	31.50
26.50	26.50	1.06"	26.50		28.00*	28.00*	28.00*
–	25.00	1.00"	25.00		25.00	25.00	25.00
22.40	22.40	7/8"	22.40		22.40	22.40	22.40
19.00	19.00	3/4"	19.00		20.00*	20.00*	20.00*
16.00	16.00	5/8"	16.00		16.00	16.00	16.00
13.20	13.20	.530"	13.20		14.00*	14.00*	14.00*
–	12.50	1/2"	12.50		12.50	12.50	12.50
11.20	11.20	7/16"	11.20		11.20	11.20	11.20
9.50	9.50	3/8"	9.50		10.00*	10.00*	10.00*
8.00	8.00	5/16"	8.00		8.00	8.00	8.00
6.70	6.70	.265"	6.70		7.10*	7.10*	7.10*
–	6.30	1/4"	6.30		6.30	6.30	6.30
5.60	5.60	No. 3 1/2	5.60	3	5.60	5.60	5.60
4.75	4.75	4	4.75	3 1/2	–	–	–
4.00	4.00	5	4.00	4	4.00	4.00	4.00
3.35	3.35	6	3.35	5	3.15*	3.15*	3.15*
2.80	2.80	7	2.80	6	2.80	2.80	2.80
2.36	2.36	8	2.36	7	2.50*	2.50*	2.50*
2.00	2.00	10	2.00	8	2.00	2.00	2.00
1.70	1.70	12	1.70	10	1.60*	1.60*	1.60*
1.40	1.40	14	1.40	12	1.40	1.40	1.40
1.18	1.18	16	1.18	14	1.12*	1.12*	1.12*
1.00	1.00	18	1.00	16	1.00	1.00	1.00
850µm	850µm	20	850µm	18	800*µm	800*µm	800*µm
710µm	710µm	25	710µm	22	710µm	710µm	710µm
600µm	600µm	30	600µm	25	–	–	–
500µm	500µm	35	500µm	30	500µm	500µm	500µm
425µm	425µm	40	425µm	36	400*µm	400*µm	400*µm
355µm	355µm	45	355µm	44	355µm	355µm	355µm
300µm	300µm	50	300µm	52	315*µm	315*µm	315*µm
250µm	250µm	60	250µm	60	250µm	250µm	250µm
212µm	212µm	70	212µm	72	200*µm	200*µm	200*µm
180µm	180µm	80	180µm	85	180µm	180µm	180µm
150µm	150µm	100	150µm	100	140*µm	140*µm	140*µm
125µm	125µm	120	125µm	120	125µm	125µm	125µm
106µm	106µm	140	106µm	150	100*µm	100*µm	100*µm
90µm	90µm	170	90µm	170	90µm	90µm	90µm
75µm	75µm	200	75µm	200	71*µm	71*µm	71*µm
63µm	63µm	230	63µm	240	63µm	63µm	63µm
53µm	53µm	270	53µm	300	56*µm	56*µm	56*µm
45µm	45µm	325	45µm	350	45µm	45µm	45µm
38µm	38µm	400	38µm	400	36*µm	36*µm	36*µm

Closest equivalent to ISO 565 (TBL 2)

Table 3

RECOMMENDED REPRESENTATIVE BULK VOLUMES OF TEST SAMPLES

Used In 8" (203mm) Testing Sieves

Standard Sieve Designation		Bulk Volume of Material	
Standard	Alternate	Recommended Volume of Material for Test Sample	Maximum Permitted Volume on Sieve on Completion of Sieving
1	2	3	4
25.0mm	1 in.	1800cm³	900cm³
22.4	7/8	1600	800
19.0	3/4	1400	700
16.0	5/8	1000	500
12.5	1/2	800	400
11.2	7/16	800	400
9.5	3/8	600	300
8.0	5/16	500	250
6.30	1/4	400	200
5.60	No. 3 1/2	400	200
4.0	No. 5	350	150
2.80	No. 7	240	120
2.0	No. 10	200	100
1.40	No. 14	160	80
1.0	No. 18	140	70
710μm	No. 25	120	60
500	No. 35	100	50
355	No. 45	80	40
250	No. 60	70	35
180	No. 80	60	30
125	No. 120	50	25
90	No. 170	40	20
63	No. 230	35	17
45	No. 325	30	15
38	No. 400	25	12

The recommended weight of material for a sieve test sample is calculated by multiplying the bulk volume figure in Column 3 by the particular bulk density in grams per cubic centimeter of the material, rounded out within a tolerance of ±25 percent.

Table 4

BULK DENSITY OF PULVERIZED MATERIALS IN FREELY POURED CONDITION[a]

Material	Avg. Wt. lbs/ft.³	Avg. Wt. g/cm³	Material	Avg. Wt. lbs/ft.³	Avg. Wt. g/cm³	Material	Avg. Wt. lbs/ft.³	Avg. Wt. g/cm³
Alumina	44	1.23	Garnet	168	2.69	Salt, rock	66	1.06
Aluminum,			Glass beads	76	1.22	Salt, table	75	1.20
calcined	128	2.05	Glass, crushed	66	1.06	Sand	90 to 100	1.44 to 1.60
Aluminum oxide	122	1.96	Glass cullet	93	1.49	Sand, silica	90 to 100	1.44 to 1.60
Aluminum shot	96	1.54	Granite,			Sawdust	13	0.22
Ammonium nitrate	48	0.77	crushed	95 to 100	1.52 to 1.60	Seacoal	42	0.67
Ammonium - sulfate			Gravel	90 to 100	1.44 to 1.60	Shale	100	1.60
	61	0.98	Gypsum, calcined	58	0.93	Shot, metal	200	3.69
Asbestos ore	54	0.87	Gypsum,			Silica Flour	27	0.43
Bauxite ore	75 to 85	1.20 to 1.36	crushed	90 to 100	1.44 to 1.60	Silica gel	45	0.72
Bentonite	50 to 65	0.80 to 1.04	Iron ore	120 to 150	1.92 to 2.40	Soapstone,		
Bicarbonate of			Kaolin	160	2.56	pulverized	40	0.64
soda	57	0.91	Kyanite	68	1.09	Soda ash,		
Borax	50 to 61	0.80 to 0.98	Lime, ground	60 60	0.96	light	25 to 35	0.40 to 0.56
Boric acid	58	0.93	Lime, hydrated	25	0.40	Soda ash,		
Calcite	90 to 105	1.44 to 1.68	Limestone,			heavy	55 to 65	0.88 to 1.04
Calcium carbide	75	1.20	crushed	85 to 100	1.36 to 1.60	Soda, bicarbonate	57	0.91
Calcium			Limestone,			Sodium nitrate	78	1.25
carbonate	49	0.79	agricultural	70	1.12	Sodium phosphate	43	0.69
Calcium chloride	64	1.03	Magnesite	106	1.70	Sodium sulfate	96	1.54
Calcium			Magnetite	155	2.49	Steel grit	228	3.66
phosphate	57	0.91	Manganese ore	120 to 136	1.92 to 2.18	Stone, crushed	85 to 95	1.36 to 1.52
Carbon black	24	0.33	Marble, crushed	90 to 95	1.44 to 1.52	Sugar, granulated	50	0.80
Cellulose powder	16	0.26	Metals, powdered					
Cement,			Aluminum	80	1.28	Sugar, powdered	37	0.59
portland	90 to 100	1.44 to 1.60	Copper	169	2.71	Sulphur, crushed	50 to 65	0.80 to 1.04
Cement clinker	75 to 80	1.20 to 1.28	Copper-lead	364	5.84	Talc, powder	34	0.55
Chrome ore	140	2.25	Iron	243	3.90	Talc, granular	44	0.71
Clay	30 to 75	0.48 to 1.20	Nickel	253	4.22	Traprock, crushed	105 to 110	1.68 to 1.76
Coal, anthracite	55	0.88	Stainless steel	240	3.85			
Coal, bituminous	50	0.88	Tantalum	300	4.80	Triple superphos-		
Coke breeze	25 to 35	0.40	Mica	42	0.67	phate, granular	64	1.03
Coke,			Ore, sintered	144	1.82	Tungsten carbide	530	8.52
petroleum	25 to 40	0.40 to 0.64	Oyster shells,			Urea prills	43	0.69
Copper ore	100 to 150	1.60 to 2.40	ground	29	0.47	Vermiculite ore	80	1.28
Coquina shell	80	1.28	Perlite ore	63 to 75	1.04 to 1.20	Wood chips	10	0.16
Corn starch	40	0.64	Plaster, calcined	64	1.03	Zinc dust	344	5.51
Diatomaceous			Polyethylene			Zirconium shale	271	4.34
earth	31	0.50	pellets	36	0.58	Zirconium sand	161	2.61
Dicalcium			Polyethylene					
phosphate	64	1.03	powder	18	0.29			
Dolomite,			Polyvinyl					
crushed	90 to 100	1.44 to 1.60	chloride	30	0.48			
Feldspar, crushed	65 to 84	1.04 to 1.35	Potash	77	1.23			
Ferrophosphorus	196	3.14	Potassium					
Fire clay	80	1.28	carbonate	79	1.27			
Flour, wheat	24	0.38	Pumice	40	0.64			
Flour, maize	37	0.59	Rubber, chopped	36	0.58			
Fluorspar	90 to 100	1.44 to 1.92	Rubber, ground	32	0.52			
Fly ash	49	0.79	Phosphate rock	75 to 85	1.20 to 1.36			
Fullers earth	30 to 40	0.48 to 0.64	Salt, flake	61	0.98			

a — Where a single figure is given, it represents an actual weight of a typical sample sample as it is measured, recorded by a research laboratory; therefore, the figure can be expected to vary from sample to sample of the same material.

Table 5

LIST OF ASTM PUBLISHED STANDARDS ON SIEVE ANALYSIS PROCEDURES FOR SPECIFIC MATERIALS OR INDUSTRIES

Material	ASTM Designation	Title of Standard	Sieve No. or Size Range	Dry	Wet
[illegible]	C 117	Test for Materials Finer Than No. 200 Sieve in Mineral Aggregates by Washing [a]	200		X
	C 125	Definitions of Terms Relating to Concrete and Concrete Aggregates			
	C 136	Test for Sieve or Screen Analysis of Fine and Coarse Aggregates [a]	3½ in.-200	X	
	C 142	Test for Friable Particles in Aggregates	1½ in.-20	X	
	C 330	Specifications for Lightweight Aggregates for Structural Concrete	1 in.-100	X	X
	C 331	Specifications for Lightweight Aggregates for Concrete Masonry Units	3/4 in.-100	X	X
Asbestos	D 2589	Test for Bauer-McNett Wet Classification of Asbestos Fiber	4-325		X
Asphalt	D 313	Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter			X
[illegible] black	D 1508	Test for Fines Content of Pelleted Carbon Black	100	X	
	D 1511	Test for Pellet Size Distribution of Carbon Black	10-120	X	
	D 1514	Test for Sieve Residue from Carbon Black	30-325		X
Casein	D 2592	Test for Sieve Analysis of Dasein and Isolated Soy Protein	4-200	X	
Ceramic [b]	C 325	Method for Wet Sieve Analysis of Ceramic Whiteware Clays	100-325		X
	C 371	Method for Sieve Analysis of Nonplastic Pulverized Ceramic Materials [a]	70-325		X
Coal	D 197	Sampling and Fineness Test of Powdered Coal [a]	16-200	X	
	D 310	Test for Size of Anthracite [a]	4 3/8 in.-3/16 in.	X	
	D 311	Test for Sieve Analysis of Crushed Bituminous Coal [a]	1 in.-6	X	
	D 410	Test for Screen Analysis of Coal [a]	8 in.-200	X	
	D 431	Method of Designating the Size of Coal from its Analysis	8 in.-200	X	
Coke	D 293	Test for Sieve Analysis of Coke [a]	4 in.-0	X	
Cement	C 184	Test for Fineness of Hydraulic Cement by the No. 100 and 200 Sieves	100 200	X	
	C 430	Test for Fineness of Hydraulic Cement by the No. 325 Sieve	325		X
Enamel	C 285	Method for Sieve Analysis of Wet Milled and Dry Milled Porcelain Enamel	40-325	X	X
Glass	C 429	Method for Sieve Analysis of Raw Materials for Glass Manufacture [a]	8-200	X	X
	D 1214	Test for Sieve Analysis of Glass Spheres			
Magnesia	C 239	Test for Sieve Analysis of Plastic Calcined Magnesia	4-200		X
Magnesium compositions	C 238	Method for Sieve Analysis of Magnesium Oxychloride Compositions, Aggregates and Fillers		X	
Metal Bearing ores	E 276	Test for Particle Size or Screen Analysis at No. 4 Sieve and Finer for Metal Bearing Ores and Related Materials [a]	4-200	X	X
Metal powders	B 214	Test for Sieve Analysis of Granular Metal Powders	60-325	X	
Mineral	D 451	Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles [a]	6-100	X	
	D 452	Test for Sieve Analysis of Nongranular Mineral Surfacing for Asphalt Roofing and Shingles [a]	12-200	X	
	D 546	Test for Sieve Analysis of Mineral Filler		X	
Perlite	C 549	Specification for Perlite Loose Fill Insulation [a]		X	
Pigments and paint	D 185	Tests for Coarse Particles in Pigments, Pastes and Paints	325	X	X
	D 480	Methods of Sampling and Testing Aluminum Powder and Paste [a]	100-325		X
Plastic	D 1921	Test for Particle Size (Sieve Analysis) of Plastic Materials		X	
Refractories	C 92	Tests for Sieve Analysis and Water Content of Refractory Materials	3-200	X	X
Resins	D 1457	Specification for TFE-Fluorocarbon Resin Molding and Extrusion Materials	18-325		X
	D 1705	Method for Particle Size Analysis of Powdered Polymers and Copolymers of Vinyl Chloride			X
	D 2187	Tests for Physical and Chemical Properties of Ion-Exchange Resins [a]	8-100		X
Soap	D 502	Test for Particle Size of Soaps and Other Detergents	12-100	X	
Soil	D 421	Method for Dry Preparation of Soil Samples for Grain-Size Analysis and Determination of Soil Constants	4-40		X
	D 422	Method for Grain-Size Analysis of Soils	3 in.-200		X
	D 1140	Test for Amount of Material in Soils Finer Than the No. 200 Sieve	40-200		X
	D 2217	Method for Wet Preparation of Soil Samples for Grain-Size Analysis and Determination of Soil Constants	10-40		X
Vermiculite	C 516	Specifications for Vermiculite Loose Fill Insulation [a]	3/4-100	X	

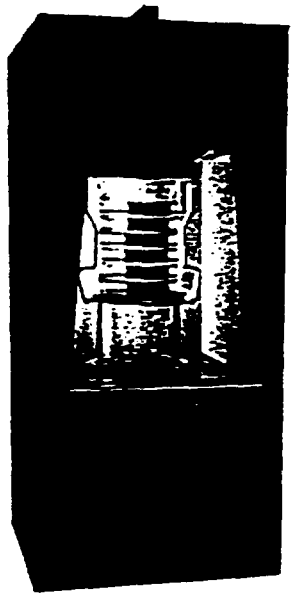
a — *Contains suggestions on sampling*

b — *Also, C 623, Method for Precision Electroformed Wet Sieve Analysis of Nonplastic Ceramic Powders.*

Table 6

Other products available from the publisher of this manual:

THE ATM SONIC SIFTERS



The Model L3P Laboratory Sonic Sifter

A viable alternative for fine powder sieving using a unique oscillating air column. The effective size range is 5660 μm (3.5 mesh) through 5 μm using 3″ diameter acrylic frame sieve. This method has the advantages of high accuracy with low test times, while showing no signs of particle attrition or screen wear.

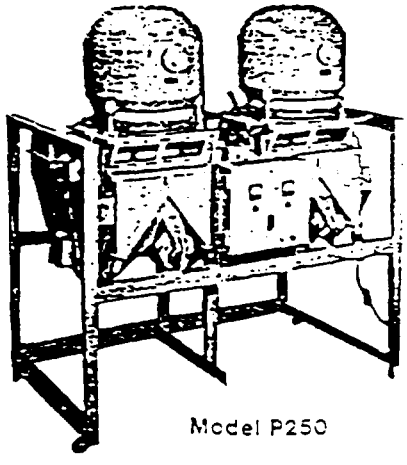
Model P60

The Model P60 Production Sonic Sifter

60 square inches of screening area are available in this single screen, single pass continuous feed sonic sifter. The effective screening range is 5660 μm (3.5 mesh) through 20 μm (635 mesh). Ideal for pilot plant and small production application, this modular sifter is capable of input rates of up to several hundred pounds per hour.

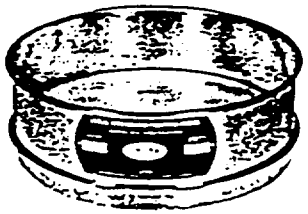
The Model P250 Production Sonic Sifter

Two 125 square inch screening modules provide input rates of up to 1000 pounds per hour in this high accuracy, single pass sifter. An upscaled version of the model P60.



Model P250

This manual was edited by Scott Nushart and Walter Mahlig.



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WARZYN

SIEVE ANALYSIS ASTM C14[illegible]-80
COMPUTER CALCULATION SHEET

JOB:

Job No. _____
Date _____
Sheet _____ of _____
Techs _____
Checked By _____

BORING/SAMPLE									
PAN NO.									
START WT. or HYD (No Tare)									
END WT./REMARKS (No Tare)									
1 1/2									
1									
3/4									
1/2									
3/8									
#4									
#8									
#10									
#16									
#30									
#40									
#50									
#80									
#100									
#200									

note: all technicians performing work are required to initial sheet.

WARZYN

SIEVE ANALYSIS

JOB:

SAMPLE NO.

PAN NO.

Report No. _____

Job No. _____

Date _____

Sheet _____ of _____

Start Sample Weight =

End Sample Weight = _____

Weight of P200 Loss =

P200 Loss Weight / Start Sample Weight = % P200 Loss

End Sample Weight =

R #4 Sieve Weight = _____

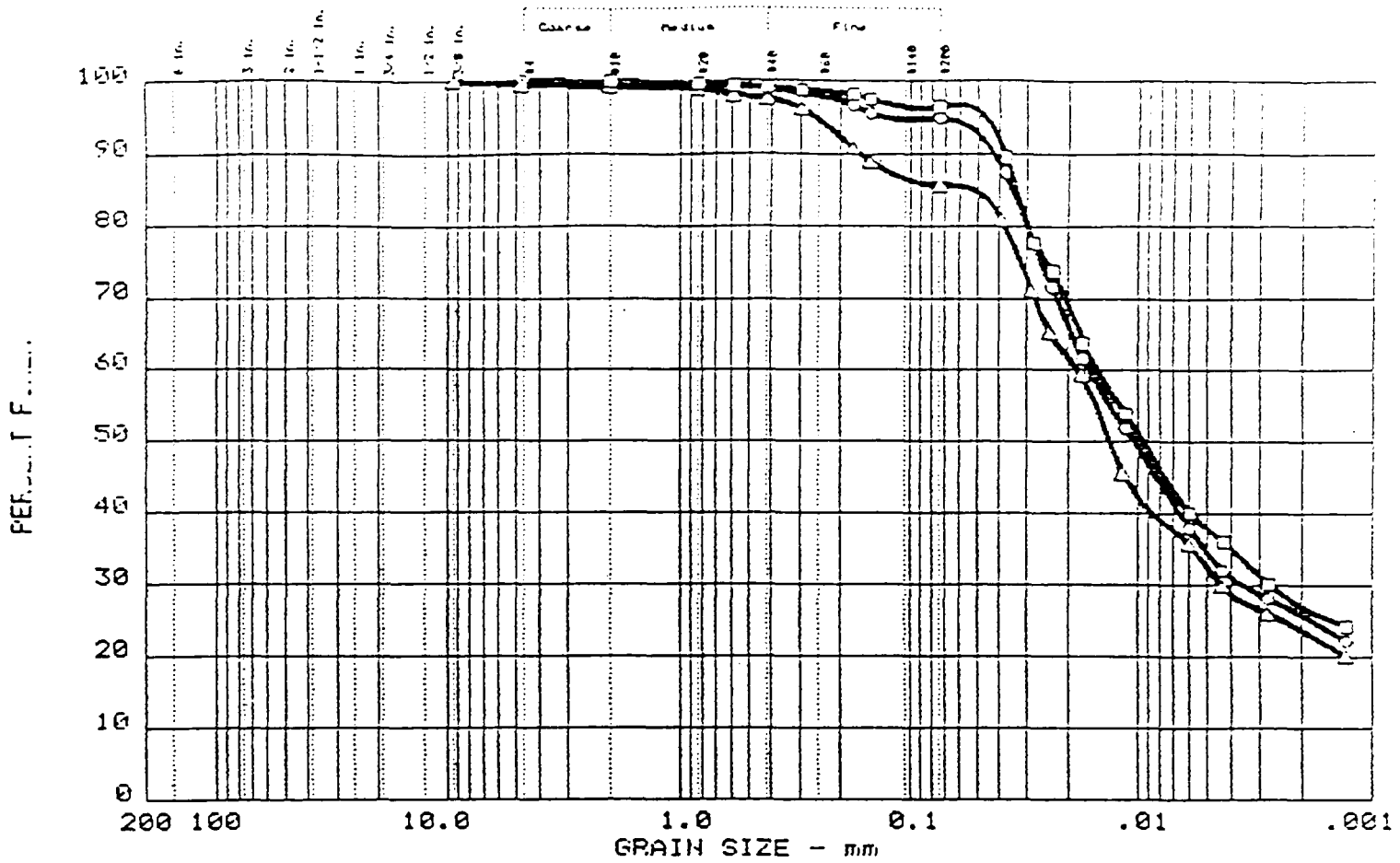
Difference in Weight =

Weight Difference / Start Sample Weight = Mult

Sieve Size	Comm. Wt. Retained	Percent Passing	Multiplier	% P200 Loss in Washing	Percent Passing	_____ Specifications
2 1/2"		X	X	X		
2"		X	X	X		
1 1/2"		X	X	X		
1"		X	X	X		
3/4"		X	X	X		
1/2"		X	X	X		
3/8"		X	X	X		
#4		X	X	X		
Quartered P#4 Wt =						
#4						
#8						
#10						
#16						
#30						
#40						
#50						
#80						
#100						
#200						

DA/mlf
[WCI-15-35]

GRAIN SIZE DISTRIBUTION TEST REPORT



Symbol	%+3"	% GRAVEL	% SAND	% SILT	% CLAY
○	0.0	0.0	5.2	60.4	34.4
△	0.0	0.6	13.8	53.3	32.4
□	0.0	0.0	3.7	58.5	37.8

	LL	PI	D_{85}	D_{60}	D_{50}	D_{30}	D_{15}	D_{10}	C_c	C_u
	39	19			0.01	0.004				
	36	16			0.01	0.004				
□	44	21			0.01	0.003				

MATERIAL DESCRIPTION	USCS
○ Brown Lean CLAY, Little Sand	CL
△ Brown Lean CLAY, Some Sand, Trace Gravel	CL
□ Brown Lean CLAY, Trace Sand	CL

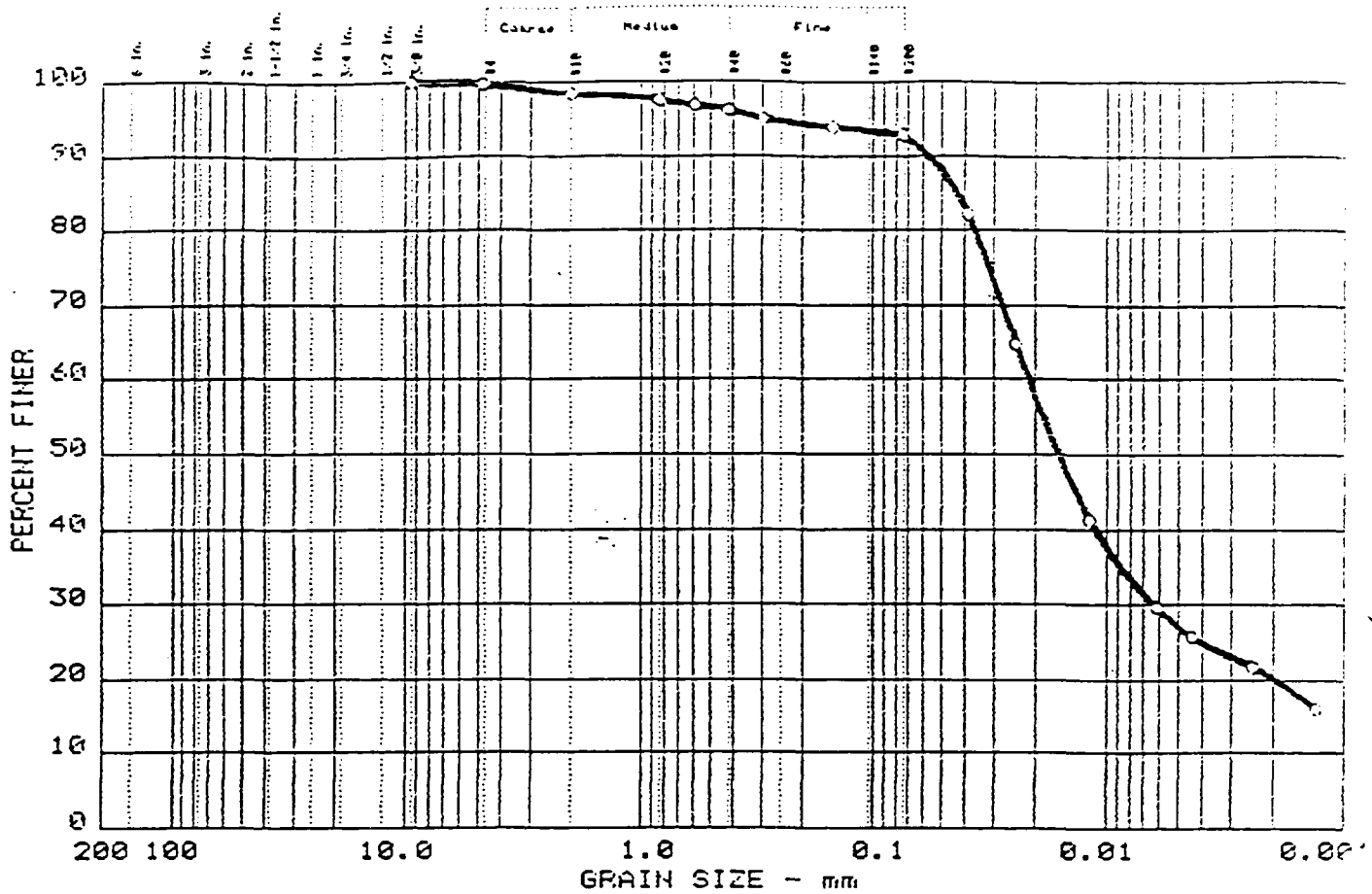
Project No.: 5822.41
Project:
○ Sample: TP-1 SAMPLE # 3 @ 1.5-3.0 FT
△ Sample: TP-2 SAMPLE # 3 @ 1.0-2.5 FT
□ Sample: TP-3 SAMPLE # 3 @ 1.0-2.5 FT
Date: 5/14/91

Remarks:
TESTED BY DWA | RP
CHECKED BY DWA
APPROVED BY VJR

GRAIN SIZE DISTRIBUTION TEST REPORT
WARZYN ENGINEERING INC.

Sheet No.

GRAIN SIZE DISTRIBUTION TEST REPORT



Symbol	% +3"	% GRAVEL	% SAND	% SILT	% CLAY
O	0.0	0.3	6.7	66.0	27.0

	LL	PI	D_{85}	D_{60}	D_{50}	D_{30}	D_{15}	D_{10}	C_c	C_u
O	37	15			0.02	0.006				

MATERIAL DESCRIPTION	USCS
O Brown Lean CLAY, Little Sand	CL

Project No.: 12153.01
Project:
O Sample: Sample # 11

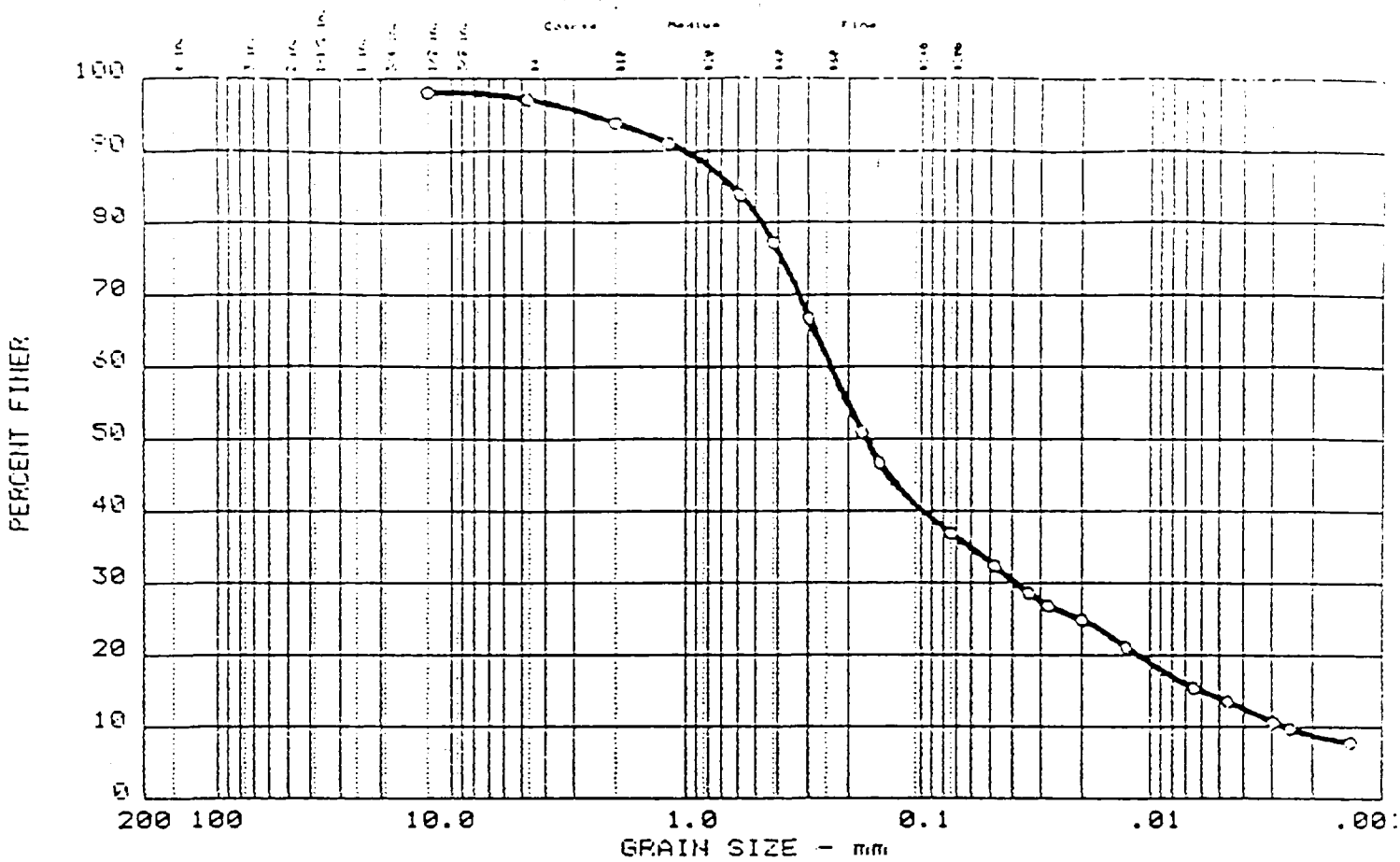
Date: 7/23/91

Remarks:
TESTED BY DWA/RWF
CHECKED BY DWA
APPROVED BY VJR

GRAIN SIZE DISTRIBUTION TEST REPORT
WARZYN, INC.

Sheet No.

GRAIN SIZE DISTRIBUTION TEST REPORT



Symbol	%+3"	% GRAVEL	% SAND	% SILT	% CLAY
O	0.0	2.9	60.1	23.0	13.9

	LL	PI	D_{85}	D_{60}	D_{50}	D_{30}	D_{15}	D_{10}	C_c	C_u
O	20	9	0.63	0.24	0.17	0.038	0.0060	0.0026	2.37	92.3

MATERIAL DESCRIPTION	USCS
O Brown Fine-Coarse SAND, Some Silt & Clay, Trace Gravel	SC

Project No.: 50422.00
Project:
O Sample: BORING: 3 SAMPLE: 6C @ 38.5-40.0 FT

Date: 07/14/89

Remarks:
TESTED BY: MML/DWA
ENTERED BY: MML
CHECKED BY:
APPROVED BY:

GRAIN SIZE DISTRIBUTION TEST REPORT
WARZYN ENGINEERING INC.

Sheet No.

WARZYN

May 6, 1988
99194

PARTICLE-SIZE ANALYSIS OF SOIL

ASTM D 422-63

Procedure to make dispersing agent and calibration:

1. Add 40 grams Sodium Hexametaphosphate to 1000 cc of De-aired Demineralized Water at 20°C. (160gm/4000cc)
2. Add 11.7 grams Sodium Bicarbonate to each 1000cc of agent (brings PH to 8.3) (35 gm/4000cc)
3. Allow to Stand for 24 Hours Before Using. Mix Thoroughly.
4. Mark Stock Solution with Date and Discard at 30 days.
5. Each Soil Sample Receives 125 ml of Stock Solution.
6. Add 125 ml of Stock Solution to a 1000 ml Sedimentation Cylinder and Fill to the 1000 ml mark with Demineralized Water at 20° C.
7. Record Correction Factor with a 152H Hydrometer. Record Temp. (should be 20°).

HYDROMETER CORRECTION

FLUID	SOLIDS ADDED	HYDROMETER READING GM/LITER (152 H Hydrometer)
Cold Tap Water	None	+1
Hot Tap Water	None	0
Hot Tap Water	Deflock Agent	+6
Cold Tap Water	Deflock Agent	+7
Distilled Water	Deflock Agent	+6

99194.00
DWA/mml/DWA
[mml-401-92]

APPENDIX B-16

DENSITY (WARZYN)

American Association State
Highway and Transportation Officials Standard
AASHTO No. T 100

Standard Test Method for SPECIFIC GRAVITY OF SOILS[1]

This standard is issued under the fixed designation D 854; 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 determination of the specific gravity of soils by means of a pycnometer. When the soil is composed of particles larger than the No. 4 (4.75-mm) sieve, the method outlined in Test Method C 127 shall be followed. When the soil is composed of particles both larger and smaller than the No. 4 sieve, the sample shall be separated on the No. 4 sieve and the appropriate test method used on each portion. The specific gravity value for the soil shall be the weighted average of the two values (Note 1). When the specific gravity value is to be used in calculations in connection with the hydrometer portion of Method D 422, it is intended that the specific gravity test be made on that portion of the soil which passes the No. 10 (2.00-mm) sieve.

NOTE 1—The weighted average specific gravity should be calculated using the following equation:

$$G_{avg} = \frac{1}{\frac{R_1}{100G_1} + \frac{P_1}{100G_2}}$$

where:

G_{avg} = weighted average specific gravity of soils composed of particles larger and smaller than the No. 4 (4.75-mm) sieve,

R_1 = percent of soil particles retained on the No. 4 sieve,

P_1 = percent of soil particles passing the No. 4 sieve,

G_1 = apparent specific gravity of soil particles retained on the No. 4 sieve as determined by Test Method C 127, and

G_2 = specific gravity of soil particles passing the No. 4 sieve as determined by this test method.

1.2 The values stated in acceptable metric units are to be regarded as the standard.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Documents

2.1 *ASTM Standards:*

C 127 Test Method for Specific Gravity and Absorption of Coarse Aggregate[2]

C 670 Practice for Preparing Precision Statements for Test Methods for Construction Materials[2]

D 422 Method for Particle-Size Analysis of Soils[3]

E 12 Definitions of Terms Relating to Density and Specific Gravity of Solids, Liquids, and Gases[4]

3. Definition

3.1 *specific gravity*—the ratio of the mass of a unit volume of a material at a stated temperature to the mass in air of the same volume of gas-free distilled water at a stated temperature (per Definitions E 12).

---

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

Current edition approved Nov. 28, 1983. Published January 1984. Originally issued as D 854 – 45. Last previous edition D 854 – 58 (1979).

[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vol 04.08.

[4] *Annual Book of ASTM Standards*, Vol 15.05.

4. Significance and Use

4.1 The specific gravity of a soil is used in almost every equation expressing the phase relationship of air, water, and solids in a given volume of material.

4.2 The term "solid particles," as used in geotechnical engineering, is typically assumed to mean naturally occurring mineral particles that are not very soluble in water. Therefore, the specific gravity of materials containing extraneous matter (such as cement, lime, etc.), water-soluble matter (such as sodium chloride), and soils containing matter with a specific gravity of less than one, typically require special treatment or a qualified definition of specific gravity.

5. Apparatus

5.1 *Pycnometer*—Either a volumetric flask having a capacity of at least 100 mL or a stoppered bottle having a capacity of at least 50 mL (Note 2). The stopper shall be of the same material as the bottle, and of such size and shape that it can be easily inserted to a fixed depth in the neck of the bottle, and shall have a small hole through its center to permit the emission of air and surplus water.

NOTE 2—The use of either the volumetric flask or the stoppered bottle is a matter of individual preference, but in general, the flask should be used when a larger sample than can be used in the stoppered bottle is needed due to maximum grain size of the sample.

5.2 *Balance*—Either a balance sensitive to 0.01 g for use with the volumetric flask, or a balance sensitive to 0.001 g for use with the stoppered bottle.

6. Calibration of Pycnometer

6.1 The pycnometer shall be cleaned, dried, weighed, and the weight recorded. The pycnometer shall be filled with distilled water (Note 3) essentially at room temperature. The weight of the pycnometer and water, W_a, shall be determined and recorded. A thermometer shall be inserted in the water and its temperature T_i determined to the nearest whole degree.

NOTE 3—Kerosine is a better wetting agent than water for most soils and may be used in place of distilled water for oven-dried samples.

6.2 From the weight W_a determined at the observed temperature T_i a table of values of weights W_a shall be prepared for a series of temperatures that are likely to prevail when weights W_b are determined later (Note 4). These values of W_a shall be calculated as follows:

$$W_a \text{ (at } T_x) = (\text{density of water at } T_x/\text{density of water at } T_i) \times (W_a(\text{at } T_i) - W_f) + W_f$$

where:

W_a = weight of pycnometer and water, g,
W_f = weight of pycnometer, g,
T_i = observed temperature of water, °C, and
T_x = any other desired temperature, °C.

NOTE 4—This method provides a procedure that is most convenient for laboratories making many determinations with the same pycnometer. It is equally applicable to a single determination. Bringing the pycnometer and contents to some designated temperature when weights W_a and W_b are taken, requires considerable time. It is much more convenient to prepare a table of weights W_a for various temperatures likely to prevail when weights W_b are taken. It is important that weights W_a and W_b be based on water at the same temperature. Values for the relative density of water at temperatures from 18 to 30°C are given in Table 1.

7. Sampling

7.1 The soil to be used in specific gravity test may contain its natural moisture or be oven-dried. The weight of the test sample on an oven-dry basis shall be at least 25 g when the volumetric flask is to be used, and at least 10 g when the stoppered bottle is to be used.

7.2 *Samples Containing Natural Moisture*—When the sample contains its natural moisture, the weight of the soil, W_m, on an oven-dry basis shall be determined at the end of the test by evaporating the water in an oven maintained at 230 ±9°F (110 ±5°C) (Note 5). Samples of clay soils containing their natural moisture content shall be dispersed in distilled water before placing in the flask, using the dispersing equipment specified in Method D 422 (Note 6).

7.3 *Oven-Dried Samples*—When an oven-dried sample is to be used, the sample shall be dried for at least 12 h, or to constant weight, in an oven maintained at 230 ±9°F (110 ±5°C) (Note 5), cooled in a desiccator, and weighed upon removal from the desiccator. The sample shall then be soaked in distilled water for at least 12 h.

NOTE 5—Drying of certain soils at 110°C may bring about loss of moisture of composition or hydration, and in such cases drying shall be done, if desired, in reduced air pressure and at a lower temperature.

NOTE 6—The minimum volume of slurry that can be prepared by the dispersing equipment specified in Method D 422 is such that a 500-mL flask is needed at the pycnometer.

8. Procedure

8.1 Place the sample in the pycnometer, taking care not to lose any of the soil in case the weight of the sample has been determined. Add distilled water to fill the volumetric flask about three-fourths full or the stoppered bottle about half full.

8.2 Remove entrapped air by either of the following methods: (*1*) subject the contents to a partial vacuum (air pressure not exceeding 100 mm Hg) or (2) boil gently for at least 10 min while occasionally rolling the pycnometer to assist in the removal of the air. Subject the contents to reduced air pressure either by connecting the pycnometer directly to an aspirator or vacuum pump, or by use of a bell jar. Some soils boil violently when subjected to reduced air pressure. It will be necessary in those cases to reduce the air pressure at a slower rate or to use a larger flask. Cool samples that are heated to room temperature.

8.3 Fill the pycnometer with distilled water, clean the outside and dry with a clean, dry cloth. Determine the weight of the pycnometer and contents, W_b, and the temperature in degrees Celsius, T_x, of the contents as described in Section 6.

9. Calculation and Report

9.1 Calculate the specific gravity of the soil, based on water at a temperature T_x, as follows:

$$\text{Specific gravity, } T_x/T_x = W_o/[W_o + (W_a - W_b)]$$

where:

W_o = weight of sample of oven-dry soil, g,

W_a = weight of pycnometer filled with water at temperature T_x (Note 7), g,

W_b = weight of pycnometer filled with water and soil at temperature T_x, g, and

T_x = temperature of the contents of the pycnometer when weight W_b was determined, °C.

NOTE 7—This value shall be taken from the table of values of W_a, prepared in accordance with 6.2, for the temperature prevailing when weight W_b was taken.

9.2 Unless otherwise required, specific gravity values reported shall be based on water at 20°C. The value based on water at 20°C shall be calculated from the value based on water at the observed temperature T_x, as follows:

$$\text{Specific gravity, } T_x/20\text{ C} = K \times \text{specific gravity, } T_x/T_x$$

where:

K = a number found by dividing the relative density of water at temperature T_x by the relative density of water at 20°C. Values for a range of temperatures are given in Table 1.

9.3 When it is desired to report the specific gravity value based on water at 4°C, such a specific gravity value may be calculated by multiplying the specific gravity value at temperature T_x by the relative density of water at temperature T_x.

9.4 When any portion of the original sample of soil is eliminated in the preparation of the test sample, the portion on which the test has been made shall be reported.

10. Precision and Bias

10.1 Criteria for judging the acceptability of specific gravity test results obtained by this test method on material passing the No. 4 (4.75-mm) sieve are given as follows (Note 8):

Material and Type Index	Standard Deviation[A]	Acceptable Range of Two Results (percent of mean)[A]
Single-operator precision:		
Cohesive soils	0.021	0.06
Noncohesive soils	[B]	[B]
Multilaboratory precision:		
Cohesive soils	0.056	0.16
Noncohesive soils	[B]	[B]

[A] These numbers represent, respectively, the (1S) and (D2S limits as described in Practice C 670.

[B] Criteria for assigning standard deviation values for noncohesive soils are not available at the present time.

NOTE 8—The figures given in Column 2 are the standard deviations that have been found to be appropriate for the materials described in Column 1. The figures given in Column 3 are the limits that should not be exceeded by the difference between the results of two properly conducted tests.

TABLE 1 Relative Density of Water and Conversion Factor K For Various Temperatures

Temperature, °C	Relative Density of Water	Correction Factor *K*
18	0.9986244	1.0004
19	0.9984347	1.0002
20	0.9982343	1.0000
21	0.9980233	0.9998
22	0.9978019	0.9996
23	0.9975702	0.9993
24	0.9973286	0.9991
25	0.9970770	0.9989
26	0.9968156	0.9986
27	0.9965451	0.9983
28	0.9962652	0.9980
29	0.9959761	0.9977
30	0.9956780	0.9974

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K.3

WARZYN

PROJECT
6095300

QUALITY ASSURANCE PROJECT PLAN (QAPP)
REMEDIAL INVESTIGATION/FEASIBILITY STUDY

H.O.D. LANDFILL
ANTIOCH, ILLINOIS

VOLUME 2 OF 2

SEPTEMBER 1992

PREPARED FOR:
WASTE MANAGEMENT OF ILLINOIS, INC.
WESTCHESTER, ILLINOIS

• • •

PREPARED BY:
WARZYN INC.
ADDISON, ILLINOIS

QUALITY ASSURANCE PROJECT PLAN (QAPP)
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
H.O.D. LANDFILL SITE
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LIST OF ACRONYMS AND ABBREVIATIONS

AOC	Administrative Order on Consent
ARAR	Applicable or Relevant and Appropriate Requirements
ASTM	American Standards for Testing Materials
CDO	Central District Office
CLP	Contract Laboratory Program
CRDL	Contract Required Detection Limits
CRQL	Contract Required Quantitation Limits
CRL	Central Regional Laboratory
DQO	Data Quality Objective
ESI	Expanded Site Inspection
ETC	Environmental Testing and Certification Corporation
FIT	Field Investigation Team
HRS	Hazard Ranking System
HSP	Site Specific Health and Safety Plan
IEPA	Illinois Environmental Protection Agency
LL TAL	Low Level Detection Target Analyte List
LL TCL	Low Level Detection Target Compound List
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NPL	National Priorities List
PA	Preliminary Assessment
PRP	Potentially Responsible Party
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QAS	Quality Assurance Section
QC	Quality Control
RI/FS	Remedial Investigation/Feasibility Study
RMAL	Rocky Mountain Analytical Laboratory
RPD	Relative Percent Difference
RPM	Remedial Project Manager
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SOW	Statement of Work
TAL	Target Analyte List
TDS	Total Dissolved Solids
TCL	Target Compound List
TIC	Tentatively Identified Compound
TOC	Total Organic Carbon
U.S. EPA	United States Environmental Protection Agency, Region V
VOA	Volatile Organic Analysis
Warzyn	Warzyn Inc.
WMII	Waste Management of Illinois, Inc.

APPENDIX C

FIELD MEASUREMENT SOPs

APPENDIX C-1

PH

pH

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: EPA 1983, p. 150.1

Sensitivity: 0.01 pH unit

Optimum Range: pH 1.00 to 12.00

Sample Handling: Determine on-site, if possible, otherwise within 24 hours.

Reagents and Apparatus:

1. pH meter
2. Combination pH electrode.
3. Magnetic stirrer and stir bars (for lab use).
4. Beakers or plastic cups.
5. pH buffer solutions, pH 4.00, 7.00, and 10.0.
6. Deionized water

Notes:

1. The pH test is temperature dependent. Therefore, temperatures of buffers and samples should be within 2°C of each other. For refrigerated or cool samples, use refrigerated buffers to calibrate meter.

2. Interferences in pH measurements occur with presence of weak organic and inorganic salts, and oil and grease. If oil and grease are visible, note on data sheet. Clean electrode with soap and water, followed by 10% HCl and deionized water. Then recalibrate meter before analysis of next sample.

3. Electrode should be stored in pH 7.00 buffer, or electrode storage solution.

4. **Before leaving laboratory for field work:**

   a. Check batteries.

   b. Do quick calibration at pH 7.00 and 4.00 to check electrode response and batteries.

   c. Obtain fresh pH buffer solutions.

5. **Following field measurements:**

   a. Report any problems with meter or electrode.

b. Clean meter and meter case.

c. Make sure electrode is stored in pH 7.00 buffer.

Calibration:

1. Place combination electrode in fresh pH 7.00 buffer solution.
2. After allowing meter to stabilize, turn calibration dial until reading of 7.00 is obtained.
3. Rinse electrode with deionized water and place in pH 4.00 buffer solution.
4. Wait for reading to stabilize and then turn slope adjustment dial until reading of 4.00 is obtained.
5. Rinse electrode with deionized water and place in pH 7.00 buffer. If meter reading is not 7.00 + 0.05, follow Steps 2-5 again.
6. Rinse electrode with deionized water and place in pH 10.00 buffer. Reading must be in the range of 9.90-10.10 or calibration must be repeated.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed and deionized water rinsed prior to analyses.
2. Calibrate meter using calibration procedure.
3. Pour the sample into clean beaker or plastic cup.
4. Place stir bar in beaker and put on magnetic stirrer (low speed) for lab measurement of pH. Swirl cup gently for field measurement of pH.
5. Check temperature of sample. It should be $\pm$ 2°C of the buffer solutions.
6. Rinse electrode with deionized water.
7. Immerse electrode in sample. The white KCl junction on side or bottom of electrode must be fully immersed in solution. Allow sufficient time for reading to stabilize. Record pH. Rinse electrode with deionized water.
8. Check calibration with pH 7.00 buffer solution initially and after every 10 samples and at the end of the analytical run and record on data sheet. Buffer solution should read $\pm$0.05 of true value.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates should be within acceptable ranges. Average the results.

Revision Dates

7-22-86

6-28-90

1-8-92

Manager, Operations

Supervisor, Quality Assurance

Kim Johnson

Director, Analytical Services

APPENDIX C-2

CONDUCTIVITY

CONDUCTIVITY YSI METER

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Specific Conductance (Electrical Conductivity), umhos/cm @ 25°C

Reference: EPA 1983, Method 120.1

Detection Limit: 10 umhos/cm @ 25°C

Sample Handling: Determine on-site or within 28 days

Reagents and Apparatus:

1. Conductivity meter, YSI 33 SCT
2. Deionized water
3. conductivity standard, 1000 umhos/cm @ 25°C. Commercially Available

Notes:

1. All conductivity readings must be corrected to 25°C.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed and deionized rinsed prior to analysis.

2. With mode switch of the meter in the OFF position. Check the zero setting. If not at zero, use meter adjusting screw to zero (on front of the meter).

3. Plug probe into jack located on side of meter.

4. Turn mode switch to red line, and turn red line knob until needle aligns with red line on dial. Change batteries if meter cannot be aligned.

5. Analyze the conductivity standard. If the result is within 90-110% of the standard value, analyze samples. A standard should be analyzed after every 10 samples and at the end of the analytical run. Table 1 lists non-temperature compensated values for both of the standards. Use this table as a reference when a calculator is not available to perform temperature correction on the standards.

6. Totally immerse and suspend the probe in the water sample. Do not allow probe to touch the sides of the sample container.

7. Turn mode switch to the appropriate conductivity scale (X100, X10, or X1). Use the scale that produces a mid-range output on the meter.

8. Wait for needle to stabilize (about 15 seconds) and record conductivity. Multiply the conductivity reading by the scale setting.

9. While gently agitating the probe, take sample temperature (°C) to nearest 0.5°C and record.

Table 1
NON-TEMPERATURE COMPENSATED CONDUCTIVITY STANDARD READINGS FOR 1,000 AND 10,000 umhos/cm STANDARDS

Conductivity Standard 1,000 umhos/cm			Conductivity Standard 10,000 umhos/cm		
Conductivity Standard Should Read	at	Degrees C°	Conductivity Standard Should Read	at	Degrees C°
500		0	5000		0
520		1	5200		1
540		2	5400		2
560		3	5600		3
580		4	5800		4
600		5	6000		5
620		6	6200		6
640		7	6400		7
660		8	6600		8
680		9	6800		9
700		10	7000		10
720		11	7200		11
740		12	7400		12
760		13	7600		13
780		14	7800		14
800		15	8000		15
820		16	8200		16
840		17	8400		17
860		18	8600		18
880		19	8800		19
900		20	9000		20
920		21	9200		21
940		22	9400		22
960		23	9600		23
980		24	9800		24
1000		25	10,000		25
1020		26	10,200		26
1040		27	10,400		27
1060		28	10,600		28
1080		29	10,800		29
1100		30	11,000		30
1120		31	11,200		31
1140		32	11,400		32
1160		33	11,600		33
1180		34	11,800		34
1200		35	12,000		35

10. Rinse probe with deionized water.

11. Record specific conductivity and temperature.

12. Store probe in D.I. water between uses whenever possible.

13. Turn mode switch to the OFF position when finished.

Quality Control:

1. A quality control calibration standard of 1000 umhos/cm is to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard is still required. The last sample analyzed in the run is to be the calibration standard. These standards must be within 90-110% of the standard value or the samples run after the last acceptable check standard are to be reanalyzed.

2. Duplicate a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates are to be averaged. Duplicate values are to be within acceptable ranges.

Calculation:

1. **Calculate specific conductivity at 25°C using following formula:**

$$G_{25} = \frac{G_T}{[1 + 0.02\,(T-25)]}$$

G_{25} = Specific conductivity at 25°C, umhos/cm

T = Temperature of sample, °C

G_T = Conductivity of sample at temperature T, umhos/cm

	Revision Date
	9-06-86
	4-27-87
	2-17-88
	2-09-90
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Supervisor, Quality Assurance	
Director, Analytical Services	

APPENDIX C-3

TEMPERATURE

Effective Date: 3-13-92

FIELD TEMPERATURE

Scope and Application: This method is applicable to surface water, wastewater, and groundwater.

Method: Thermometric

Reference: EPA 1983, Method 170.1

Reagents and Apparatus:

1. Thermometer; mercury-filled, or temperature probe, °C
2. Plastic bottles, 250 mL
3. Deionized water
4. Kimwipes

Notes:

1. The thermometer or temperature probe must be calibrated annually against an ASTM certified thermometer.

2. It is recommended the thermometer or temperature probe have a range of -10 to 100°C with 0.5° sensitivity.

Procedure:

1. Pour approximately 100-150 mL sample into a plastic bottle.

2. Place thermometer or temperature probe in sample and swirl gently for 30-60 seconds.

3. Record the temperature to the nearest 0.5 degree.

4. Rinse thermometer with deionized water and blot dry with a kimwipe.

5. Continue as above with next sample.

Quality Control:

1. Duplicate minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates are to be averaged. Duplicate values are to be within acceptable ranges.

APPENDIX C-4

DISSOLVED OXYGEN

YSI DISSOLVED OXYGEN METER AND PROBE

Scope and Application: The instructions outlined below are to be followed for the daily calibration and routine operation of the YSI Dissolved Oxygen Meter and Probe.

Reference: Instruction Manual YSI Model 54ARC Dissolved Oxygen Meter and YSI 5700 Dissolved Oxygen Probe.

Reagents and Apparatus:

1. YSI 54ARC Dissolved Oxygen Meter
2. YSI 5720A B.O.D. Bottle Probe
3. Membrane and KCl kit, standard, YSI 5775
4. Replacement "O" ring, YSI Part #5945
5. Beater boot assembly, YSI Part #5486

Operating Procedure:

I. Preparing the Probe

All YSI 5700 Series Probes have similar sensors and should be cared for in the same manner. They are precision devices relying on good treatment if high accuracy measurements are to be made. Prepare the probe as follows.

ALL PROBES ARE SHIPPED DRY - FOLLOW THESE INSTRUCTIONS TO PREPARE FOR USE

1. Prepare the electrolyte by dissolving the KCl crystals in a dropper bottle with Milli-Q water. Fill the bottle to the top.

2. Remove the "O" ring and membrane. Thoroughly rinse the sensor with KCl solution.

3. Fill the probe with electrolyte as follows (see Figure 1):

   a. Grasp the probe with your left hand.

   b. Fill the sensor body until no more air bubbles appear. Tap the probe against the countertop to dislodge any air bubbles adhering to the sensor.

   c. Secure a membrane under your left thumb. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, keeping it clean and dust free, touching it only at the ends.

   d. With the thumb and forefinger of your other hand, grasp the free end of the membrane.

e. Using a continuous motion stretch the membrane UP, OVER, and DOWN the other side of the sensor. Stretching forms the membrane to the contour of the probe. The membrane can be stretched to approximately 1 1/2 times its normal size.

f. Secure the end of the membrane under the forefinger of the hand holding the probe.

g. Roll the "O" ring over the end of the probe. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the "O" ring.

h. Trim off excess membrane with scissors or sharp knife. Check that the stainless steel temperature sensor is not covered by excess membrane.

4. Shake off excess KCl.

5. Store the probe in a BOD bottle containing about 1 inch of water.

6. Membranes average replacement is 2-4 weeks. If the electrolyte in the probe is allowed to evaporate, air bubbles form under the membrane. If air bubbles are noted under the membrane or if the membrane becomes damaged, thoroughly flush the reservoir with fresh KCl and install a new membrane as described above.

7. Replace the membrane if erratic readings are observed or if calibration is not stable.

NOTE: The gold cathode should always be bright and untarnished. If it is tarnished (which can result from contact with certain gases) or plated with silver (which can result from extended use with a loose or wrinkled membrane), return it to the factory for service. Never use chemicals or abrasives in an attempt to clean it.

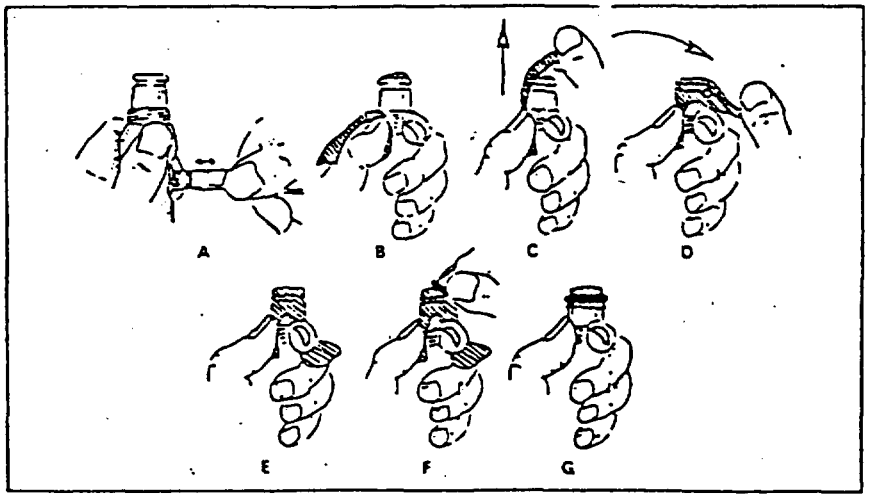


Figure 1

II. Preparing the YSI Instrument

It is important that the instrument be placed in the intended operating position; vertical, tilted, or on its back - before it is prepared for use and calibrated. (See Figure 8). Recalibration may be necessary when the instrument operating position is changed. After preparing the probe proceed as follows:

1. With switch in the OFF position, adjust the meter pointer to Zero with the screw in the center of the meter panel. Readjustment may be necessary if the instrument operating position is changed.

2. Switch to RED LINE and adjust with the RED LINE knob until the meter needle aligns with the red mark at the 31°C position.

3. Switch to ZERO and adjust to zero with zero control knob.

4. Attach the prepared probe to the PROBE connector of the instrument and adjust the retaining ring finger tight.

5. For optimum probe stabilization, let the meter and probe equilibrate for 15 minutes before calibrating.

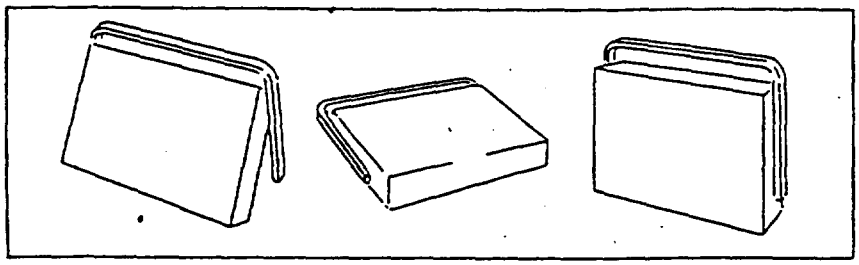


Figure 2

III. Calibration

The operator has a choice of three calibration methods: Winkler titration, Saturated Water, and Air. The three methods are described in the following paragraphs. The Winkler titration is the preferred method of calibration.

Winkler Titration

1. Determine the dissolved oxygen in two samples from the aerated water source using the Winkler titration technique (refer to the Dissolved Oxygen SOP) and average the values. If the values differ by more than 0.5 mg/L, discard the samples and repeat.

2. Place the YSI probe in the third sample and stir.

3. Switch to desired mg/L scale range and adjust with the CALIBRATION control to the average value determined in Step 1. Allow the probe to remain in the sample for at least two minutes before setting the calibration value, and leave in the sample for an additional 2 minutes to verify stability (Readjust if necessary).

Saturated Water Calibration

1. Air saturate a volume of water by aerating for at least 1 hour at a constant temperature (preferably room temperature).

2. Place the probe in the sample and stir. Switch to TEMPERATURE scale. Refer to Calibration Table I for the mg/L value corresponding to the temperature.

3. Determine local altitude or the "true" atmospheric pressure (note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level). Using Calibration Table II determine the correction factor for your pressure or altitude.

4. Multiply the mg/L value from Table I by the correction factor from Table II to determine the corrected calibration value for your conditions.

   EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/L. From Table II the correction factor for 1000 feet is about 0.96. The corrected calibration value is 8.9 mg/L x 0.96 = 8.54 mg/L.

5. Switch to an appropriate mg/L scale range and adjust the CALIBRATE knob while stirring until the meter reads the corrected calibration value from Step 4. Leave the probe in the sample for two minutes to verify calibration stability. Readjust if necessary.

Air Calibration - Fresh Water

1. Place the probe in a BOD bottle partially filled with water. Wait approximately 10 minutes for temperature stabilization. This may be done simultaneously while the probe is stabilizing.

2. Switch to TEMPERATURE and read. Refer to Table I - Solubility of Oxygen in Fresh Water, and determine calibration value.

3. Determine altitude or atmospheric correction factor using Table II.

4. Multiply the calibration value from Table I by the correction factor from Table II.

   EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/L. From Table II the correction factor for 1000 feet is about 0.96. Therefore the corrected calibration value is 8.9 mg/L x 0.96 = 8.54 mg/L.

5. Switch to an appropriate mg/L range and adjust the CALIBRATE knob until the meter reads the corrected calibration value from Step 4. Wait two minutes to verify calibration stability. Readjust if necessary.

IV. Dissolved Oxygen Measurement

1. With the instrument prepared for use and the probe calibrated, place the probe in the sample to be measured and turn on stirring boot.

2. Allow sufficient time for probe to stabilize to sample.

3. Read dissolved oxygen directly from scale.

V. Maintenance of the Stirrer Boot

1. The probe uses a flexible stirring boot to transmit motion from the sealed motor housing to the sample. If the boot shows signs of cracking or other damage which may allow leaking into the motor housing, the boot must be replaced.

2. In fresh water applications boot life is normally several years, but this may be shortened by exposure to hydrocarbons, moderate to strong acids or bases, ozone, or direct sunlight. For maximum life rinse the boot with deionized water after use in contaminated samples.

3. Boot replacement is as follows:

   a. Pull off old assembly and clean shaft.

   b. Slide on new assembly making sure the back spring is on the grooved area of the shaft. A small amount of rubber cement may be used.

   c. Check that there is sufficient clearance between the tip and the end of the shaft to permit turning without binding.

TABLE I

Solubility of Oxygen in Fresh Water

Temperature °C	mg/L Dissolved Oxygen	Temperature °C	mg/L Dissolved Oxygen
0	14.60	23	8.56
1	14.19	24	8.40
2	13.81	25	8.24
3	13.44	26	8.09
4	13.09	27	7.95
5	12.75	28	7.81
6	12.43	29	7.67
7	12.12	30	7.54
8	11.83	31	7.41
9	11.55	32	7.28
10	11.27	33	7.16
11	11.01	34	7.05
12	10.76	35	6.93
13	10.52	36	6.82
14	10.29	37	6.71
15	10.07	38	6.61
16	9.85	39	6.51
17	9.65	40	6.41
18	9.45	41	6.31
19	9.26	42	6.22
20	9.07	43	6.13
21	8.90	44	6.04
22	8.72	45	5.95

Source: Derrived from 16th Edition "Standard Methods for the Examination of Water and Wastewater".

This table shows the amount of oxygen in mg/L that is dissolved in air saturated fresh water at sea level (760 mm Hg atmospheric pressure) as temperature varies from 0° to 45°C.

Table II

Correction for Atmospheric Pressure

Atmospheric Pressure mm/Hg	or	Equivalent Altitude Ft.	=	Correction Factor
775		540		1.02
760		0		1.00
745		542		.98
730		1094		.96
714		1688		.94
699		2274		.92
684		2864		.90
669		3466		.88
654		4082		.86
638		4756		.84
623		5403		.82
608		6065		.80
593		6744		.78
578		7440		.76
562		8204		.74
547		8939		.72
532		9694		.70
517		10472		.68
502		11273		.66

Source: Derrived from 16th Edition "Standard Methods for the Examination of Water and Wastewater".

This table shows the correction factor that should be used to correct calibration value for the effects of atmospheric pressure or altitude. Find true atmospheric pressure in the left hand column and read across to the right hand column to determine the correction factor. (Note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level.) If atmoshperic pressure is unknown, the local altitude may be substituted. Select the altitude in the center column and read across to the right hand column for the correction factor.

YSI DISSOLVED OXYGEN METER AND PROBE

Detection Limit: 1.00 mg/L Dissolved Oxygen

Working Concentration Range: 0 - 15 mg/L Dissolved Oxygen

YSI 5700 SERIES DISSOLVED OXYGEN PROBES INSTRUCTIONS

The probes described in these instructions are designed for direct use with YSI Models 50, 51B, 54ABP, 54ARC, 56, 57 and 58 Dissolved Oxygen Meters. The probes can also be used with discontinued YSI Models 51A, 54BP and 54RC Dissolved Oxygen Meters when the YSI 5735 Cable Adapter is employed.

PRINCIPLES OF OPERATION

YSI 5700 Series Probes are polarographic sensors. A thin permeable membrane stretched over the sensor isolates the electrodes from the environment, but allows gases to enter. When a polarizing voltage is applied across the sensor, oxygen that has passed through the membrane reacts at the cathode, causing a current to flow.

The membrane passes oxygen at a rate proportional to the difference across it in partial pressure of oxygen. Since oxygen is rapidly consumed at the cathode, it can be assumed that the oxygen pressure under the membrane is zero. Hence, the force causing the oxygen to diffuse through the membrane is proportional to the partial pressure of oxygen outside the membrane. As the oxygen partial pressure varies, both the oxygen diffusion through the membrane and the probe current will change proportionally.

SPECIFICATIONS

Cathode: Gold
Anode: Silver
Membrane: .001" FEP Teflon, standard
Electrolyte: Half saturated KCl
Temperature Range: -5° to 45°C
15° to 35°C for the 5760 probe
Temperature Accuracy: ±0.2°C
Temperature Compensation: (see instrument specifications)
Polarizing Voltage: 0.8 Volts (nominal)
Probe Current in Air at 30°C: 19 microamps (nominal)
in Nitrogen at 30°C: 0.15 microamps or less
Response Time: Typical response for dissolved oxygen, using standard membranes, is 90% in 10 seconds at a constant temperature of 30°C.
Response at low dissolved oxygen levels is typically 90% in 30 seconds.

ACCESSORIES AND REPLACEMENT PARTS

YSI 5492A Battery Pack for Models 51B and 54A (Powers the submersible stirrers.)
YSI 5735 Cable Adapter (Mates 5700 Series probes with discontinued YSI Models 51A, 54BP and 54RC Dissolved Oxygen Meters)

Accessories for the 5720A, 5739 and 5750

YSI 5680 Probe Reconditioning Kit. Includes a sanding tool and ten adhesive disks.
YSI 5775 Membrane and KCl Kit, Standard. Includes two 15-membrane packets (.001" thick standard FEP Teflon membranes) and a 30 ml bottle of KCl with Kodak Photo Flo.
YSI 5776 Membrane and KCl Kit, High Sensitivity. Includes two 15-membrane packets (.0005" thick FEP Teflon membranes) and a 30 ml bottle of KCl with Kodak Photo Flo. Used for measurements below 15°C and/or for low oxygen levels
YSI 5793 .001" membranes, 10-membrane packet
YSI 5794 .0005" membranes, 10-membrane packet
YSI 5945 O-ring pack (Contains replacement sensor O-rings)

Accessories for the 5720A Only

YSI 5486 Stirrer Boot Assembly

Accessories for the 5739 Only

YSI 5075A Calibration Chamber
YSI 5986 Diaphragm Kit

YSI 5740-10 detachable 10' cable
YSI 5740-25 detachable 25' cable
YSI 5740-50 detachable 50' cable
YSI 5740-100 detachable 100' cable
YSI 5740-150 detachable 150' cable
YSI 5740-200 detachable 200' cable

YSI 5791A Submersible Stirrer with 50' cable for stirrer only
YSI 5795A Submersible Stirrer with 50' combined probe and stirrer cable

YSI 5720A BOD BOTTLE PROBE

The 5720A bottle probe (Figure 1) is used for measuring dissolved oxygen in standard BOD bottles. It is provided with a stirrer powered by a DC supply available for 115 or 230 VAC input.

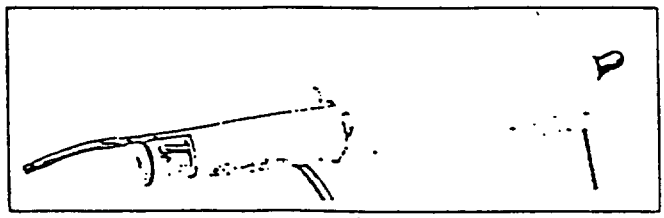


Figure 1. The YSI 5720A Probe

YSI Incorporated
Yellow Springs Instrument Co., Inc., Yellow Springs, Ohio 45387 USA • Phone 513 767-7241 • 800 343-HELP • Fax 513 767-9353 • Telex 205437

To use the 5720A, plug the stirrer power supply into line power and the probe plug in the instrument. With the stirrer off, place the tapered probe end into a filled the BOD bottle and turn on the stirrer. The probe should be operated with a minimum of trapped air in the bottle. A slight amount of air in the unstirred region at the top may be neglected, but no bubble should be permitted around the sensor. CAUTION: The motor housing is not waterproof; do not submerge this probe beyond the part that is inserted into a BOD bottle.

Stirrer Boot (YSI 5486)

The 5720A uses a flexible stirring boot to transmit motion from the motor housing to the sample. If the boot shows signs of cracking or other damage liable to allow leakage into the motor housing, it must be replaced. Running the 5720A with a damaged stirring boot could cause permanent motor damage. Boot life may be shortened by exposure to hydrocarbons, moderate to strong acids or bases, ozone, or direct sunlight. For maximum life, rinse the boot after each use. Boots are replaced as follows:

1. Pull off the old assembly and clean the stir rod housing.

2. Slide on the new assembly, making sure the back spring is over the grooved area of the stir rod housing. A drop of alcohol will aid installation by providing lubrication.

3. Do not permit the stir rod to press against the end of the stirrer boot tip or it will bind.

YSI 5739 DISSOLVED OXYGEN PROBE

The 5739 probe system consists of the probe body plus a detachable cable (see Figure 2). The detachable cable is a convenience feature that facilitates changing cable lengths and replacing damaged cables or probes. The probe and cable assembly is held together with a threaded retainer. The assembly is not intended for casual disconnection; cable and probe should be separated only when necessary.

To detach the cable, unscrew the retainer and slide it down the cable to expose the connector. Pull gently on the connector until it comes away from the probe body. If the O-ring is frayed or damaged, replace it: a replacement O-ring is supplied with each 5740 cable. Reassemble by pushing the connector into the probe body, rotating it until the two halves mate. A light coating of silicone grease on the O-ring will make reassembly easier. Be sure the connector is dry: otherwise, erratic readings may result. Screw on the retainer <u>finger-tight only.</u>

Pressure Compensation

The 5739 probe has a unique pressure compensating system that helps assure accurate readings at great depths. Pressure compensation is effective to 1/2% of reading with pressures up to 100 psi (230 feet of water). The compensating system does not normally require service and should not be taken apart. However, if electrolyte is leaking through the diaphragm, or if there is an obvious puncture, the diaphragm must be replaced. A spare is supplied with the probe. Use a coin to unscrew the retaining plug and remove the washer and diaphragm. With distilled water, flush any salt crystals from the reservoir, install a new diaphragm (flat side out), replace the washer and securely screw in the retaining plug.

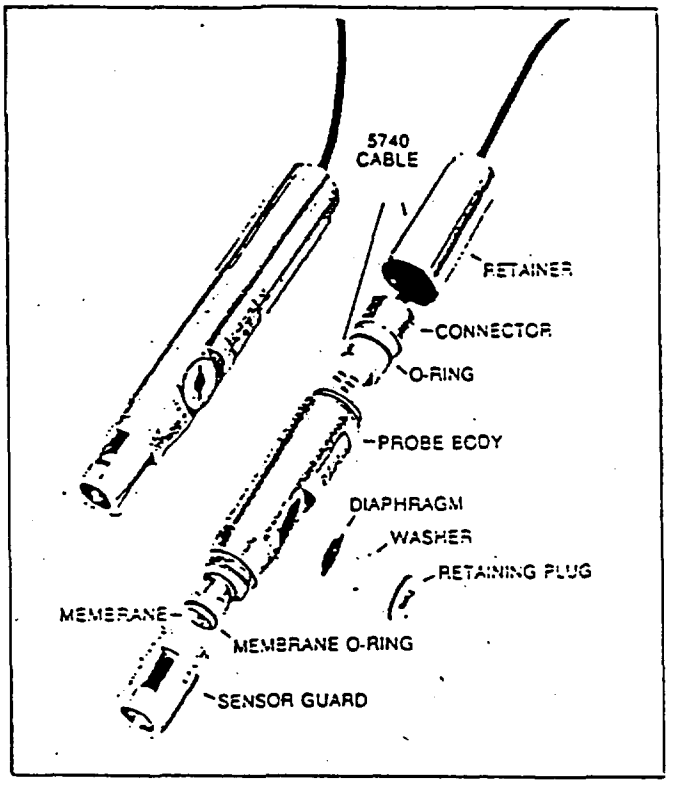


Figure 2. The YSI 5739 Probe

YSI 5750 BOD BOTTLE PROBE

The 5750 (Figure 3) is similar to the 5720A except that it does not have a stirrer. Agitation of the sample must be provided by other means, such as a magnetic stirrer.

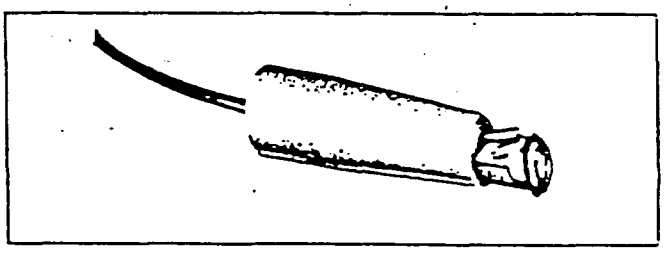


Figure 3. The YSI 5750 Probe

PROBE PREPARATION

All probes are shipped dry. You must follow these instructions when preparing a new probe or when changing membranes. Prepare the electrolyte by dissolving the KCl crystals which are supplied in a dropper bottle that should be filled to the neck with distilled water and shaken until the crystals are dissolved.

1. Unscrew the sensor guard (5739 only). Remove the O-ring and membrane, then thoroughly rinse the sensor with distilled water.

2. To fill the probe with electrolyte and install a new membrane, follow these steps:

a. Grasp the probe in your left hand. (See the sketches in Figure 4.) When preparing the 5739 probe, the pressure compensating port should be to the right. Successively fill the sensor body with electrolyte while pumping the diaphragm with the eraser end of a pencil or a similar soft, blunt tool. Continue filling and pumping until no more

air bubbles appear. For ease in preparing the 5720A, the stirring rod should be to the left. When preparing the 5720A or 5750 probes, simply fill the sensor body until no more air bubbles appear.

b. Secure a membrane between your left thumb and the probe body. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, touching it at the ends only.

c. With the thumb and forefinger of your other hand, grasp the free end of the membrane.

d. With a continuous motion, stretch it up, over and down the other side of the sensor. Stretching forms the membrane to the contour of the probe.

e. Secure the end of the membrane under the forefinger of your left hand while holding the probe.

f. Roll the O-ring over the end of the probe, being careful not to touch the membrane surface. For the 5720A, start at the right side of the sensor and roll the O-ring toward the stirring rod. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the O-ring.

g. Trim off excess membrane with scissors or sharp knife. Check that the stainless steel temperature sensor is not covered by excess membrane.

3. Shake off excess KCl. On the 5739, reinstall the sensor guard.

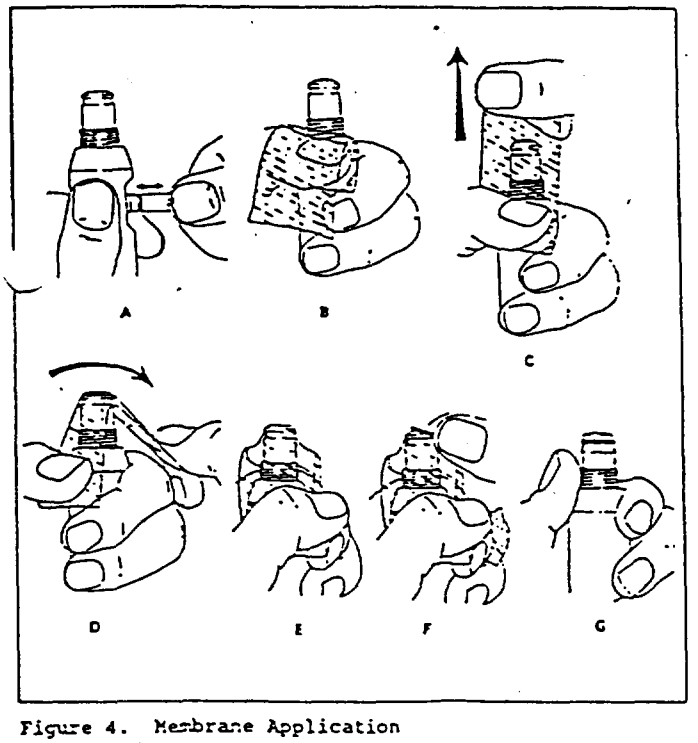


Figure 4. Membrane Application

Probe Storage

A bottomless plastic bottle is provided with the YSI 5739 probe for convenient storage. Place a small piece of moist towel or sponge in the bottle and insert the probe into the open end. This keeps the electrolyte from drying out. The 5720A and 5750 probes can be stored in a BOD bottle containing at least 1" of water.

OPERATING PRECAUTIONS, ALL PROBES

1. Membrane life depends on use. Membranes will last a long time if installed properly and treated with care during use. Erratic readings result from loose, wrinkled or fouled membranes, or from large bubbles in the electrolyte reservoir. If erratic readings, or evidence of membrane damage occur, you should replace the membrane and KCL. The average replacement interval is two to four weeks; electrolyte in constant or heavy use will be exhausted in about two weeks.

If the sensor O-ring on any probe is worn or loose, replace it with the O-ring provided in the YSI 5945 O-ring Pack.

2. The gold cathode should always be bright and untarnished. If it is tarnished (which can result from contact with certain gases) or plated with silver (which can result from extended use with a loose or wrinkled membrane), it needs to have its surface restored. Probes may either be returned to the factory, or cleaned with the YSI 5680 Probe Reconditioning Kit; never use chemicals or any abrasive not supplied with this kit.

3. It is also possible that the silver anode may become contaminated, which will prevent successful calibration. Try soaking the probe overnight in a 3% ammonia solution; rinse with deionized water, recharge with electrolyte, and install a new membrane. If still unable to calibrate after several hours, return the probe for service.

4. Hydrogen sulfide, sulfur dioxide, halogens, and neon are interfering gases. If you suspect erroneous readings, it may be necessary to determine if these are the cause.

These gases have been tested for response:

100% Carbon Monoxide	less than 1%
100% Carbon Dioxide	around 1%
100% Hydrogen	less than 1%
100% Chlorine	2/3 O_2 response
100% Helium	none
100% Nitrous Oxide	1/3 O_2 response
100% Ethylene	none
100% Nitric Oxide	1/3 O_2 response

5. The correct liquid level in BOD bottles is achieved by overfilling, then inserting a stopper and pouring off the excess. When using a YSI 5760 or a 5720A probe in a filled BOD bottle, be careful to insert it slowly to avoid sample overflow.

6. When using the 5720A in samples containing heavy particulate solids, additional stirring may be needed. Inverting the stoppered bottle immediately before use will usually provide adequate mixing.

CALIBRATION

Daily calibration is generally appropriate. Calibration can be disturbed by physical shock, touching the membrane, fouling of the membrane or drying out of the electrolyte. Check calibration after each series of measurements, and in time you will develop a realistic schedule for recalibration. When probes are not in use, store them as recommended in Probe Preparation.

Probes may be calibrated by Winkler Titration or by the Water Saturated Air method. Experience has shown that air calibration is quite reliable, yet far simpler than titration. Both methods are described here. Consult the manual for your particular instrument for more complete instructions.

inkler Titration

Draw a volume of water from a single source and refully divide it into four samples. Determine the ygen in three of the samples using the Winkler Titra-on technique and average the three values. If one of e values differs from the other two by more than 0.5 /L, discard it and average the two values remaining.

. Using the probe-meter system you are calibrating, ace the probe into the fourth sample and stir.

. Switch to the desired mg/L range and adjust the LIBRATION control to the average value determined in ep 1. Allow the probe to remain in the sample for at east 5 minutes before setting the calibration value, en leave it in the sample for an additional two inutes to verify stability. Readjust if necessary.

ir Calibration

. Place the probe in a BOD bottle containing about 1 ch of water. Wait approximately ten minutes for mperature stabilization.

e 5739 probe can be placed in the YSI 5075A Calibra-ion Chamber or in the small calibration bottle sup-lied with the probe (the one with the hole in the ottom) along with a few drops of water, or a moistened owel or cloth.

. Read the temperature and refer to the instrument alibration Table to determine the calibration value. OTE: To achieve the stated accuracy of measurement, e probe must be stabilized before calibrating. The alibration temperature should be within 5 degrees of e sample temperature.

3. Determine the atmospheric correction factor (see Instrument instructions).

4. Multiply the calibration value by the correction factor.

5. Switch your instrument to an appropriate mg/L range and adjust the CALIBRATE control until the meter reads the corrected calibration value from step 4. Without changing the calibration setup, monitor the readings for an additional 3 minutes to verify calibration stability. Readjust if necessary.

WARRANTY AND REPAIR

All YSI products carry a one-year warranty on workmanship and parts, exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge, if possible, when the item is returned to the factory or to an authorized YSI dealer. Electrode cleaning is not covered by warranty.

If you are experiencing difficulty with any YSI product, it may be returned for repair, even if the warranty has expired. YSI maintains complete facilities for prompt servicing on all its products. This warranty is limited to repair or replacement (YSI's option) at no charge.

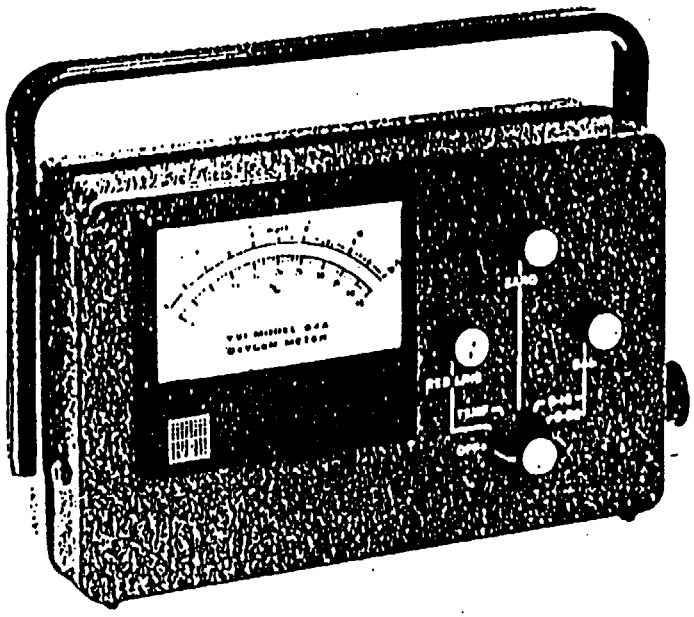
JP0614
Memo to Jill

YSI Inc,
attn: Product Service
Billing address
name P.O. ~~54~~
& ph no. order 45387

YSI Incorporated
Yellow Springs Instrument Co., Inc., Yellow Springs, Ohio 45387 USA • Phone 513 767-7241 • 800 343-HELP • Fax 513 767-9353 • Telex 205437

Item 004483 Part A05730 N November 1988

INSTRUCTION MANUAL
YSI MODELS 54ARC AND 54ABP
DISSOLVED OXYGEN METERS



Scientific Division
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Yellow Springs, Ohio 45387, U.S.A. • Phone 513-767-7241

ITEM 066001 P/N A05457AJ FEBRUARY 1988

PRICE INCLUDING HANDLING $5.00

SUMMARY OF OPERATING INSTRUCTIONS

1. CALIBRATION
   A. Switch instrument to OFF and adjust meter mechanical zero.
   B. Switch to RED LINE and adjust.
   C. Prepare probe for operation, connect to instrument, wait up to 15 minutes for probe to stabilize. Probe can be in calibration chamber or ambient air.
   D. Switch to ZERO and adjust to "0" on mg/l scale.
   E. Switch to TEMP and read on °C scale.
   F. Use probe temperature and true local atmospheric pressure (or feet above sea level) to determine calibration values from Tables I and II. (See pages 14 and 15).
   EXAMPLE: Probe temperature = 21°C; Altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/l. From Table II the altitude factor for 1000 feet is approximately .96. The correct calibration value, then, is:
   8.9 mg/l X .96 factor = 8.54 mg/l
   G. Switch to 0-10 or 0-20 mg/l range and adjust meter with CAL control to calibration value determined in Step F.
   NOTE: It is desirable to calibrate probe in a high humidity environment. (See calibration section for more detail).

2. MEASUREMENT
   A. Place probe in sample and stir.
   B. Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen.
   C. Read dissolved oxygen on appropriate range (1-10 or 0-20 mg/l)
   D. We recommend the instrument be left on between measurements to avoid the necessity to repolarize the probe.

3. GENERAL CARE
   A. Recharge batteries in the YSI Model 54ARC when the instrument can no longer be red lined. Recharge 16-20 hours. Replace with Burgess CD-6 or equivalent. Replace batteries in the YSI Model 54ABP when red line cannot be set with Panasonic UM-2N or equivalent.
   B. Membranes will last indefinitely, depending on usage. Average replacement is 2-4 weeks. Probe should be stored in humid environment to prevent drying out.
   C. Calibrate daily.

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SCHEMATIC DIAGRAM ..... CENTER SPREAD

GENERAL DESCRIPTION

The YSI Models 54ARC and 54ABP Dissolved Oxygen Meters are intended for dissolved oxygen and temperature measurement in water and wastewater applications, but are also suitable for use in certain other liquids. Dissolved Oxygen is indicated in mg/l (milligrams per liter) on 0-10 and 0-20 mg/l scales. Temperature is indicated in °C on a -5° to +45°C scale. Both dissolved oxygen ranges are automatically temperature compensated for solubility of oxygen in water and permeability of the probe membrane.

The probes use Clark-type membrane covered polarographic sensors with built-in thermistors for temperature measurement and compensation. A thin, permeable membrane stretched over the sensor isolates the sensor elements from the environment, but allows oxygen and certain other gases to enter. When a polarizing voltage is applied across the sensor, oxygen that has passed through the membrane reacts at the cathode, causing a current to flow.

The membrane passes oxygen at a rate proportional to the pressure difference across it. Since oxygen is rapidly consumed at the cathode, it can be assumed that the oxygen pressure inside the membrane is zero. Hence, the force causing the oxygen to diffuse through the membrane is proportional to the absolute pressure of oxygen outside the membrane. If the oxygen pressure increases, more oxygen diffuses through the membrane and more current flows through the sensor. A lower pressure results in less current.

Power to operate the system is provided by internal batteries in the instruments, rechargeable batteries in the YSI Model 54ARC and disposable batteries in the YSI Model 54ABP.

SPECIFICATIONS

I. Instrument

Oxygen Measurement

Ranges: 0-10 and 0-20 mg/l (0-5 and 0-10 mg/l with YSI 5776 High Sensitivity Membrane)

Accuracy: ±1% of full scale at calibration temperature (±0.1 mg/l and 0-10 scale).

Readability: .05 mg/l on 0-10 scale; 0.1 mg/l on 0-20 scale.

Temperature Measurement

Ranges: -5° to +45°C

Accuracy: ± 0.7°C, including probe

Readability: 0.25°C

Temperature Compensation

± 1% of D.O. reading for measurements made within ± 5°C of calibration temperature.

± 3% of D.O. reading over entire range of -5 to +45°C Probe temperature.

System Response Time

Typical response for temperature and D.O. readings is 90% in 10 seconds at constant temperature of 30°C with YSI 5775 Membranes. D.O. response at low temperature and low D.O. is typically 90% in 30 seconds. YSI 5776 High Sensitivity Membranes can be used to improve response at low temperatures and low D.O. concentrations. If response time under any operating conditions exceeds two minutes, probe service is indicated.

Operating Temperature Range

Instrument and probe operating range is -2° to +45°C. Large ambient temperature changes will result in 2% loss of accuracy unless Red Line and Zero are reset.

Recorder Output

0 to 114-136 mV. Recorder should have 50,000 ohms minimum input impedance.

Power Supply

YSI Model 54ABP: (4) 1.5 volt carbon zinc batteries provide approximately 1000 hours operation. Replace with Panasonic UM-2N or equal.

YSI Model 54ARC: (4) 1.25 volt Ni-Cad rechargeable cells (Burgess CD-6 or equal) provide approximately 100 hours of operation between charges.

II. Probe

Cathode: Gold
Anode: Silver
Membrane: .001" FEP Teflon (.0005" FEP Teflon available)
Electrolyte: Half Saturated KCl
Temperature Compensation: (See SPECIFICATIONS, I. Instrument)
Pressure Compensation: Effective 1/2% of reading to pressures of 100 psi (230 ft. water)
Polarizing Voltage: 0.8 volts nominal
Probe Current: Air at 30°C = 19 microamps nominal
Nitrogen at 30°C = .15 microamps or less

III. Accessories and Replacement Parts

YSI 5720A — Self Stirring B.O.D. Bottle Probe
YSI 5750 — Non Stirring B.O.D. Bottle Probe
YSI 5739 — Oxygen Temperature Probe for field use. Combine with one of the following cables for desired lead length:
YSI 5401 — Battery Charger Eliminator 115V
YSI 5402 — Battery Charger Eliminator 230V

Detachable leads for use with YSI 5739:

YSI 5740-10	10' cable
YSI 5740-25	25' cable
YSI 5740-50	50' cable
YSI 5740-100	100' cable
YSI 5740-150	150' cable
YSI 5740-200	200' cable

YSI 5492A — Battery Pack Operates YSI 5791A and 5795A Submersible Stirrers

YSI 5791A — Submersible Stirrer for field use

YSI 5795A — Submersible Stirrer for field use

YSI 5075A — Calibration Chamber for use with field probe

YSI 5890 — Carrying Case

YSI 5775 — Membrane and KCl Kit, Standard — includes 2 each 15-membrane packets (.001" thick standard membranes) and a 30 ml bottle KCl with Kodak Photo Flo.

YSI 5776 — Membrane and KCl Kit, High Sensitivity — includes 2 each 15-membrane packets (.0005" thick membranes) and a 30 ml bottle KCl with Kodak Photo Flo.

YSI 5945 — "O" Ring Pack — includes (6) "O" rings for each YSI D.O. Probe.

YSI 5486 — Beater Boot Kit — includes (1) A-05486 Boot, (1) A-05484 Tip, (2) A-05485 Spring. Used only on 5720A and discontinued 5420A and 5720.

YSI 5986 — Diaphragm Kit for use only with YSI 5739 D.O. Probe.

YSI 5734 — Adaptor makes it possible to use discontinued YSI 5400 Series Probes with YSI Models 54ARC and 54ABP.

YSI 5735 — Adaptor makes it possible to use YSI 5739, 5720A and 5750 Probes with discontinued YSI Models 54RC and 54BP.

OXYGEN PROBES AND EQUIPMENT

There are three oxygen probes for use with the YSI Models 54ARC and 54ABP Dissolved Oxygen Meters. Descriptions of where they are used are contained in the following paragraphs.

I. YSI 5739 D.O. Probe

The YSI 5739 probe, with built-in load weight and pressure compensation, is an improved design that replaces the discontinued YSI 5418, 5419, 5718 and 5719 probes. (See Figure 1)

For user convenience the probe is equipped with a disconnecting cable to facilitate changing cable lengths and replacing damaged cables or probes. The probe and cable assembly is held together with a threaded retaining nut. The connection is *not* designed for casual disconnection and should only be disconnected when necessary.

To disconnect the cable unscrew the retaining nut and slide it down the cable to expose the connector. Pull gently on the cable and connector until the connector comes away from the probe body.

To reassemble, inspect the connector and "O" ring for cleanliness. If the "O" ring is frayed or damaged remove it by squeezing it in the groove causing it to bulge, then roll it out of the groove and off the connector. A replacement "O" ring is supplied with the cable.

Push the connector into the probe body, rotating it until the two halves mate. A light coating of vaseline or silicone grease on the "O" ring will make reassembly easier. Air trapped between the connector halves which may cause them to spring apart slightly, is normal. Screw on the retaining nut, *hand tight only*. NOTE: If erratic readings are experienced, disconnect the cable and inspect for water. If present, dry out and reconnect, replacing the "O" ring, if necessary.

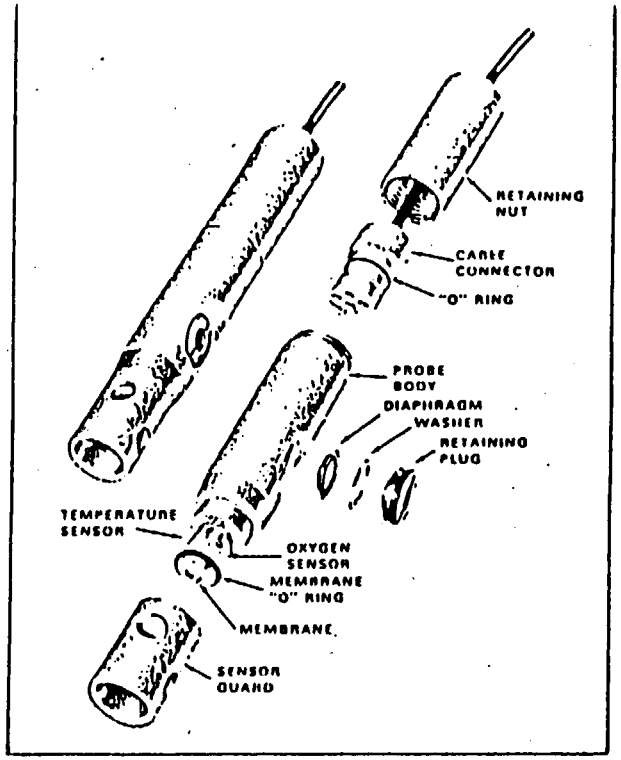


Figure 1

Pressure Compensation

The vent on the side of the probe is part of a unique pressure compensating system that helps assure accurate readings at great depths of water. Pressure compensation is effective to 1/2% of reading with pressures to 100 psi (230 ft. water). The quantity of air bubbles trapped under the membrane determines how serious the pressure error will be, which is why proper preparation of the probe is essential. (See OPERATING PROCEDURES.) The system is designed to accommodate a small amount of trapped air and still function properly, but the amount should be kept to a minimum

The compensating system normally does not require servicing and should not be taken apart. However, if electrolyte is leaking through the diaphragm or if there is an obvious puncture, the diaphragm must be replaced. A spare is supplied with the probe. Using a coin unscrew the retaining plug and remove the washer and the diaphragm, flush any salt crystals from the reservoir, install the new diaphragm (convolution side in), replace the washer, and screw in the retaining plug.

II. YSI 5720A B.O.D. Bottle Probe

The YSI 5720A B.O.D. Bottle Probe replaces the discontinued YSI 5420A B.O.D. Bottle Probe for measuring dissolved oxygen and temperature in standard B.O.D. bottles. It is provided with an agitator for stirring the sample solution, available in models for 117VAC (95-135VAC, 50-60 Hz) or 230VAC (190-250VAC, 50-60 Hz) operation. (See Figure 2)

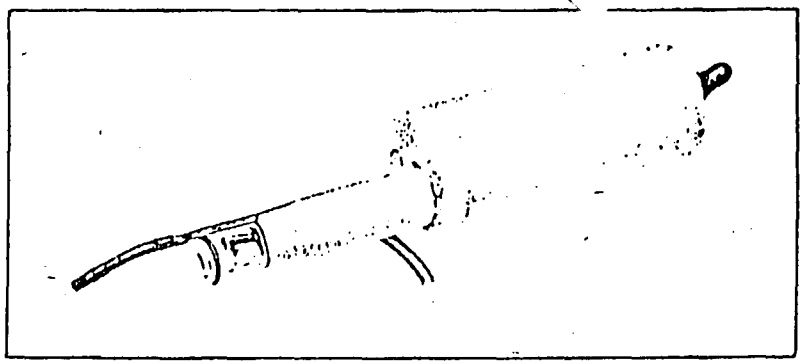


Figure 2

When using the probe, plug the agitator power supply into line power and the probe plug into the instrument. With the agitator turned off place the tapered probe end into the B.O.D. bottle and switch agitator "ON" with switch on top of probe. The probe should be operated with a minimum of trapped air in the B.O.D. bottle. A slight amount of air in the unstirred region at the top of the bottle may be neglected, but no bubbles should be around the thermistor or oxygen sensor.

Stirrer Boot

The probe uses a flexible stirring boot to transmit motion from the sealed motor housing to the sample. If the boot shows signs of cracking or other damage likely to allow leaking into the motor housing, the boot must be replaced.

In fresh water applications boot life is normally several years, but this may be shortened by exposure to hydrocarbons, moderate to strong acids or bases, ozone, or direct sunlight. For maximum life rinse the boot after use in contaminated samples. (See Figure 3)

Boot replacement is as follows:

1. Pull off old assembly and clean shaft.
2. Slide on new assembly making sure the back spring is on the grooved area of the shaft. A small amount of rubber cement may be used.
3. Check that there is sufficient clearance between the tip and the end of the shaft to permit turning without binding.

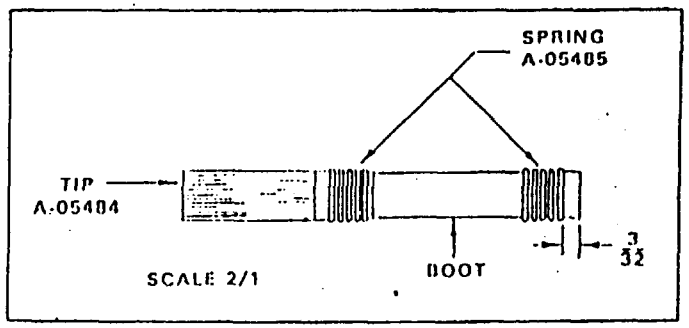


Figure 3

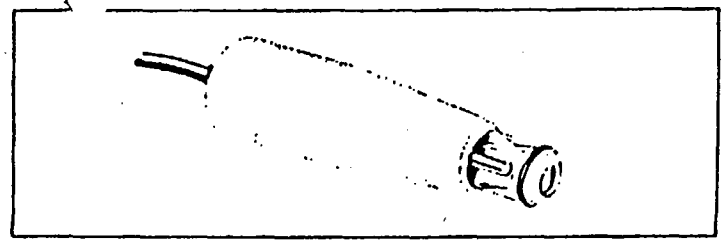


Figure 4

III. YSI 5750 B.O.D. Bottle Probe

The YSI 5750 B.O.D. Bottle Probe replaces the discontinued YSI 5450 B.O.D. Bottle Probe. It is similar to the YSI 5720A B.O.D. Bottle Probe, except that it does not have a stirrer. Agitation of the sample must be provided by other means, such as a magnetic stirrer. (See Figure 4)

IV. Cable Adaptors

All YSI 5700 Series Probes are designed for direct use with the YSI Models 54ARC and 54ABP Dissolved Oxygen Meters. However, to use YSI 5700 probes with the discontinued YSI Models 54RC and 54BP, cable adaptor YSI 5735 is required.

V. YSI 5791A and 5795A Submersible Stirrers

The YSI submersible stirrers are accessories that perform the function of stirring the sample being studied when making dissolved oxygen measurements in the field. The YSI 5791A stirrer can be used with the following dissolved oxygen probes: YSI 5418, 5419, 5718, 5719, and 5739. The YSI 5795A stirrer is only for use with the YSI 5739 Probe. (See Figure 6)

When a stirrer and probe are assembled, the stirrer agitates the sample directly in front of the sensor by means of a rotating eccentric weight which causes the spring-mounted hermetically sealed motor housing to vibrate. An impeller on the end of the motor housing flushes the media across the oxygen sensor. (See sales literature and instruction sheets for further information).

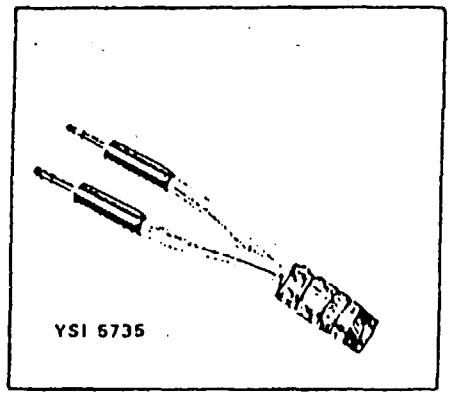


Figure 5

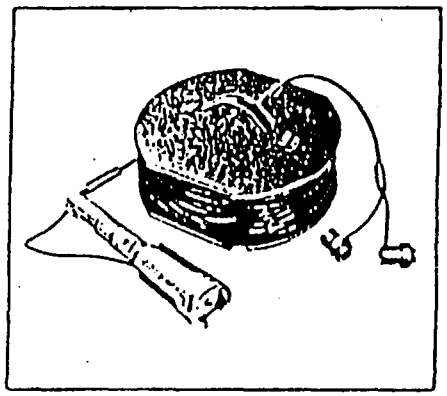


Figure 6

VI. YSI 5492A Battery Pack

The YSI 5492A Battery Pack is designed to attach to the case of all YSI Model 54 Dissolved Oxygen Meters to provide power for operating the submersible stirrers. (See sales literature and instruction sheets for further information).

OPERATING PROCEDURES

1. Preparing the Probe

All YSI 5700 Series Probes have similar sensors and should be cared for in the same manner. They are precision devices relying on good treatment if high accuracy measurements are to be made. Prepare the probes as follows. (See Figure 7)

ALL PROBES ARE SHIPPED DRY — YOU MUST FOLLOW THESE INSTRUCTIONS

1. Prepare the electrolyte by dissolving the KCl crystals in the dropper bottle with distilled water. Fill the bottle to the top.
2. Unscrew the sensor guard from the probe (YSI 5739 only) and then remove the "O" ring and membrane. Thoroughly rinse the sensor with KCl solution.
3. Fill the probe with electrolyte as follows:
   A. Grasp the probe in your left hand. When preparing the YSI 5739 probe the pressure compensating vent should be to the right. Successively fill the sensor body with electrolyte while pumping the diaphragm with the eraser end of a pencil or similar soft, blunt tool. Continue filling and pumping until no more air bubbles appear. (With practice you can hold the probe and pump with one hand while filling with the other.) When preparing the YSI 5720A and 5750 probes, simply fill the sensor body until no more air bubbles appear.
   B. Secure a membrane under your left thumb. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, keeping it clean and dust free, touching it only at the ends.
   C. With the thumb and forefinger of your other hand, grasp the free end of the membrane.
   D. Using a continuous motion *stretch* the membrane *UP, OVER,* and *DOWN* the other side of the sensor. Stretching forms the membrane to the contour of the probe. The membrane can be stretched to approximately 1-1/2 times its normal length.
   E. Secure the end of the membrane under the forefinger of the hand holding the probe.
   F. Roll the "O" ring over the end of the probe. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the "O" ring.
   G. Trim off excess membrane with scissors or sharp knife. Check that the stainless steel temperature sensor is not covered by excess membrane.
4. Shake off excess KCl and reinstall the sensor guard.
5. A bottomless plastic bottle is provided with the YSI 5739 probe for convenient storage. Place a small piece of moist towel or sponge in the bottle and insert the probe into the open end. This keeps the electrolyte from drying out. The YSI 5720A and 5750 probes can be stored in a B.O.D. bottle containing about 1" of water.
6. Membranes will last indefinitely, depending on usage. Average replacement is 2-4 weeks. However, should the electrolyte be allowed to evaporate and an excessive amount of bubbles form under the membrane, or the membrane become damaged, thoroughly flush the reservoir with KCl and install a new membrane.
7. Also replace the membrane if erratic readings are observed or calibration is not stable.
8. "Home brew" electrolyte can be prepared by making a saturated solution of reagent grade KCl and distilled water, and then diluting the solution to half strength with distilled water. Adding two drops of Kodak Photo Flo per 100 ml of solution assures good wetting of the sensor, but is not absolutely essential.
9. The gold cathode should always be bright and untarnished. If it is tarnished (which can result from contact with certain gases) or plated with silver (which can result from extended use with a loose or wrinkled membrane), return it to the factory for service. Never use chemicals or any abrasive.
10. H_2S, SO_2, Halogens, Neon, Nitrous Oxide and CO are interfering gases. If you suspect erroneous readings, it may be necessary to determine if these are the cause. These gases have been tested for response.

100% Carbon Monoxide-Less than 1%	100% Helium-none
100% Carbon Dioxide-Around 1%	100% Nitrous Oxide-1/3 O_2 response
100% Hydrogen-Less than 1%	100% Ethylene-none
100% Chlorine-2/3 O_2 response	100% Nitric Oxide-1/3 O_2 response

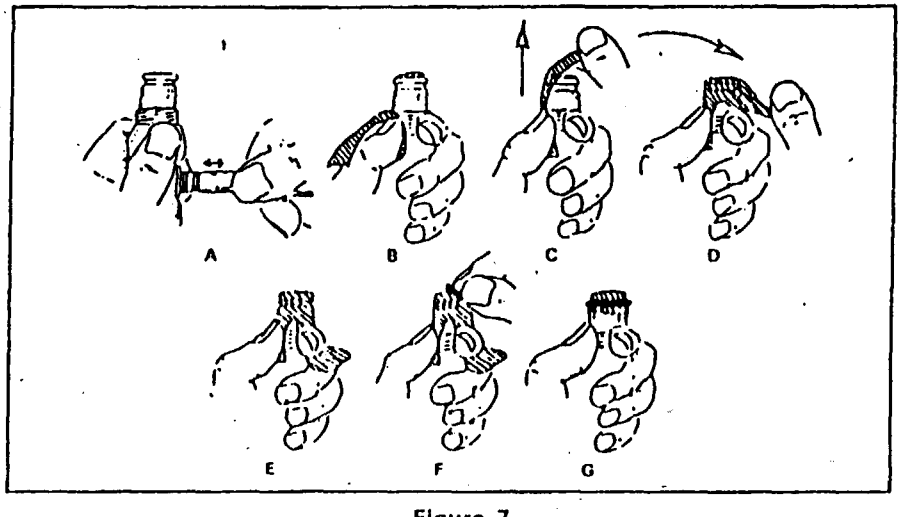


Figure 7

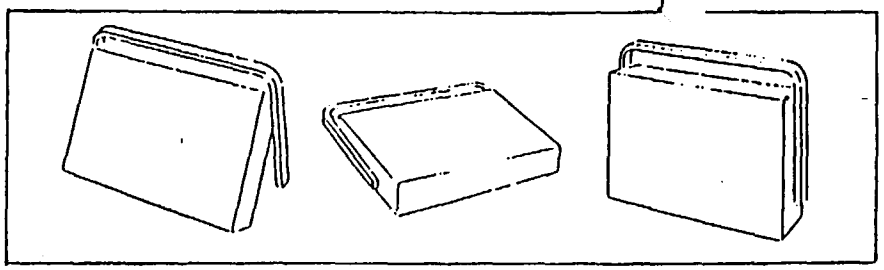


Figure 8

II. Preparing the Instrument

It is important that the instrument be placed in the intended operating position vertical, tilted, or on its back — before it is prepared for use and calibrated. (See Figure 8). Readjustment may be necessary when the instrument operating position is changed. After preparing the probe proceed as follows:

1. With switch in the OFF position, adjust the meter pointer to Zero with the screw in the center of the meter panel. Readjustment may be necessary if the instrument position is changed.
2. Switch to RED LINE and adjust the RED LINE knob until the meter needle aligns with the red mark at the 31°C position.
3. Switch to ZERO and adjust to zero with zero control knob.
4. Attach the prepared probe to the PROBE connector of the instrument and adjust the retaining ring finger tight.
5. Before calibrating allow 15 minutes for optimum probe stabilization. Repolarize whenever the instrument has been OFF or the probe has been disconnected.

III. Calibration

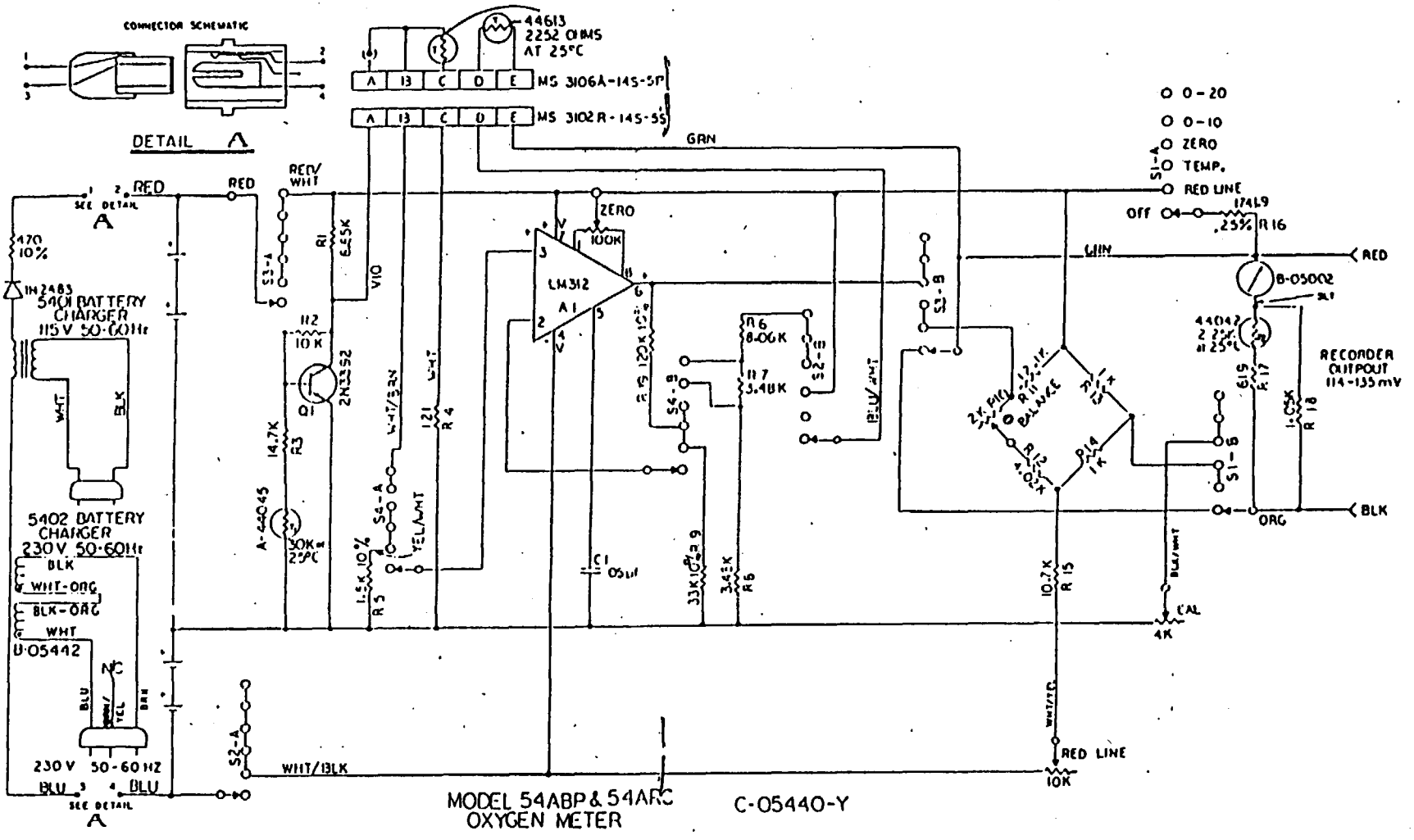
The operator has a choice of three calibration methods — Winkler Titration, Saturated Water, and Air. Experience has shown that air calibration is quite reliable, yet far simpler than the other two methods. The three methods are described in the following paragraphs.

Winkler Titration

1. Draw a volume of water from a common source and carefully divide into four samples. Determine the oxygen in three samples using the Winkler Titration technique and average the three values. If one of the values differs from the other 2 by more than 0.5 mg/l, discard that value and average the remaining two.
2. Place the probe in the fourth sample and stir.
3. Switch to desired mg/l range and adjust the CALIBRATION control to the average value determined in Step 1. Allow the probe to remain in the sample for at least two minutes before setting the calibration value, and leave in the sample for an additional 2 minutes to verify stability. (Readjust if necessary).

Saturated Water

1. Air saturate a volume of water (300-500 cc) by aerating or stirring for at least 15 minutes at a relatively constant temperature.



MODEL 54ABP & 54ARC
OXYGEN METER

C-05440-Y

GENERAL NOTES:

1. ALL RESISTOR VALUES ARE IN OHMS; K=1,000, M=1,000,000. UNLESS OTHERWISE SPECIFIED, RESISTORS ARE 1/4W, 1% METAL FILM.

2. FOR 54ABP BATTERIES ARE 4 EA 1.5V PANASONIC LM-ZM OR EQUIV.
FOR 54ARC BATTERIES ARE 4 EA 1.25V BURGESS CD6 OR EQUIV. NiCad.

3. BATTERY CHARGERS AND CONNECTOR APPLY TO 54ARC VERSION ONLY.

4. THE VALUES SHOWN ON THE SCHEMATIC MAY DIFFER FROM THOSE IN THE INSTRUMENT; IF SO, EITHER VALUE CAN BE USED FOR REPLACEMENT PURPOSES.

2. Place the probe in the s[illegible]e and stir. Switch to TEMPERATURE. Refer to Calibration Table I for the mg/l value corresponding to the temperature.
3. Determine local altitude or the "true" atmospheric pressure (note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level). Using Calibration Table II determine the correction factor for your pressure or altitude.
4. Multiply the mg/l value from Table I by the correction factor from Table II to determine the corrected calibration value for your conditions.

   EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/l. From Table II the correction factor for 1000 feet is about 0.96. The corrected calibration value is 8.9 mg/l X 0.96 = 8.54 mg/l.
5. Switch to an appropriate mg/l range and adjust the CALIBRATE knob while stirring until the meter reads the corrected calibration value from Step 4. Leave the probe in the sample for two minutes to verify calibration stability. Readjust if necessary.

Air Calibration — Fresh Water

1. Place the probe in moist air. B.O.D. probes can be placed in partially filled (50 ml) B.O.D. bottles. Other probes can be placed in the YSI 5075A Calibration Chamber (refer to the following section describing CALIBRATION CHAMBER) or the small calibration bottle (the one with the hole in the bottom) along with a few drops of water. The probe can also be wrapped loosely in a damp cloth taking care the cloth does not touch the membrane. Wait approximately 10 minutes for temperature stabilization. This may be done simultaneously while the probe is stabilizing.
2. Switch to TEMPERATURE and read. Refer to Table I — Solubility of Oxygen in Fresh Water, and determine calibration value.
3. Determine altitude or atmospheric correction factor using Table II.
4. Multiply the calibration value from Table I by the correction factor from Table II.

   EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/l. From Table II the correction factor for 1000 feet is about 0.96. Therefore, the corrected calibration value is 8.9 mg/l X 0.96 = 8.54 mg/l.
5. Switch to the appropriate mg/l range and adjust the CALIBRATE knob until the meter reads the corrected calibration value from Step 4. Wait two minutes to verify calibration stability.

   Readjust if necessary.

Air Calibration — Sea Water

1. Place the probe in moist air. B.O.D. probes can be placed in partially filled (50 ml) B.O.D. bottles. Other probes can be placed in the YSI 5075A Calibration Chamber (refer to the following section describing Calibration Chamber) or the small storage bottle (the one with the hole in the bottom) along with a few drops of water. The probe can also be wrapped loosely in a damp cloth taking care the cloth does not touch the membrane. Wait approximately 10 minutes for temperature stabilization. This may be done simultaneously while the probe is polarizing.

2. Switch to TEMPERATURE and read. Refer to Table III — Solubility of Oxygen in Sea Water, and determine calibration value.
3. Switch to the appropriate mg/l range, and adjust the CALIBRATE knob until the meter reads the calibration value determined in Step 2. Wait 2 minutes to verify calibration stability. Readjust if necessary.

The probe is now calibrated and should hold this calibration value for many measurements. Calibration can be disturbed by physical shock, touching the membrane, or drying out of the electrolyte. Check calibration after each series of measurements and in time you will develop a realistic schedule for recalibration. For best results when not in use, follow the storage procedures recommended for the various probes described under OXYGEN PROBES AND EQUIPMENT. This will reduce drying out and the need to change membranes.

Calibration Chamber

The YSI 5075A Calibration Chamber is an accessory that helps obtain optimum calibration in the field and is also a useful tool for measuring at shallow depths (less than 4').

As shown in Figure (A), it consists of a 4-1/2 foot stainless steel tube (1) attached to the calibration chamber (2), the measuring ring (3), and two stoppers (4) and (5).

For calibration, insert the solid stopper (4) in the bottom of the calibration chamber (2). Push the oxygen probe (6) through the hollow stopper (5) as shown in Figure (B). Place the probe in the measuring ring, Figure (C), and immerse the probe in the sample to be measured for five minutes to thermally equilibrate the probe. Quickly transfer the probe to the calibration chamber (5) draining excess water from the chamber and shaking any excess droplets from

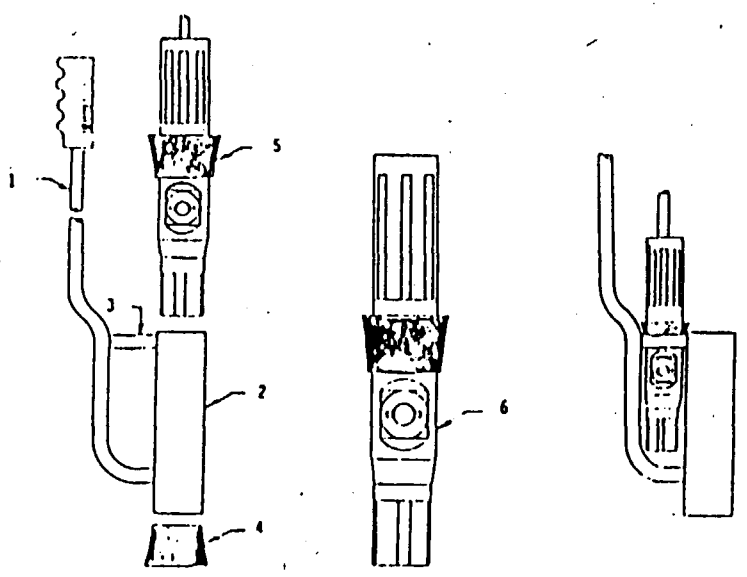


Figure A Figure B Figure C

Figure 9

the probe membrane. For maximum accuracy, wet the inside of the calibration chamber with fresh water. This creates a 100% relative humidity environment for calibration. Place the chamber in the sample for an additional five minutes for final thermal equilibrium. Calibrate the probe as described in the air-calibration procedure. Keep the handle above water at all times.

After calibration, return the probe to the measurement ring for shallow measurements. Move the probe up and down, or horizontally, approximately one foot a second while measuring. In rapidly flowing streams (greater than 5'/second) install the probe in the measuring ring with the pressure compensating diaphragm towards the chamber.

IV. Dissolved Oxygen Measurement

With the instrument prepared for use and the probe calibrated, place the probe in the sample to be measured and provide stirring.

1. Stirring for the YSI 5739 Probe can best be accomplished with a YSI submersible stirrer. If the submersible stirrer is not used, provide manual stirring by raising and lowering the probe about 1 ft. per second. If the 5075 Calibration Chamber is used, the entire chamber may be moved up and down in the water at about 1 ft. per second.
2. The YSI 5720A has a built-in power driven stirrer.
3. With the YSI 5750 sample stirring must be accomplished by other means, such as with the use of a magnetic stirring bar.
4. Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen.
5. Read dissolved oxygen.

V. High Sensitivity Membrane

Use of high sensitivity .0005" membranes (YSI 5776) in place of standard .001" membrane (YSI 5775) is recommended when measurements are to be made consistently at low temperatures (less than 15°C). Calibration and readings will be made just as if the standard YSI 5775 Membrane was being used.

The YSI 5776 High Sensitivity Membranes can also be used in certain situations to increase sensitivity at temperatures about 15°C. The ranges thus become 0-5 and 0-10 mg/l. When calibration with high sensitivity membranes is attempted at temperatures greater than 15°C the selector switch must be set to 0-20 mg/l. Multiply the calculated calibration value by 2. For example: at 21°C and 1000 ft. altitude the calibration value would be 8.6 x 2 or 17.2. Remember the 0-10 and 0-20 mg/l ranges are now 0-5 and 0-10 mg/l, and all mg/l readings must be divided by 2 for a final reading. When operating in this manner accuracy will be degraded slightly.

VI. Recorder Output

Red and black recorder jacks are provided on the YSI Models 54ARC and 54ABP, if you wish to record data while measuring. The high terminal of the recorder is connected to the red tip jack and the low terminal to the black. Output of the YSI 54A at full scale is between 114 to 136 mV.

Use a 50K or higher input impedance recorder and operate it with the terminals ungrounded. The recorder should be operated with its terminals ungrounded. Calibration of the instrument should be checked after connection of the recorder.

Many recorders have an adjustable full scale sensitivity feature. When these recorders are used with the Model 54A, use the 100 millivolt range and adjust the full scale chart deflection when there is full scale meter deflection. Refer to the instruction book for the recorder. For recorders without this feature, a simple divider network as shown below can be constructed. This is adequate to adjust the signal for full scale chart and meter deflection on the 100 mV fixed range recorders.

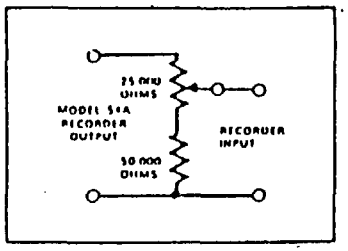


Figure 10

VIII. Calibration Tables

Table I shows the amount of oxygen in mg/l that is dissolved in air saturated fresh water at sea level (760 mmHg atmospheric pressure) as temperature varies from 0° to 45°C.

Table I — Solubility of Oxygen in Fresh Water

Temperature °C	mg/l Dissolved Oxygen	Temperature °C	mg/l Dissolved Oxygen
0	14.60	23	8.56
1	14.19	24	8.40
2	13.81	25	8.24
3	13.44	26	8.09
4	13.09	27	7.95
5	12.75	28	7.81
6	12.43	29	7.67
7	12.12	30	7.54
8	11.83	31	7.41
9	11.55	32	7.28
10	11.27	33	7.16
11	11.01	34	7.05
12	10.76	35	6.93
13	10.52	36	6.82
14	10.29	37	6.71
15	10.07	38	6.61
16	9.85	39	6.51
17	9.65	40	6.41
18	9.45	41	6.31
19	9.26	42	6.22
20	9.07	43	6.13
21	8.90	44	6.04
22	8.72	45	5.95

Source: Derived from 15th Edition "Standard Methods for the Examination of Water and Wastewater."

Table II — Correction for Atmospheric Pressure

Table II shows the correction factor that should be used to correct the calibration value for the effects of atmospheric pressure or altitude. Find true atmospheric pressure in the left hand column and read across to the right hand column to determine the correction factor. (Note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level.) If atmospheric pressure is unknown, the local altitude may be substituted. Select the altitude in the center column and read across to the right hand column for the correction factor.

Table II

Atmospheric Pressure mmHg	or	Equivalent Altitude Ft.	=	Correction Factor
775		540		1.02
760		0		1.00
745		542		.98
730		1094		.96
714		1688		.94
699		2274		.92
684		2864		.90
669		3466		.88
654		4082		.86
638		4756		.84
623		5403		.82
608		6065		.80
593		6744		.78
578		7440		.76
562		8204		.74
547		8939		.72
532		9694		.70
517		10472		.68
502		11273		.66

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater."

The temperature-solubility relationship of oxygen in sea water is not the same as that in fresh water. For this reason the compensation error when used with sea water is greater than when used with fresh water. For a ±5°C span the error could be +2.2% of reading and over the temperature range of -2° to +30°C the error could be 6.3% of reading.

Table III — Solubility of Oxygen in Sea Water

SOLUBILITY OF OXYGEN IN SEA WATER
(Chloride concentration 20,000 mg/1)

Temp. °C	Solubility mg/l	Temp. °C	Solubility mg/l
0	11.41	16	7.91
1	11.11	17	7.78
2	10.83	18	7.61
3	10.56	19	7.47
4	10.30	20	7.33
5	10.05	21	7.20
6	9.82	22	7.07
7	9.59	23	6.95
8	9.37	24	6.83
9	9.16	25	6.71
10	8.96	26	6.60
11	8.77	27	6.49
12	8.58	28	6.38
13	8.41	29	6.28
14	8.24	30	6.18
15	8.07		

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater."

Correcting for Salinity

When measuring dissolved oxygen in water samples with a salinity or chlorinity between sea water and fresh water, calibrate the instrument for fresh water and make your measurements. Then correct the data according to the following formula:

FORMULA:

$$A = M\left[1.0 - \left(\frac{(Cs/Co\,[Sf - So])}{Sf}\right)\right]$$

Where: A = Actual DO of sample. (mg/l dissolved O_2)
M = Measured DO with instrument
*Co = Chlorinity of ocean water (20 o/oo Cl ion)
*Cs = Chlorinity of sample (o/oo Cl ion)
Sf = DO of saturated fresh water at 760 mm pressure and at same temperature as sample (mg/l DO, obtain data from charts in instruction manual)
So = DO of saturated ocean water (20,000 mg/l Chloride ion) at 760 mm pressure and at same temperature as sample (mg/l DO, obtain data from instruction manual)

*NOTE: If salinity is used instead of chlorinity the ratio Cs/Co is computed using 36.11 o/oo for Co (salinity of ocean water), and the salinity of your sample of Cs.

EXAMPLE: Measured Data
DO = 4.1
Temp = 22°C
Salinity = 31 o/oo salinity
M = 4.1 mg/l DO from data
Co = 36.11 o/oo salinity from manual
Cs = 31.0 o/oo salinity from data
Sf = 8.8 mg/l DO from Table I in manual
So = 7.1 mg/l DO from Table II in manual

$$A = 4.1\left[1.0 - \left(\frac{([31.0/36.11]\ [8.8 - 7.1])}{8.8}\right)\right]$$

$$= 4.1\left[1.0 - \left(\frac{([.86]\ [1.7])}{8.8}\right)\right]$$

$$= \frac{(1.46)}{(8.8)}$$

$$= 4.1\left[1.0 - (8.8)\right]$$

$$= 4.1\left[1.0 - 0.166\right]$$

$$= 4.1\left[0.834\right]$$

$$= 3.41 \text{ mg/l}$$

DISCUSSION OF MEASUREMENT ERRORS

There are three basic types of errors which can occur. Type I errors are related to limitations of the instrument design and tolerances of the instrument components. These are chiefly the meter linearity and resistor tolerances. Type II errors are due to basic probe accuracy tolerances, chiefly background signal, probe linearity, and variations in membrane temperature coefficient. Type III errors are related to the operator's ability to determine the conditions at the time of calibration. If calibration is performed against more accurately known conditions, Type III errors are appropriately reduced.

Individual Sources of Error

This description of sources of error can be used to attach a confidence to any particular reading of dissolved oxygen. The particular example given is for a near extreme set of conditions. As a generality, overall error is diminished when the probe and instrument are calibrated under conditions of temperature and dissolved oxygen which closely match the sample temperature and dissolved oxygen.

Type I

A — is the error due to meter linearity
Error = +1% full scale of the measurement range.

B — is the error due to tolerances in the instrument when transferring a reading from one range to another. Error = ±1% of the reading.

Type II

A — errors due to probe background current

$$\text{Error} = 1.0\%\left(1 - \left(\frac{\text{Meter Reading mg/l}}{\text{Calibration Value mg/l}}\right)\right) \times \text{Calib. Value, mg/l}$$

B — errors due to probe non-linearity. Error = ± 0.3% of reading.

C — error caused by variability in the probe membrane temperature coefficient.
Error = zero if readings are taken at the calibration temperature
Error = ±1% of meter reading if readings are taken within 5°C of the calibration temperature.
Error = ±3% of meter reading for all other conditions.

Type III

A — errors due to the accuracy of the instrument thermometer when used to measure the exact probe temperature during calibration.
Error = ±1.5% of reading.

B — errors due to the assumption of mean barometric pressure. Daily variation is usually less than 1.7%.
Error = ±1.7% of reading.

C — errors assume an ability to estimate altitude to within +500 ft. when computing the altitude correction factor.
Error = ± 1.8% of reading.

D — errors consider the possibility of only 50% relative humidity when calibrating the probe. If the actual relative humidity is 50% instead of 100% the errors will be as follows:

Calibration Temperature ± C	Error in percent of reading
0	(-) 0.3
10	(-) 0.6
20	(-) 1.15
30	(-) 2.11
40	(-) 3.60

Example of a Typical Error Calculation

The example given presumes the air calibration technique. If calibration is done with air saturated water, the relative humidity consideration (III-D) is eliminated. If the Winkler calibration method is used, Type III errors are deleted and replaced by the uncertainty attributable to the overall Winkler determination.

Data: Instrument calibrated at 25°C, elevation estimated at 2000' ± 500', normal barometric pressure assumed, calibrated on 0-10 mg/l range at 7.8 mg/l, readings taken on 0-20 mg/l range at 10.5 mg/l at 8°C.

Type	Description		Calculations		Error mg/l
IA	Linearity	=	.01 X 10.5 mg/l	=	.10
IB	Range Change	=	.01 X 10.5 mg/l	=	.10
IIA	Probe Background	=	$.01 \times \left(1 - \frac{10.5}{7.8}\right) 7.8$ mg/l	=	.03
IIB	Probe Linearity	=	.003 X 10.5 mg/l	=	.03
IIC	Temp. Compensation	=	.03 X 10.5 mg/l	=	.31
IIIA	Temp. Measurement	=	.015 X 10.5 mg/l	=	.16
IIIB	Pressure	=	.017 X 10.5 mg/l	=	.18
IIIC	Altitude	=	.18 X 10.5 mg/l	=	.19
IIID	R.H.	=	.016 X 10.5 mg/l	=	.17
			Maximum Possible Error	=	1.27 mg/l
			Probable Error	=	± .63 mg/l

Considering a statistical treatment of the probable error at any time for any instrument, it is likely that the actual error in any measurement will be about 1/2 of the possible error. In this case the probable error is about ±.5 mg/l out of a reading of 10.5 mg/l, or 4.8% of the reading.

INSTRUMENT BATTERIES

Battery replacement or recharging on the YSI Model 54A is indicated if the "red line" adjustment cannot be made or O_2 calibration cannot be achieved. (Warning: a faulty probe will also not permit O_2 calibration.)

To replace batteries remove the four screws holding the rear cover of the instrument. The four batteries will be found on the battery terminal board inside. CAUTION: disconnect battery charger on YSI Model 54ARC before removing cover.

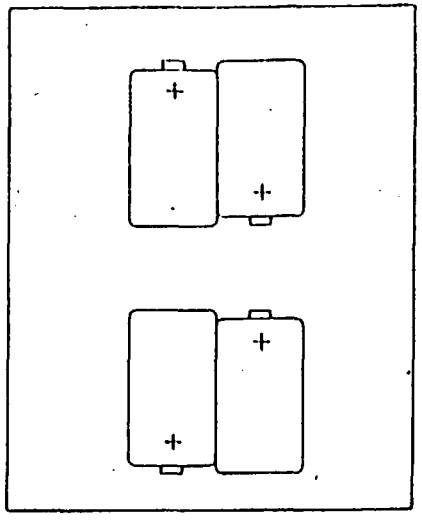


Figure 11

The YSI Model 54ARC contains four 1.25V Ni-Cd batteries (Burgess CD6 or equivalent). (See Figure 11). These batteries should be recharged when the instrument can no longer be red lined. Battery life should be three years or longer. Deeper discharge because of longer intervals between recharge will result in shorter battery life. The batteries should be recharged overnight, about 16 hours with the instrument off or 20 hours with the YSI Model 54ARC turned on.

The YSI Model 54ABP contains four 1.5V carbon-zinc (Panasonic UM-2N or equivalent). The life of these batteries is 1000 hours. Replace batteries every six months to minimize danger of corrosion due to dead or leaky batteries.

Battery holders are color coded. Positive (+ button) end of battery must go to red. (See Figure 11).

WARRANTY AND REPAIR

All YSI products carry a one-year warranty on workmanship and parts exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge, if possible, when the item is returned to the factory or to an authorized YSI dealer.

If you are experiencing difficulty with any YSI product, it may be returned for repair, even if the warranty has expired. YSI maintains complete facilities for prompt servicing for all YSI products.

YELLOW SPRINGS INSTRUMENT CO., INC.
SERVICE DEPARTMENT
P.O. BOX 279
YELLOW SPRINGS, OHIO 45387, U.S.A.

PHONE: 513-767-7241

APPENDIX C-5

REDOX POTENTIAL

FIELD MEASUREMENT OF OXIDATION-REDUCTION POTENTIAL

Method: Electrometric

Reference: Beckman Instruments, 1987

Sensitivity: 1 mV

Optimum Range: -999.9 mV to +999.9 mV

Sample Handling: Determine on-site or within 4 hours

Reagents and Apparatus:

1. pH meter in absolute millivolt mode,
2. Platinum combination electrodes,
3. Beakers or plastic cups,
4. Certified pH buffer solutions, pH 4 and 7 saturated with a few crystals of quinhydrone,
5. Deionized water in squirt bottle.
6. All glassware soap and water washed, followed by two hot water rinses and two deionized water rinses.

Calibration:

1. Short the meter glass and reference inputs, and adjust the STANDARDIZE control until zero millivolts is displayed.
2. Place electrode in pH 4 buffer solution saturated with quinhydrone.

3. Record mV reading and compare to chart on Table 1.

4. Rinse electrode with deionized water and place in pH 7 buffer solution saturated with quinhydrone.

5. Record mV reading and compare to chart on Table 1.

6. If mV readings do not agree within ± 10 mV of the Table 1 values at the given temperature, follow electrode maintenance procedures described in the attached manual and recalibrate.

Procedure:

1. Calibrate meter using calibration procedure.

2. Pour the sample into a cleaner beaker or plastic cup.

3. Immerse electrode in solution allowing several minutes for meter to stabilize. Make sure the white AgCl junction on side of electrode is in the solution. The level of electrode solution must be approximately one inch above sample to be measured.

4. Rinse electrode with deionized water between samples. Recheck calibration with pH 4 buffer solution saturated with quinhydrone after every 5 samples.

Notes:

1. Eh is temperature and pH dependent. Therefore, the temperature and pH of samples should be measured at the same time as redox. For refrigerated or cool samples, use refrigerated buffers to calibrate meter.

2. Weak organic and inorganic salts and oil and grease are interferences in Eh measurements. If oil and grease are visible, note on data sheet. Clean electrode with soap and water, polish with scouring powder and rinse with distilled water. Then recalibrate meter.

3. Before going into the field:

   a. Report any problems;
   b. Do a quick calibration with quinhydrone saturated pH buffer solution to check electrode;
   c. Prepare fresh quinhydrone saturated pH 4 and pH 7 solutions daily.

4. Following field measurements:

   a. Report any problems;
   b. Compare with previous data;
   c. Clean all dirt off of meter and inside case;
   d. Store electrode as follows;

      1) Slide rubber sleeve into position over the filling hole.
      2) Place cot over tip of electrode by threading platinum wire through opening and sliding cot onto glass body until porous plug is completely covered.

JDD/jkk/CAW
[wpmisc-600-39]
60776.05

Table 1

Redox Potential Calibration Chart

Quinhydrone Saturated pH 4 Solution

Temperature °C	20°	25°	30°
Theoretical Value (mV)	+268 mV	+263 mV	+258 mV

Quinhydrone Saturated pH 7 Solution

Temperature °C	20°	25°	30°
Theoretical Value (mV)	+92 mV	+86 mV	+79 mV

Instrument reading should be within ± 10 mV of Theoretical

[wpmisc-400-12]

BECKMAN

Φ™10 pH Meter
Φ™11 pH Meter
Φ™12 pH/ISE Meter

ASSC.

WARRANTY

Your Φ™ (pH™) 10, 11, or 12 pH Meter is warranted to be free of manufacturing defects for one (1) year from the date of purchase. This does not include any defects that are the result of abuse or misuse of the instrument. Beckman Instruments, Inc., will, at Beckman's option, repair or replace your instrument with a comparable unit. This is a limited warranty. You may have additional rights under your state laws. Batteries are not included in this warranty.

WARNING: *This equipment generates, uses, and can radiate radio frequency energy and may cause interference to radio communications. Improper installation or modification of the equipment may increase interference. It has been tested and found to comply with the limits for a Class A computing device pursuant to Subpart J of Part 15 of FCC Rules, which are designed to provide reasonable protection against such interference when operated in a commercial environment.*

Operation of this equipment in a residential area may cause interference, in which case the user at his own expense will be required to take whatever measures may be required to correct the interference.

Beckman Instructions 015-246800-B

BECKMAN

Φ™10 pH Meter
Φ™11 pH Meter
Φ™12 pH/ISE Meter

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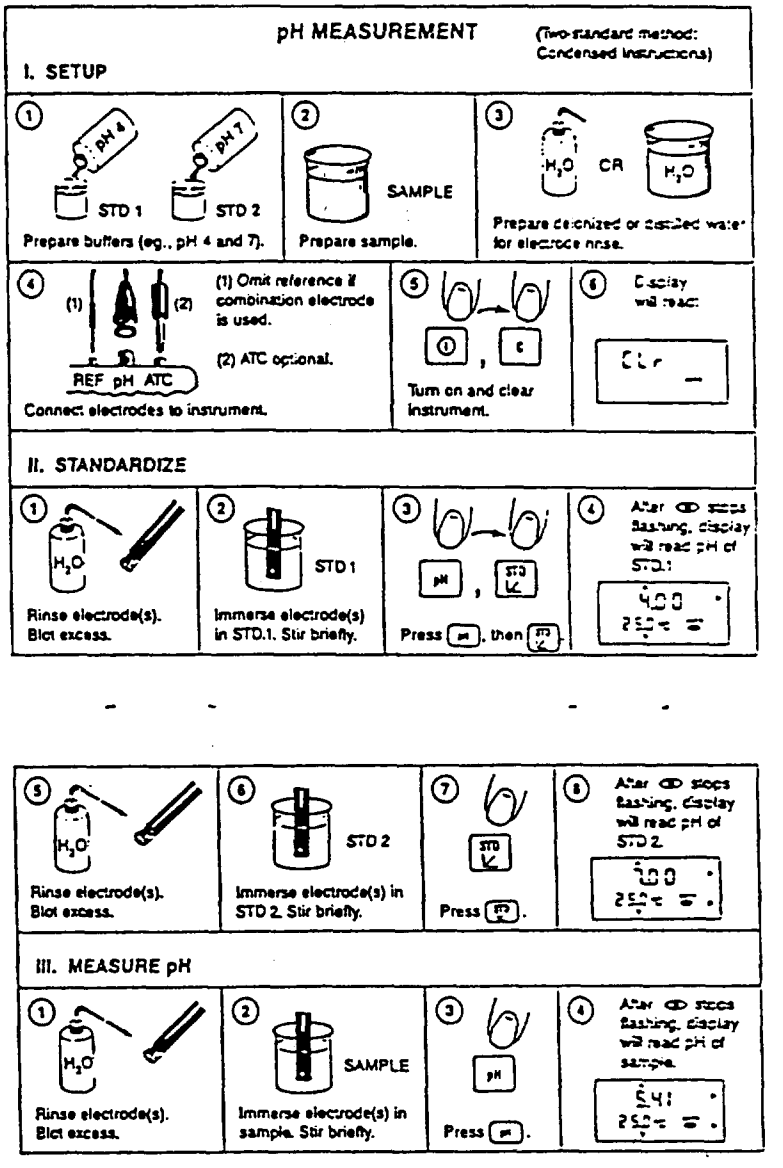


FOR MORE DETAILED INSTRUCTIONS ON pH MEASUREMENT, SEE NEXT PAGE.

MEASURING mV AND RELATIVE mV (Φ11, Φ12)

MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

SPECIFICATIONS

pH MEASUREMENT: DETAILED INSTRUCTIONS

METHODS: The pHI 10, 11, and 12 can measure pH from 0 to 15.99. They will perform one- or two-point standardization automatically, using any buffer listed below, at any temperature between −5°C and 100°C.

STANDARD pH BUFFERS RECOGNIZED BY THE pHI 10, 11, AND 12:

1.68, 4.00, 7.00, 10.01, 12.45.

TWO-POINT STANDARDIZATION METHOD:

Two-point standardization, the preferred and more accurate method of pH measurement, should be used when pH accuracy of beyond ±0.1 pH is required. Use buffers as close to the sample pH as possible; one above, and one below. (For example, if sample pH is about 8.5, use 7.00 and 10.01 pH buffers).

ONE-POINT STANDARDIZATION METHOD:

One-point standardization, a somewhat faster procedure, is recommended only if (a), accuracy of ±0.1 pH unit is acceptable, and (b), sample pH is within 1.5 pH of that of the buffer used for standardization.

pH MEASUREMENT PROCEDURE:

1. Connect electrode(s) to appropriate input(s):
   a. If a *combination electrode* is used, connect it to the input marked "pH".
   b. If an *electrode pair* is used, connect the indicating electrode to the input marked "pH" and the reference electrode to the input marked "REF".
   c. For better accuracy, or when measuring and/or standardizing at a temperature of other than 25°C, connect a Beckman 598115 Automatic Temperature Compensator probe to input marked "ATC".

2. Press [⏻] to turn on instrument, then press [C] to clear. Display will show [Cx AUTO].

3. Rinse electrode(s) (and ATC if used) with deionized water. Blot excess.

4. Immerse electrode(s) (and ATC if used) in first standard. Stir briefly with electrodes to remove bubbles from electrode surfaces. Press [pH]. Displayed pH value will have a resolution of 0.01. If 0.1 resolution is desired, press [1/pH].

5. Press [STD/CAL]. When [⏲] stops flashing, display will show [pH value locked, ⏲, ▶ STD].

6. Rinse electrode(s) (and ATC probe if used) with deionized water. Blot excess. Proceed to appropriate step, according to desired type of standardization:
   a. If ONE-POINT standardization is to be used, instrument is ready for sample measurement; proceed to Step 9.
   b. If TWO-POINT standardization is desired, proceed to Step 7.

7. Immerse electrode(s) (and ATC if used) in second standard. Stir briefly with electrodes to remove bubbles from electrode surfaces. Press [STD/CAL]. When [⏲] stops flashing, display will show [pH value locked, ⏲, ▶ STD, ▶ STD].

8. Rinse electrode(s), (and ATC probe if used) with deionized water. Blot excess.

9. Immerse electrode(s) (and ATC if used) in sample. Stir briefly with electrodes. Press [pH]. When [⏲] stops flashing, display will show [pH value locked, ⏲]. Measurement is now complete. Repeat Steps 8 and 9, above, for additional samples.

10. If *continuous* pH monitoring is desired, press [AUTO] to turn off Auto Read function.

MEASURING mV AND RELATIVE mV (Φ11, Φ12)

MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

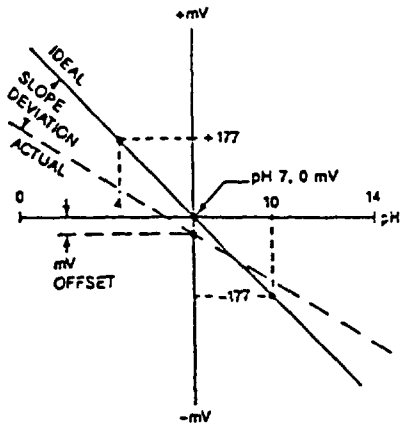
BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

SPECIFICATIONS

pH MEASUREMENT: PRINCIPLES AND THEORY

The pHI 10/11/12 pH Meter is essentially a high-impedance voltmeter with a microcomputer that translates voltage and temperature data into pH units. At 25°C, the ideal pH electrode system develops −59 mV per pH unit increase, with 7.00 pH = 0 mV.

Standardization allows the meter to compensate for non-ideal electrode characteristics. One-point standardization compensates for millivolt offset; two-point standardization compensates for both millivolt offset and slope deviation. See diagram below.



Ideal and Actual Electrode Response Compared

The pHI 10, 11, and 12 may be standardized with any of five standard pH buffers: 1.68, 4.00, 7.00, 10.01, and 12.45. Standardization may be accomplished with any two buffers, used in any order (and at any temperature, if ATC is used). When [pH] is pressed, the instrument automatically recognizes the buffer.

The relationship between pH and electrode voltage changes with temperature. For precise pH measurements or temperatures not close to 25°C, a Beckman 598115 ATC (Automatic Temperature Compensator) probe should be used. With this probe, the instrument automatically compensates for the temperature characteristics of the buffer, permitting a sample to be measured at any temperature, even if different from the buffer temperatures. With ATC, the instrument measures and displays temperatures from −5°C to 100°C.

If an ATC probe is *not* used, the instrument defaults and displays 25°C.

The pH calculation is based on the Nernst equation:

$$E = E_0 - \frac{2.3\,RT}{nF} \log a_i$$

E is the total potential, in millivolts, developed between the sensing and reference electrodes; E_0 varies with the choice of electrodes, temperature, and pressure; $2.3RT/nF$ is the Nernst factor (R and F are constants, n is the charge on the ion, including sign, T is the temperature in degrees Kelvin), and a_i is the activity of the ion to which the electrode is responding.

For further information on principles and theory of pH measurement, refer to The Beckman Handbook of Applied Electrochemistry (Beckman Bulletin 7739).

MEASURING mV AND RELATIVE mV (Φ11, Φ12)

MEASURING CONCENTRATION (Φ12)

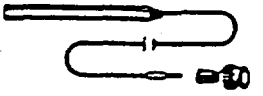
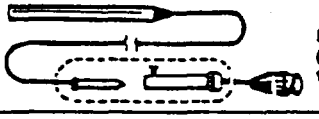
INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

SPECIFICATIONS

FOR ACCURATE mV MEASUREMENTS WITH THE Φ11 OR Φ12, THE FOLLOWING ITEMS ARE RECOMMENDED:

1.		pH indicating electrode, Futura II. Futura II cable with BNC connector. NOTE: If combination pH electrode is used, omit separate reference electrode (item 2, below).
OR		
1a.		Metallic electrode with 2 mm pin connector + pin-to-BNC adaptor.
OR		
1b.		Ion-Selective electrode with BNC connector (or U.S. standard connector + U.S. standard-to-BNC adaptor).
2.		Reference electrode, Futura II. Cable with 2 mm pin connector, Futura II. NOTE: Omit reference electrode if combination pH electrode is used.
3.		Standard solution(s) appropriate to the application.
4.		Clean beaker(s) or equivalent container(s), 100–250 mL, for containing standard solution(s).
5.	OR	Squirt bottle or beaker containing deionized or distilled water for rinsing electrodes.
6.		Clean towels, "Kimwipes™", etc., for blotting electrodes.
7.	SAMPLE	The sample to be measured.

For part numbers, see "Electrodes, Buffers, and Accessories."
For mV measurement procedures, see next page.

MEASURING mV AND RELATIVE mV (Φ11, Φ12)

MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

SPECIFICATIONS

mV MEASUREMENT: CONDENSED INSTRUCTIONS

I. SETUP

① STANDARD Prepare standard solution.	② SAMPLE Prepare sample.	③ H_2O OR H_2O Prepare deionized or distilled water for electrode rinse.
④ REF pH ATC Connect electrodes to instrument.	⑤ ⓘ , ⓒ Turn on and clear instrument.	⑥ Display will read:

II. mV MEASUREMENT, ABSOLUTE. FOR RELATIVE mV MEASUREMENT, SEE III, BELOW.

① H_2O Rinse electrode(s). Blot excess H_2O.	② STANDARD OR SAMPLE Immerse electrode(s) in standard or sample. Stir briefly.	③ mV Press mV.	④ After stops flashing, display will read absolute mV of solution. mV

For RELATIVE mV Measurement, Proceed with following steps:

III. mV MEASUREMENT, RELATIVE

① H_2O Rinse Electrodes. Blot excess.	② STANDARD Immerse electrodes in standard solution to be used to establish zero mV point. Stir briefly.	③ mV , STD Press mV then STD.	④ After stops flashing, display will read 000.0 mV. REL mV
⑤ H_2O Rinse electrodes. Blot excess.	⑥ SAMPLE Immerse electrodes in sample. Stir briefly.	⑦ mV Press mV.	⑧ After stops flashing, display will read mV relative to the standard. REL mV

NOTE
INSTRUMENT WILL REMAIN IN RELATIVE mV MODE UNTIL EITHER pH, CONC, OR ⓒ IS PRESSED.

FOR MORE DETAILED INSTRUCTIONS ON mV MEASUREMENT, PROCEED TO NEXT PAGE.

MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

mV MEASUREMENT: DETAILED INSTRUCTIONS

mV MEASUREMENT: TYPICAL USES

Some uses of the mV mode are monitoring chemical reactions, quantifying ions, and determining the oxidizing-reducing potential (ORP) of a given sample. Because such measurements are usually not specific for a particular ion or species, readings must be interpreted carefully to obtain meaningful results. The user should have an understanding of the reaction that is occurring, or is desired, and of any sample components that could potentially interfere. For more detailed information, refer to the Beckman Handbook of Applied Electrochemistry (Beckman Bulletin 7739).

The mV mode may also be used with ion-selective electrodes. The relative mV mode can be used in the standard addition or standard subtraction method of ion analysis.

STANDARD SOLUTION(S)

Make up appropriate standard solution(s) to provide known voltage(s), depending on the reference electrode used and the temperature. For example, common standards used in redox measurements are pH 4 and pH 7 buffers saturated with quinhydrone.

mV MEASUREMENT PROCEDURE

1. Connect electrodes to appropriate inputs:
   a. Connect indicating electrode to input marked "pH". A Pin-to-BNC Adaptor may be required as most metallic electrodes have a pin connector.
   b. Connect reference electrode to input marked "REF".
2. Press [⏻] to turn on instrument, then press [E] to clear. Display will show [Clr, AUTO].
3. Rinse electrodes with deionized water. Blot excess.
4. Immerse electrodes in desired solution. Press [mV]. Displayed value is *absolute mV*, as indicated by display of [mV]. When [⊙] stops flashing, display will show [mV reading locked, ⊙].

RELATIVE mV MEASUREMENT PROCEDURE

1. Perform Steps 1 through 3 of mV MEASUREMENT PROCEDURE, above.
2. Immerse electrodes in standard solution to be used to establish the zero mV point. Press [mV], then [pH/V]. When [⊙] stops flashing, display will read [000.0 mV]. Note that, in mV mode, pressing [pH/V] causes the instrument to establish the zero mV point at the value of the current reading. If desired, this step may be repeated at any time to re-establish the zero mV point.
3. Rinse electrodes with deionized water. Blot excess.
4. Immerse electrodes in sample. Press [mV]. Displayed value is *relative mV*, as indicated by display of [REL mV]. When [⊙] stops flashing, display will show [sample relative mV value locked, ⊙]. Absolute mV reading of the standard solution is *automatically* subtracted from the absolute mV reading of the sample, resulting in a relative mV reading for the sample.
5. If *continuous* readout of relative mV is desired, press [AUTO] to turn off Auto Read function.

NOTE

VOLTAGE DIFFERENCE BETWEEN STANDARD SOLUTION AND SAMPLE MUST NOT EXCEED 1000 mV. MAXIMUM DISPLAY RANGE IN mV MODE IS ±999.9 mV.

NOTE

IN mV MODE, THE 598115 AUTOMATIC TEMPERATURE COMPENSATOR PROBE MAY BE USED FOR TEMPERATURE *MEASUREMENT* AND *DISPLAY*, BUT DOES *NOT* HAVE ANY *TEMPERATURE-COMPENSATING* EFFECT.

MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

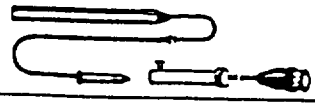
BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

FOR ACCURATE CONCENTRATION MEASUREMENTS WITH THE Φ12, THE FOLLOWING ITEMS ARE RECOMMENDED:

1.

   Ion-Selective electrode with BNC connector.

   OR

1a. 

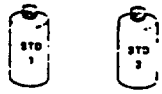
   Ion-Selective electrode with U.S. standard connector + U.S. standard-to-BNC adapter.

2.

   Reference electrode, Futura II.

   Futura II cable with 2 mm pin connector.

   NOTE: Depending on the application, a salt-bridge or double-junction electrode may be required.

3. 

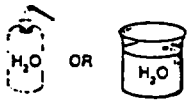
   Two standard solutions of appropriate concentration, selected from the following values: 1.00, 2.50, 5.00, 10.00, 25.0, 50.0, 100.0, 250.0, 500, and 1000 units. Concentration can be expressed in any desired units such as ppm, mM, mg/L, and ozl/gal.

   Make up these solutions per procedure or by diluting a stock solution to suit your requirement.

NOTE
CONCENTRATION UNITS FOR BOTH STANDARD SOLUTIONS MUST BE THE SAME AS DESIRED FOR SAMPLE READINGS.

4.

   Two clean beakers or equivalent containers, approximately 100–250 mL, for containing the two standard solutions.

5. 

   Squirt bottle or beaker containing deionized or distilled water for rinsing electrodes.

6.

   Clean towels, "Kimwipes™", etc., for blotting electrodes.

7.

   The sample to be measured.

For part numbers, see "Electrodes, Buffers, and Accessories."
For concentration measurement procedure, see next page.

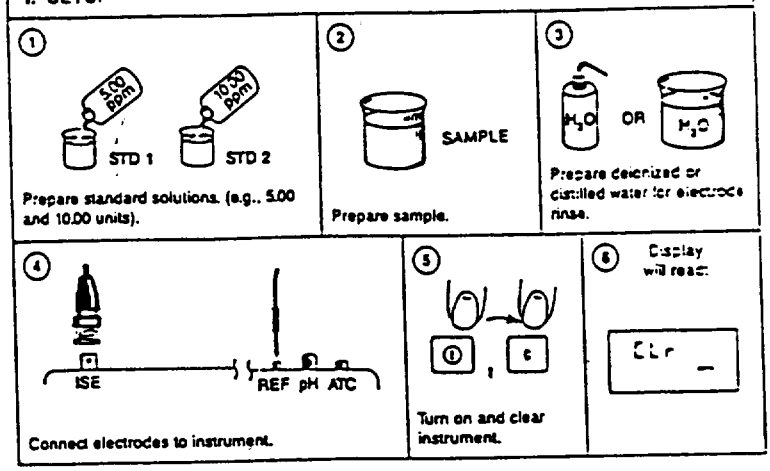
MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

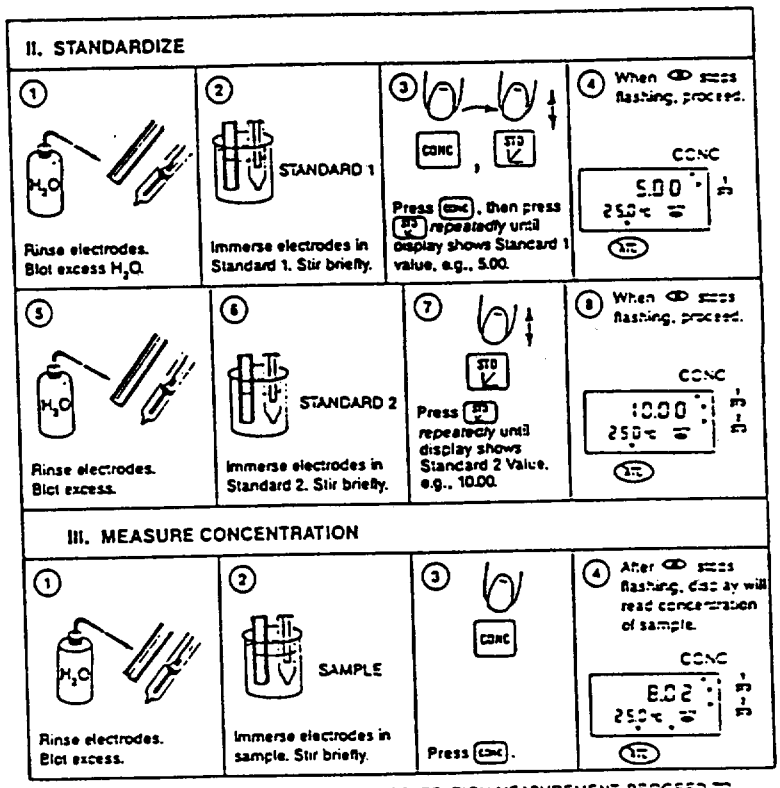
ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

I. SETUP



II. STANDARDIZE



III. MEASURE CONCENTRATION

FOR MORE DETAILED INSTRUCTIONS ON CONCENTRATION MEASUREMENT, PROCEED TO NEXT PAGE.

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

The following procedure, utilizing two-point standardization, can be used to measure concentrations of ions in almost any desired units.

STANDARD SOLUTIONS:

Standards can be made from any type of solution, with concentrations selected from the following values: 1.00, 2.50, 5.00, 10.00, 25.0, 50.0, 100.0, 250.0, 500, and 1000 units.

Units of concentration may be any that the user finds convenient. CONCENTRATION UNITS FOR BOTH STANDARD SOLUTIONS MUST BE THE SAME AS DESIRED FOR SAMPLE READINGS.

Some examples of units are: parts per million, percent, moles per liter, parts per billion, milliequivalents per liter, and ounces per gallon.

Select two standard values as close as possible to the anticipated sample value, preferably with one standard value below and one standard value above the sample. For example, if sample solution is about 150 millimoles per liter (mM), make up standards of 100 mM and 250 mM. If sample concentration varies widely, for example, between 10 molal and 75 molal, make up standards of 10 molal and 100 molal.

Standards and samples should be at the same temperature to avoid temperature-dependent variations in readings.

NOTE

Standard and sample solutions may require ionic strength adjustment or interfering ion removal. Consult electrode instructions for details.

CONCENTRATION MEASUREMENT PROCEDURE:

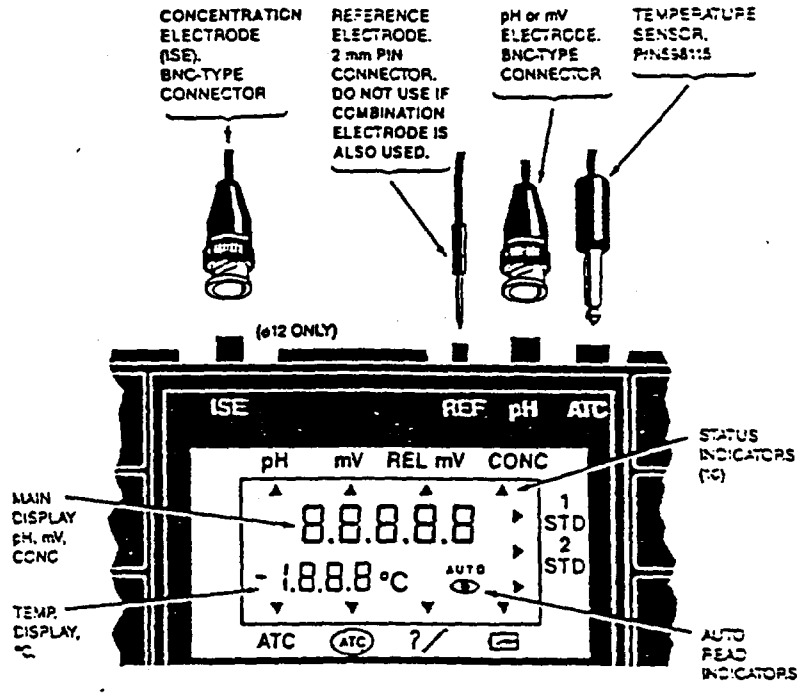
1. Connect electrodes to appropriate inputs:
   a. Connect ion-selective electrode to input marked "ISE".
   b. Connect reference electrode to input marked "REF".

NOTE

If, in addition to the ion-selective electrode, a combination pH electrode is connected to the instrument AND is to be immersed in the same solution, DO NOT use a separate reference electrode.

2. Press [⏻] to turn on instrument, then press [c] to clear. Display will show [Clr, AUTO].
3. Rinse electrodes with deionized water. Blot excess.
4. Immerse electrodes in first standard solution. Press [conc], then press [F1] as many times as needed for the concentration value of the first standard to show on the display. When [F1] is pressed repeatedly, the display steps through the following values: 1.00, 2.50, 5.00, 10, 25, 50, 100, 250, 500, and 1000 concentration units. For example, if the concentration of the standard is 100 units, press [F1] seven times and the display will show [100]. When [⊂⊃] stops flashing, display will show [100 locked, ⊂⊃ , ▶ 1/STD].
5. Rinse electrodes with deionized water. Blot excess.
6. Immerse electrodes in second standard solution. The first and second standards must be different, but can be measured in any order. Press [F1] as many times as needed for the display to show the concentration value of the second standard, e.g., 250. When [⊂⊃] stops flashing, display will show [250 locked, ⊂⊃ , ▶ 2/STD].
7. Rinse electrodes with deionized water. Blot excess.
8. Immerse electrodes in sample. Press [conc]. When [⊂⊃] stops flashing, display will show [sample value locked, ⊂⊃]. Measurement is now complete. Repeat Steps 7 and 8, above, for additional samples.
9. If *continuous* concentration readout is desired, press [auto] to turn off Auto Read function.

ELECTRODE CONNECTIONS



DISPLAY FEATURES AND STATUS INDICATORS

DISPLAY

Main Display — The large digits show the following:

1. Readout of the measured variable: pH, mV, or concentration.
2. [Clr] is displayed, indicating that instrument is cleared, when [c] is pressed.
3. Error message:

   [Er] indicates an excessive, potentially damaging, input voltage, typically caused by static electricity when the electrode pair is not in solution. In this case, immerse electrodes in solution, press [c], and proceed with measurement. If [Er] again appears, check connections and electrodes for possible open circuit.

Temperature Display (°C) — The small digits display temperature in °C. Will read 25°C if ATC not plugged in.

AUTO — AUTO READ ON/OFF Indicator for AUTO READ ON/OFF Key, described subsequently

AUTO READ Status Indicator (eye symbol). Functions during standardization and when instrument is in AUTO mode. During *standardization*, the eye symbol starts flashing when [RS] is pressed, and locks on when the reading has stabilized. During sample measurement in AUTO mode, the eye symbol starts flashing when a mode key is pressed, and locks on when the reading has stabilized. The reading remains locked until a mode key is pressed. If an interval of approximately 30 minutes elapses without a key being pressed, the instrument turns off automatically to conserve the batteries, but retains all standardization data in memory

STATUS INDICATORS

ATC — Indicates that ATC is plugged in. The instrument measures and displays temperature within the range of −5°C and 100°C. Display of [Er] indicates that the temperature sensed is outside the measurement range, or the ATC is nonfunctional.

ATC — Indicates that ATC is *not* plugged in. The temperature reading defaults to 25°C.

pH — Indicates that instrument is in pH mode.

mV — Indicates that instrument is in mV mode. } ϕ11 and ϕ12 only.

REL mV — Indicates that instrument is in relative mV mode. } ϕ11 and ϕ12 only.

Conc — Indicates that instrument is in concentration mode. ϕ12 only.

1 STD — Indicates that one standard has been used to standardize for the selected measurement mode (pH or CONC).

1 STD — Indicates that two standards have been used to standardize for the selected measurement mode (pH or CONC).

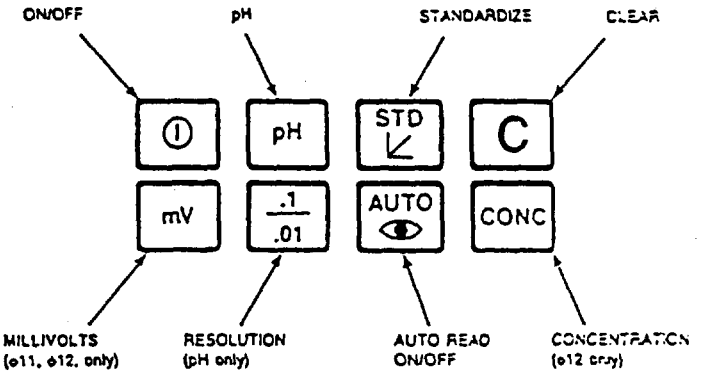
?/ — Indicates a questionable electrode and/or standardization.

Indicates that batteries should be replaced.

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

KEYPAD



KEYPAD FUNCTIONS

KEY

Instrument ON/OFF Key. When OFF, the instrument retains the standardization data in memory. Instrument shuts off automatically after 30 minutes of inactivity if AUTO READ is ON. (See below.)

Clear Key. Clears instrument, resetting all standardization data to default values, and returning instrument to AUTO Mode.

Auto Read Key. Turns Auto Read function ON and OFF:

1. When Auto Read is ON:
   a. The word [AUTO] appears on the display.
   b. The instrument tests the electrode signal for stability. During this test, [ ] flashes ON and OFF. When the signal has met the stability requirement (see SPECIFICATIONS), [ ] remains on continuously, and the digital display locks onto the reading. No further measurements are made until a key is pressed.
   c. After 30 minutes without keypad input, the instrument turns off automatically but retains all standardization data.
2. When Auto Read is OFF:
   a. [AUTO] disappears from display.
   b. The instrument continuously measures and displays in the selected mode: pH, mV, or CONC.
   c. After 1 to 2 hours without keypad input, the instrument turns off automatically but retains all standardization data.

pH Resolution Selection Key. Changes resolution of the displayed pH reading from 0.01 to 0.1 pH unit, or vice versa. At the lower resolution (0.1), time required for the Auto Read to lock is shorter. (See SPECIFICATIONS).

Selects the pH measurement mode.

Selects the mV mode (pH11, pH12), for measurement of either absolute or relative millivolts. See MEASURING mV AND RELATIVE mV.

Selects the concentration measurement mode (pH12). Used with specific ion electrodes.

Standardize Key. Standardizes instrument. Depends upon mode.

a. pH Mode: [STD] Key causes the instrument to automatically identify the pH value of the buffer from any one of the following: 1.68, 4.00, 7.00, 10.01, and 12.45.
b. mV Mode (pH11, pH12): [STD] Key causes the instrument to establish the zero-millivolt level at the value of the current reading. Instrument is now in Relative mV mode.
c. CONC Mode (pH12): Repeated pressing of [STD] Key causes the instrument to step through the following sequence of values: 1.00, 2.50, 5.00, 10, 25, 50, 100, 250, 500, and 1000 concentration units.

INSTRUMENT BECKMAN PART NO.	pHI 10 123132	pHI 11 123133	pHI 12 123134
pH MEASUREMENT			
Range	0 to 15.99 pH	0 to 15.99 pH	0 to 15.99 pH
Resolution (Selectable)	0.01, 0.1 pH unit	0.01, 0.1 pH unit	0.01, 0.1 pH unit
Relative Accuracy	± 0.01 pH	± 0.01 pH	± 0.01 pH
Auto Read Mode	0.1 pH Resolution: Display locks after reading is stable within 1.0 mV for 4 seconds. 0.01 pH Resolution: Display locks after reading is stable within 0.5 mV for 8 seconds.		
Buffers Recognized by Instrument: 1.68, 4.00, 7.00, 10.01, 12.45 pH.			
MILLIVOLT MEASUREMENT			
Range	—	−999.9 to + 999.9 mV	−999.9 to + 999.9 mV
Resolution	—	0.1 mV	0.1 mV
Accuracy	—	± 0.2 mV ± 0.02% of reading, relative mV	± 0.2 mV ± 0.02% of reading, relative mV
Auto Read Mode	—	Display locks after reading is stable within 0.5 mV for 8 seconds.	
CONCENTRATION MEASUREMENT			
mV Accuracy	—	—	± 0.1 mV
Auto Read Mode	—	—	Display locks after reading is stable within 0.25 mV for 8 seconds.
Useable Standard Values	—	—	Two values, in any desired units, selected from the following: 1.0, 2.5, 5.0, 10, 25, 50, 100, 250, 500, and 1000.

TEMPERATURE MEASUREMENT (ALL MODELS)

Range: −5°C to 100°C
Resolution: 0.1°C
Accuracy (with Beckman 598115 Probe): ± 0.5°C

MISCELLANEOUS (ALL MODELS)

Input Connections:
1. BNC input for pH, mV, and concentration.
2. 2-mm pin connector for reference electrode.
3. Miniature phone jack for Beckman 598115 Automatic Temperature Compensator.

Operating Temperature: 15°C to 40°C, ambient, non-condensing.
Power Source: Two lithium cells, 3.6 volts each, AA Size.

Error Indications:
1. Input overvoltage (all modes)
2. Temperature compensation non-functional
3. Low batteries
4. Questionable electrode/standardization.

Size: 5.2 inches x 3.9 inches x 1.3 inches.

Beckman Co
National
West Poplar Ave
... CA 93257

E-11

Platinum Combination Electrode

Catalog No. 13-639-82

The Fisher platinum combination electrode combines a silver/silver chloride reference element and a platinum-wire indicating element in a single probe. This "dual element" configuration eliminates the need for two separate electrodes and is especially applicable to measurements in narrow-neck flasks and other restricted-entry receptacles.

The platinum combination electrode is recommended for use with automatic titrators and similar electroanalytical equipment. The close physical proximity of the porous-plug liquid junction to the platinum-wire indicator section results in reduced resistance between these elements and produces a rapid dynamic response for both redox measurements and potentiometric titrations. Additionally, the electrode is ideally suited for any application that involves the measurement of oxidation-reduction potentials or requires the use of a "noble metal" sensor.

The electrode measures 5 inches with a 30-inch lead, and functions over a −5° to 110°C temperature range. The filling solution is 4M KCl saturated with AgCl (Fisher No. So-P-135), and flow rate at the junction is less than 8 μl per hour at an 8 cm head. Reference output is 44± 1mV vs. S.C.E., while junction resistance is less than 10K ohms.

INSTALLATION

Place the platinum combination electrode into service as follows:

1. Remove cap from supplied filling-solution bottle, and screw on dispenser spout.
2. Lower rubber sleeve on electrode body until filling hole is exposed, and fill reference cavity with electrolyte until meniscus reaches a level approximately ¼-inch below filling hole.

   NOTE: Always use 4M KCl solution saturated with AgCl (Fisher No. So-P-135) as the electrolyte. NEVER USE SATURATED KCl FILLING SOLUTION.
3. Place electrode upright in empty beaker to permit filling solution to wet and flow through porous plug, as evidenced by formation of KCl crystals on outer surface of plug.

   NOTE: If no flow is observed within 30 minutes, or if response is unsatisfactory during an analysis, soak electrode in dilute KCl (0.1M) for several hours, and then perform the following procedure:

   a. Hold electrode (cap up) at a 45° angle between thumb and forefinger on left hand, so that filling hole faces out and is directly opposite base of thumb.
   b. Insert dispensing spout into filling hole.
   c. Make sure that electrode is supported by base of thumb, then firmly press spout into filling hole to make an airtight seal.

      NOTE: Normally, spout tip will not touch internal element; while applying pressure, however, care should be exercised to prevent contact. If necessary, cut off a portion of the tip.
   d. While maintaining seal, squeeze filling bottle firmly so that electrode becomes pressurized.

      NOTE: A bead of liquid should form at liquid junction in about 30 seconds: in some cases, however, it may be necessary to maintain pressure for several minutes. If flow cannot be established, refer to REJUVENATION section.
4. Mount electrode on suitable holder and connect jacks to pH meter.

OPERATION

For optimum operation with the platinum combination electrode, observe the following general procedures:

1. Rubber sleeve should always be lowered on electrode body to expose filling hole and permit proper electrolyte leakage.
2. Level of electrolyte must always be maintained above surface of sample solution to avoid backflow of sample into electrolyte. Refill reference cavity as required.
3. After removing electrode from one solution and before immersing in another, the outer surface should be rinsed with distilled water.

STORAGE

When not in use, store the platinum combination electrode as follows:

1. Slide rubber sleeve into position over the filling hole.
2. Place supplied cot over tip of electrode by threading platinum wire through opening and sliding cot onto glass body until porous plug is completely covered.

REJUVENATION

Rejuvenation of the platinum combination electrode may only require a simple cleaning. Occasionally, a more thorough cleaning is required, or the porous-plug junction may have to be unblocked. Each is covered separately below.

Simple Cleaning

A simple cleaning of the electrode is done as follows:

1. Wash electrode surface with a good detergent.

   NOTE: RBS-25 detergent (Fisher No. So-C-181) is recommended.
2. Polish platinum wire with scouring powder.
3. Rinse electrode thoroughly with distilled water.

Thorough Cleaning

For a more thorough cleaning, perform the following:

1. Connect tip of large cable plug to negative terminal of a 22V dry cell, then immerse tip of electrode in a 1N solution of hydrochloric acid.
2. Similarly connect a platinum or graphite electrode to positive terminal of dry cell and immerse tip of electrode in same solution.

   NOTE: Hydrogen will evolve rapidly, and the metallic electrode will be cleaned by electrolysis in 5 to 10 seconds.
3. After cleaning, disconnect both electrodes and rinse

each with distilled water.

Unblocking the Junction

'f the liquid junction should become partially blocked, ɔerform the following:

1. Inspect reference cavity for crystallization.
2. If crystals are evident, proceed as follows:
   a. Remove filling solution by shaking it out through filling hole.
   b. Rinse cavity repeatedly with distilled water until all crystals are disolved.
   c. Refill cavity with fresh 4M KCl solution saturated with AgCl (Fisher No. So-P-135).

      CAUTION: Never use saturated KCl as the electrolyte.
   d. Repeat all of step 3 under INSTALLATION.
3. If difficulty persists, perform the following in sequence depending upon the severity of the blockage:
   a. Soak electrode overnight in dilute KCl (0.1M).
   b. Boil junction in dilute KCl for 5 to 10 minutes.
   c. Carefully sand or file the porous plug junction.

Litho in U.S.A.

APPENDIX C-6

PERCENT METHANE AND OXYGEN

SECTION 1
SCOPE AND APPLICATION

The GASTECHTOR (GASTECH) Model 1939 OX Gas Surveyor is a battery-powered portable instrument that can detect and indicate concentrations of combustible gases and oxygen in air. Combustible gas is measured in two ranges: 0 to 100% Lower Explosive Limit (LEL) and 0 to 100% by volume. Oxygen is measured over a range of 0 to 25% by volume. The instrument is equipped with a meter and audible alarms that sound whenever gases or oxygen concentrations reach preset levels.

REFERENCE

GASTECH Gas Surveyor Model 1939 OX Instruction Manual

APPARATUS

- GASTECH Model 1939 OX
- Battery charger
- Probe, 10 inch long, 1/4 inch OD plastic tube and dust filter
- Hose, 6 foot flexible nylon-jacketed polyethylene tube
- Calibration kit consisting of cylinders of methane, 2 to 5% LEL and 50 to 100% by volume concentrations

SECTION 2
DESCRIPTION

2.1 METER

Instrument readings are displayed on a meter, visible through a window on the top face of the instrument case. A mark on the scale, BATT CK, represents the minimum permissible battery voltage, as an indication of the state of the charge of the battery. The meter face has three scales.

2.1.1 The top scale is graduated from 0 to 25% O_2 for indicating concentration of oxygen. A calibration CAL mark indicates 21% O_2.

2.1.2 On the middle scale the meter reads gas concentration as 0 to 100% LEL. 100% LEL represents the minimum concentration of vapor in air that will ignite or explode when an ignition source is present.

2.1.3 The bottom non-linear scale reads 0 to 100% gas (%GAS) for indicating high concentrations of combustible gas.

2.2 CONTROLS

The six controls that are used in normal operation of the instrument are arranged on the left side of the instrument as viewed from the rear, and on the top. These controls are recessed to minimize possibility of accidental operation.

2.2.3 Gas/LEL Zero

A potentiometer which is used to adjust circuits to read zero in the absence of natural gas. The potentiometer shaft is fitted with a control knob for convenience in adjustment.

2.2.4 OXY/Gas-LEL Switch

An alternate-action push button switch used to select the combustible gas or oxygen range. In the OUT position, the instrument is in the GAS or LEL range. When IN, as indicated by the colored indicator dot, the circuit is in the OXY range.

2.2.5 Gas/LEL Switch

An alternate-action push button switch which selects the operating range for detection of combustible gas. In the OUT position, the instrument measures in the 0 to 100% LEL range. In the IN position, the instrument measures the 0 to 100% GAS range. A colored indicator dot shows when the switch is IN.

2.2.6 OXY Cal

A potentiometer which is used to adjust the circuit to read 21% when the detector is surrounded by normal air. The potentiometer shaft is fitted with a control knob for convenient adjustment.

2.3 INDICATORS

Alarm lights, red and amber, illuminate when the corresponding section of the instrument is in an alarm condition. The red light blinks in an on-off pattern of equal length pulses, to show that the instrument is in the combustible gas (LEL or GAS) alarm condition. Amber light blinks in a short-long pattern to show an abnormal oxygen condition.

2.4 BUZZER

A solid-state electronic buzzer is mounted at the rear interior of the instrument. The buzzer gives a pulsed tone on detection of gas, and a continuous tone in case of a malfunction, either low battery voltage or a down-scale drift of the meter. The audible tone pulses correspond to the visual pulses of the corresponding alarm light except in the case of excess O_2 which gives a steady tone.

TABLE 1
ALARM SETTINGS

% Oxygen:	<19.5% and >25%
% LEL:	>20% LEL
% Gas:	>20% Gas

SECTION 3
PRINCIPLE OF OPERATION

3.1 LEL RANGE

This range gives readings in terms of percent of the lower explosive limit (LEL), with 100% corresponding to the LEL of the specified gas.

Detection in this range is by catalytic oxidation on a platinum element, with the heat of oxidation producing a temperature rise and hence a resistance rise of the element. This change is approximately linear up to the LEL provided there is sufficient oxygen in the sample to support combustion.

3.2 OXYGEN SENSOR

The oxygen-sensing detector is an electrochemical cell in which electrodes are immersed in an alkaline electrolyte, and covered by a permeable fluorocarbon membrane. Oxygen from the surrounding atmosphere diffuses through the membrane and enters the electrolyte, causing an electrochemical reaction. The current generated by this reaction is amplified and used to drive the meter and the alarm circuit.

3.3 100 % GAS RANGE

This range gives readings in terms of gross concentration of the specified combustible gas. It uses a thermal conductivity filament, initially heated by the battery current to a point where it assumes a definite temperature and resistance. The temperature and resistance corresponds to the atmosphere, if the instrument is initially set-up in fresh air. Then when gas is sampled, the greater cooling ability of the gas (relative to air) causes the filament to become cooler and assume a lower electrical resistance. The resistance change produces a meter reading which can be calibrated in units of percent gas by volume.

The thermal conductivity phenomenon is continuous from 0 to 100%, so any concentration can be read without limitation. It is a relatively insensitive method, so is used only for the higher concentrations above the LEL. It is not dependent on combustion, hence is applicable regardless of the oxygen content of the sample.

SECTION 4
OPERATION

4.1 Attach the hose to the instrument by means of the threaded swivel fitting.

4.2 Put the OXY/GAS-LEL switch in the GAS-LEL (out) position, and GAS/LEL switch in the GAS (in) position, with its colored indicator showing.

4.3 Press the POWER switch to turn the instrument on, the orange indicator dot should be showing. The meter will normally rise up scale and a pulsing or steady alarm signal may sound. The alarm will stop when the instrument is warmed-up. The audible hum of the pump will be noticed.

4.4 Press the BATTERY CHECK button and note the meter reading. If the reading is close to or below the BATT CHECK mark on the meter, charge the batteries.

4.5 Allow the unit to warm up until the meter stabilizes (about a minute). Then, with the hose inlet in a contaminant-free location, turn GAS ZERO control knob to bring the meter to "0" indication.

4.6 The instrument is now ready to use in detecting high concentrations of the specified gas up to 100% by volume. The meter will indicate the concentration on the lower non-linear scale.

If the concentration indicated on the meter rises above the alarm point (set at 20%) the red ALARM light will blink and the buzzer will sound, both in a pulsing mode.

4.7 For readings in the LEL range, change the GAS/LEL switch to the LEL (out) position. The alarm will sound and the meter needle will move up-scale briefly. Wait about five minutes for the LEL sensor to stabilize. Move the probe about and observe meter indicators on the middle (% LEL) scale. The alarm point for this scale is set for 20% LEL.

4.8 Put the OXY/GAS-LEL switch in the OXY (in) position with the colored indicator showing. Turn the OXY CAL potentiometer to bring the meter to the oxygen CAL mark (21%). As a quick check gently breathe into the hose inlet and allow the instrument to sample air. The reading should come down to about 16% and the alarm should sound at 19.5%. Allow the meter to return to 21%, then put the switch back in the GAS-LEL (out) position.

4.9 Atmospheres containing more than the normal 21% oxygen will produce an increased oxygen reading. If the instrument is provided with a high oxygen alarm, then it will sound in a steady tone and the amber light will blink when readings reach or exceed 25%.

4.10 The instrument continuously tests for oxygen, and a pulsed audible alarm and blinking amber light will occur when oxygen drops to 19.5% or lower. It is not necessary to use the instrument with the switch in the OXY position unless oxygen meter readings are desired. If both abnormal conditions exist simultaneously (combustible gas and oxygen alarm), both lights will blink in their normal pattern, but the buzzer will sound continuously. A steady tone sounds when the oxygen reading exceeds 25%. This characteristic is provided to warn against the increased fire hazard due to excess oxygen. It also serves as a warning in case of oxygen cell failure in the high-output mode, which can occur occasionally. It further precludes accidental or intentional incorrect adjustment of the oxygen calibration control to an abnormally high level above 25%.

SECTION 5
CALIBRATION AND ADJUSTMENT

5.1 LEL CALIBRATION

5.1.1 Turn the instrument on and allow it to warm up and stabilize, preferably for five minutes. Be sure the batteries are charged sufficiently to read above the check mark.

5.1.2 Place the GAS/LEL switch in the LEL (out) position and the OXY/GAS-LEL switch in the GAS-LEL (out) position.

5.1.3 Open the instrument case by loosening the captive screw at the front of the unit. Lift the upper half of the case slightly, moving it 1/4 inch to the to rear to disengage the rear clamp; then separate the two halves. Locate the COARSE ZERO potentiometer on the underside of the circuit board (see Figure 2).

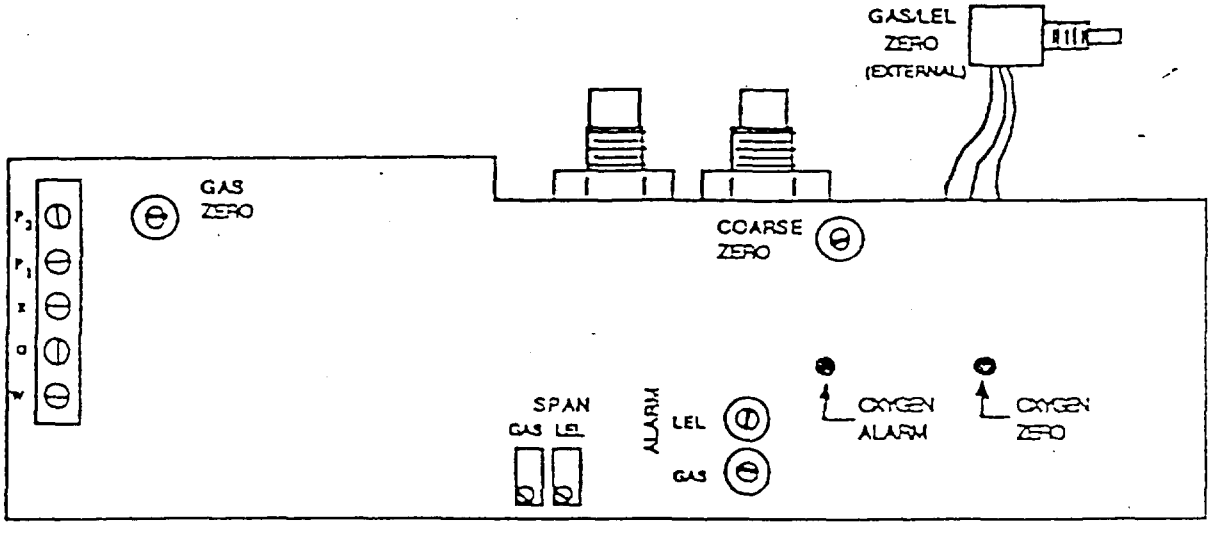


FIGURE 2

5.1.4 Turn the external GAS/LEL ZERO control to the center of its range.

5.1.5 Turn the COARSE ZERO potentiometer to bring the meter to the zero reading.

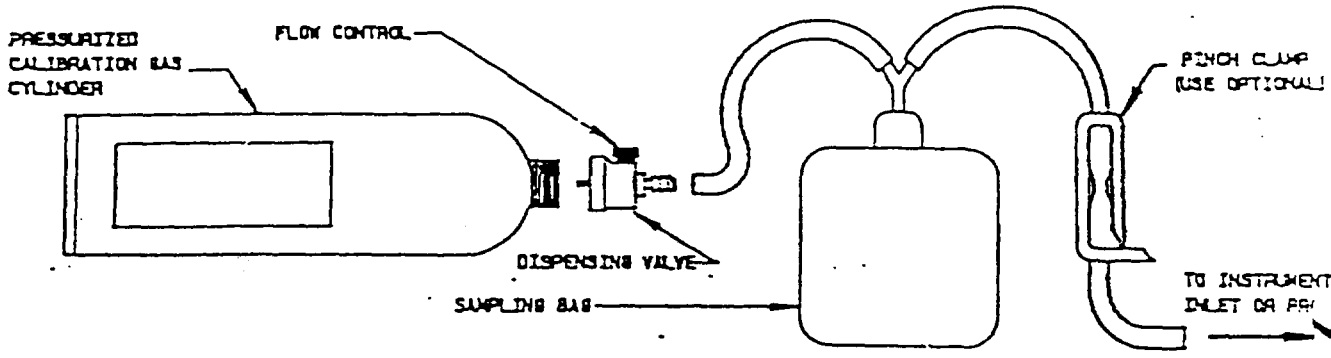


FIGURE 3

5.1.6 Remove the protective red plastic cap from the cylinder outlet of a tank of methane (2 to 5% LEL). See Figure 3.

5.1.7 Check the DISPENSING VALVE to see that the FLOW CONTROL is shut off (clockwise), but not so tightly as to damage the valve seat.

5.1.8 Screw the DISPENSING VALVE onto the cylinder outlet, being careful not to cross-thread. Tighten the valve firmly.

5.1.9 Push the short piece of flexible plastic tubing from the Y-fitting onto the barbed nipple of the DISPENSING VALVE.

5.1.10 Locate the adjustment potentiometer(s).

5.1.11 Connect the longer plastic tube with the PINCH CLAMP to the instrument. The PINCH CLAMP should be open (push latch forward to open).

5.1.12 Allow the instrument to evacuate the SAMPLING BAG until flat then close the PINCH CLAMP (squeeze paralleled faces together until tight and latched).

5.1.13 Open the FLOW CONTROL slightly so that the SAMPLING BAG begins to expand, then quickly open the PINCH CLAMP. As the instrument processes the sample, the bag will either start to expand further or to deflate. If the bag is expanding, close down on the FLOW CONTROL (clockwise), or if it is deflating, open the FLOW CONTROL slightly until the bag stays about half inflated.

5.1.14 Observe the meter on the instrument. The reading will increase, then stabilize. As soon as the reading is stable, compare with the value on the pressurized calibrating gas cylinder and record or change with the LEL SPAN potentiometer as appropriate.

NOTE Care must be taken to ensure that the LEL SPAN potentiometer and not the LEL alarm is adjusted.

5.1.15 Quickly close the FLOW CONTROL on the DISPENSING VALVE (clockwise) and disconnect the LEL calibration cylinder.

5.2 % GAS CALIBRATION

The 0 to 100% GAS range is calibrated in the same manner as the LEL range above, except adjustment is made using the GAS ZERO and GAS SPAN potentiometers and a cylinder of 100% methane (natural) gas. Place the GAS LEL switch in the 100% GAS (in) position for this calibration. **Do not change other zero adjustments.**

When the calibration is complete, remove the DISPENSING VALVE from cylinder before storage, to prevent loss of cylinder contents, then cap the cylinder. SAMPLING BAG, DISPENSING VALVE and PINCH CLAMP may be left connected to the tubing for storage.

5.3 OXYGEN CALIBRATION

5.3.1 Calibrate in fresh air.

5.3.2 Put the OXY/GAS-LEL switch in the OXY (in) position with the colored indicator showing.

5.3.3 Turn the OXY CAL potentiometer to bring the meter to the oxygen CAL mark (21%).

NOTE If problems are encountered during the calibration procedures, the unit must be replaced.

NOTE Instrument alarms are factory-set and must not be changed.

SECTION 6
MAINTENANCE/TROUBLE SHOOTING

6.1 BATTERIES

6.1.1 Check the battery voltage periodically by pressing the BATTERY CHECK switch. Charge the batteries before the voltage reaches minimum.

When connecting the charger, always follow these steps:

a. Confirm that the plug is inserted in the correct way, with the THIS SIDE UP label upwards. The plug is keyed with a non-current carrying third socket to aid in proper insertion.

b. Verify that the amber light on the charger is on. Leave connected until the green light comes on, indicating that the 16 hour charge is completed.

6.1.2 If sufficient voltage cannot be obtained after charging, the battery needs to be changed.

6.2 CATALYTIC SENSOR (LEL RANGE)

The sensor assembly may require replacement if:

a. The meter cannot be set to zero within the range of the GAS/LEL ZERO potentiometer.

b. The meter cannot be set to the desired level within the range of its SPAN potentiometer.

6.3 THERMAL CONDUCTIVITY SENSOR (GAS RANGE)

The sensor assembly may require replacement if:

a. The meter cannot be set to ZERO within the range of the GAS ZERO potentiometer.

b. The meter cannot be set to the desired level within the range of its GAS SPAN potentiometer.

6.4 OXYGEN DETECTOR

The oxygen sensor assembly may require repair if the meter cannot be set to the desired level within the range of the OXY CAL adjustment.

SECTION 7
PRECAUTIONS AND NOTES ON OPERATION

7.1 HEATED SAMPLES

When sampling spaces that are warmer than the instrument, such as hot manholes, remember that condensation can occur as the sample passes through the cooler sample line. Water vapor condensed in this way can block the flame arrestors and interfere with sensors and pump operation.

7.2 FILAMENT POISONING

Certain substances have the property of desensitizing the catalytic surface of the platinum filament. These substances are termed catalyst poisons and can result in reduced sensitivity or in failure to give a reading on samples containing combustible gas. The most commonly encountered catalyst poisons are silicone vapors. Samples containing such vapors, even in small proportions, should be avoided.

Frequent calibration checks on known-gas samples are desirable, especially if the possibility exists of exposure to silicones.

7.3 OTHER GASES AND VAPORS

The instrument is designed and calibrated specifically for detection of methane. It can be calibrated for use on other gases and vapors, by proper adjustment of the calibration control while sampling a known gas-air mixture.

7.4 RICH MIXTURES

When sampling rich mixtures, on the 100% LEL range, the following instrument action may be expected:

1. Mixtures up to 100% LEL - Reading on scale

2. Mixtures between LEL and Upper Explosive Limit (UEL) - Readings at top of meter

3. Mixtures above UEL - As sampling continues, the meter first goes to the top of the scale, then comes back down on scale. Very rich mixtures will give close to a zero reading on the catalytic sensor.

Rich mixtures should always be tested first on the 100% GAS range, to avoid any uncertainty as to actual concentration.

7.5 OXYGEN DEFICIENT MIXTURES

Samples which do not have the normal proportion of oxygen may tend to read low in the LEL range, as there is not enough oxygen to react with all the combustible gas present in the sample. As a general rule, samples containing 10% oxygen or more have enough oxygen to give a full reading on the catalytic sensor for any combustible gas sample up to the LEL. This limitation does not apply to the thermal conductivity sensor used for the 100% GAS range.

NOTE For very rich or oxygen-lean mixtures the instrument should be used in the 0 to 100% GAS range.

Erik A. Goplin
Loss Control Specialist

Mary Ann Latko, CSP
Corporate Health and Safety Manager

EAG/dlk/MAL/H&SPC
SAFETY:IOPS

APPENDIX C-7

CARBON DIOXIDE

BLUE BOOK

SENSIDYNE®

Gastec Precision Gas Detector System Manual

SENSIDYNE®

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I. GENERAL

1. Gas Detector Tube System

In order to insure environmental improvement it is most important to measure simply and rapidly very minute gas or vapor concentration in the work place.

Modern technology provides a variety of instruments for the measurement of hazardous gases and vapors. Such relatively sophisticated instruments must:

1. be capable of measuring concentrations in the range of threshold limit values.
2. must be easy to use, and available for taking quick samples in the work environment.
3. must produce results for evaluation and analysis within a few minutes, making it unnecessary to take samples back to the laboratory.
4. must provide dependable accuracy and reproducibility.
5. make it unnecessary to use expensive and delicate instrumentation.

The following three steps are essential for the chemical analysis of toxic gas or vapor:

1. collecting of samples
2. preparation of measuring apparatus and reagents
3. chemical treatment and analysis of the samples

The introduction of detector tube technology eliminates steps two and three above and makes it possible to achieve immediate results with the accuracy and reproducibility necessary for environmental assessment.

2. Principle of Gastec Detector Tube

Each detector tube contains a precise amount of detecting reagents in a constant inner diameter glass tube and is hermetically sealed at both ends.

To operate, break tips off a fresh tube and connect the tube to the Gastec sampling pump and pull the handle to take the required sample (sample gas). The chemical reagent in the detector tube will then react with the sample gas immediately and a color stain will develop starting at the inlet of the detector tube. The gas concentration is measured at the interface of stained-to-unstained reagent when staining stops.

Gastec detector tubes contain colorimetric reagents adsorbed on fine grain silica gel, activated alumina or other adsorbing media. The reagents are sensitive to particular gases or vapors and react quantitatively to provide a length-of-stain indication.

3. Characteristics of Gastec Gas Detector System

Detector Tube

1. Precision direct reading calibration scale printed on each tube.
2. Every tube and tube box displays the quality control number and chemical symbol. The expiration date is stamped on each box.
3. Controlled small internal diameter of tubes provide longer stain length with one pump stroke (100 ml), resulting in more accurate readings and shorter sampling times.
4. Sensitive reagents with high reaction rates provide a clear line of demarcation of color stain, for improved accuracy in tube reading.

Sampling Pump

1. Light weight, compact (approx. 10 oz.).
2. Pulls the highest vacuum (8.1" of Hg).
3. Gastec design does not require flow-rate orifices.
   Malfunction of the pump by clogging or leaking orifices is eliminated.
4. Handle locks at precise intake volume of 50 or 100 cc.
5. Friction-proof lubricant seal packing provides complete leakproof sampling at all times.

4. Accuracy of Gastec Detector Tube

Calibration Scale

The calibration curve on most Gastec detector tubes is a straight line, and points on the scale are at equal intervals (Fig. 1). Therefore, it is possible to measure the concentration below the lowest calibration scale by repeating pump strokes. It is also possible to measure the concentration above the highest calibration scale by using a half-pump stroke. This means that the measuring range of Gastec detector tubes is easily and accurately expanded by varying the number of pump strokes.

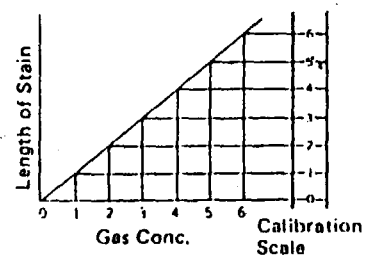


Fig. 1

Since all of the Gastec tubes are direct reading, the calibration scales are printed on the basis of individual production lots. Therefore, the variation of inner tube diameters, precision of tube packing, and the quality and reactivity of each reagent are eliminated. There is no need for extra charts or graphs whose accuracy is questionable for anything other than a single production run.

Certification of Gas Detector Tube Units

The National Institute for Occupational Safety and Health (NIOSH) has established a testing and certification program to insure that devices and instruments used in assessing and protecting against health hazards meet acceptable performance requirements. The Occupational Safety and Health Act (1970) requires the use of certified equipment unless no such equipment exists. At this point, detector tubes and sound level meters are the only instruments being tested under this program.

Following are the details of the test standard as published in the Federal Register dated May 8, 1973:

1. Every manufacturer must submit the quality control documentation including the quality control system of the company, the quality control program and the

quality control plan of the measuring apparatus to the Quality Control Section of TCL prior to the certification test. The applicant will be granted its basic qualification by the Section after the documentation check.
2. In accordance with the Federal Register, the applicant must submit the quality control documents for the detector tube to be tested, with test results certifying that the tubes satisfy the standard, and an appropriate number of tubes. After passing this check, the applicant may proceed to the next step.
3. The applicant submits the detector tubes for inspection. The inspection procedure is divided into two parts:
   1) physical inspection tests such as the longitudinal axis of glass tubes, detecting layer and calibration scale and
   2) chemical reaction tests for indicating accuracy with standard gas mixture.

   Physical inspection tests are performed during a sampling inspection in accordance with Inspection Level II as described in MIL-STD-105D. The tubes which pass the above inspection proceed to the Air Sampling Section and are tested for accuracy.
4. The accuracy test is conducted on four concentrations; one-half, one, two and five times the TLV in accordance with the Inspection Level IV as described in MIL-STD-104.
   The standard gas generation method and the standard method of analysis for the test are verified in the Microanalysis Laboratory of the National Bureau of Standards; then the concentration of the gas is confirmed by the Gas Detector Tube Standard Committee. The detector tube stained by the standard gas concentration is read by the three (3) independent tube readers. (They will read 4 end points of circumference on the longitudinal axis.) The results of the tube reading are put into the computer, and the standard deviation percentages are then calculated.
5. To be certified by NIOSH, the detector tube must satisfy the following test standards:
   1) For the accuracy test, gas detector tube units shall produce measurements of contaminant concentration with ±35% of the actual value at one-half the test standard and within ±25% of the actual value at 1, 2 and 5 times the test standard.
   2) The standard deviation of the tube readings obtained from three independent tube readers shall be within 10%.
   3) On channelling of airflow through the detector tube, the maximum variation of stain length around the circumference of the tube at the interface between stained and unstained reagents shall be within 20%.
6. Detector tubes certified under this program will be published in the Federal Register at that time, the certification number will be issued, and the government will place the item on the approved list. Thereafter, NIOSH may purchase the tubes from the market at random and test them to determine if the tubes continue to meet these exacting standards. Tubes which fail to meet the test standard will have the certification withdrawn until the defect is corrected. The following Gastec Detector Tubes have been certified by NIOSH and issued the certification number:

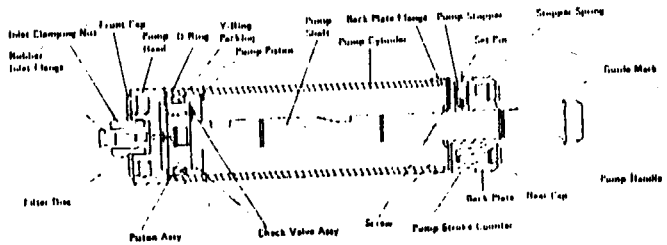
NOTE: NIOSH Certification Program has been discontinued.

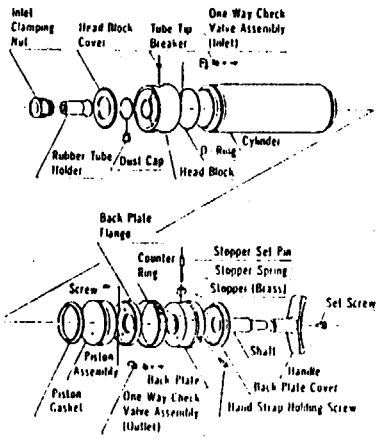
Tube No. 1La	Carbon Monixide	TC-84-014
Tube No. 5La	Sulfur Dioxide	TC-84-017
Tube No. 9L	Nitrogen Dioxide	TC-84-018
Tube No. 4LL	Hydrogen Sulfide	TC-84-020
Tube No. 2L	Carbon Dioxide	TC-84-021
Tube No. 3M	Ammonia	TC-84-023
Tube No. 134	Carbon Tetrachloride	TC-84-036
Tube No. 132H	Trichloroethylene	TC-84-038
Tube No. 8La	Chlorine	TC-84-041
Tube No. 121	Benzene	TC-84-043
Tube No. 10	Nitric Oxide	TC-84-049
Tube No. 122	Toluene	TC-84-053
Tube No. 131La	Vinyl Chloride	TC-84-060
Tube No. 12L	Hydrogen Cyanide	TC-84-068

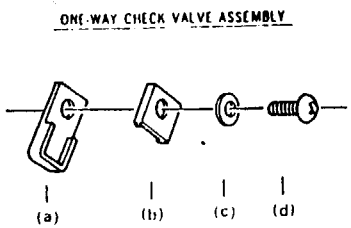
5. Gastec Pump Performance

Description of Pump

Construction of the Gastec pump is illustrated below. This pump pulls the highest vacuum for its type (8.1" of Hg). There are no flow-rate orifices to cause malfunction of the pump by clogging or leaking. A friction-proof piston gasket (lubricant seal packing) provides complete leakproof sampling at all times.







(a) Rubber valve flap
(b) Valve retainer
(c) Plain washer
(d) Valve screw

Checking Pur(erformance

A. Visually check rubber inlet flange for cracks or tears. Replace if damaged. Tighten inlet clamping out.

B. Valve Leak Check

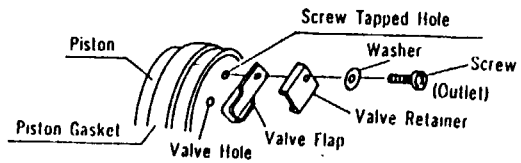
1. Insert a fresh sealed detector tube into pump. Misalign red dots on pump and handle. Pull several fairly rapid continuous full pump strokes.
2. Pull handle out 6 mm (1/4 inch) and hold in this position for 1 or 2 seconds.
3. Release handle.
4. If handle return to within 1.5 mm (1/16 inch) or less of fully closed position, continue to step C.
5. If handle does not return to within 1.5 mm (1/16 inch) of fully closed position (or less), perform the following Valve Lubrication instruction outlined below.

C. Field Volume Check

1. Insert a fresh sealed detector tube into pump.
2. Align Guide Marks on pump body and handle.
3. Pull handle firmly and at a moderate speed until handle locks into position. Wait 1 minute.
4. Unlock handle by turning it and guide it back.
   TO PROTECT PUMP STOPPER from breakage, do not release the handle and allow it to spring back when conducting a leak test. Make sure you hold your hand onto the handle and guide it back.
5. Pump handle should return to within 6 mm (1/4 inch) or less of the fully closed position.
6. If pump handle does not close to within 6 mm (1/4 inch) or less, follow lubrication instructions and retest.

D. Lubrication Instructions (Perform Laboratory Volume Check "E" after each lubrication)

1. Valve Lubrication
   a. Unscrew back plate and withdraw piston from pump cylinder.
   b. Remove check valve from piston.
   c. Clean valve and piston with lint-free cloth. Proper valve cleaning is as follows:
      Place cloth flat on desk.
      Wipe rubber valve flap in a flat position across cloth.
      Do not bend the rubber flap valve.
   d. Apply a small amount of grease evenly around the valve opening to form a thin film. A thin film is nearly invisible.
   e. Replace valve assembly loosely in the same manner as removed.



f. Before tightening the screw, align valve so that valve hole is in center of valve flap.

g. Then push the rectangular valve retainer all the way toward loose end of valve falp.

h. Now tighten screw. If a torque driver is available, tighten to 0.8 Kg-cm. Otherwise, be careful not to overtighten screw. When tightened, screw must not deform rectangular valve retainer.

2. Piston Gasket Lubrication

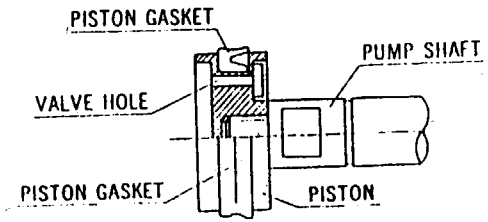
a. Wipe off piston and cylinder with a clean lint-free cloth.

b. Remove piston gasket with a small bladed screwdriver. Take care not to cut gasket.

c. Clean slot in piston with lint-free cloth. Wipe off rubber gasket.

d. Wipe an ample supply of grease into gasket slot on piston and inside gasket.

e. Replace gasket making sure that open side of gasket is toward pump handle.



f. With the excess grease from piston slot, wipe around outside of gasket and piston.

g. Wipe an ample amount of grease into cylinder at the area of piston entrance.

h. Insert piston slowly into the cylinder. Work the piston back and forth slowly in the cylinder several times.

i. Now screw back plate firmly onto cylinder.

j. Repeat leak tests.

k. If any leak remains, replace piston gasket.

l. Only if a leak persists, go to procedure below.

3. Pump Head Lubrication

a. This is only necessary where all previous procedures have failed to correct a leak.

b. Visually check pump head "O" ring for cracks.

c. Replace "O" ring if cracked.

d. Place a light coat of grease on pump cylinder head screw threads and the "O" ring.

e. Insert new "O" ring.

f. Screw pump head firmly on to "O" ring and make sure "O" ring is seated uniformly. Overtightening pump head may push "O" ring out of place. Do not overtighten.

g. Wipe off excess grease.

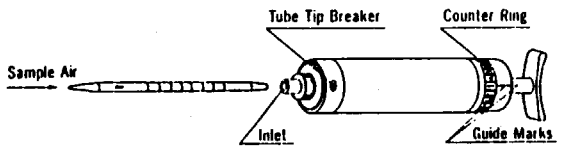
E. Laboratory Volume Check (To be performed at least once after each lubrication)

The Gastec pump can be checked periodically to assure that 100 ±5 ml are being sampled.

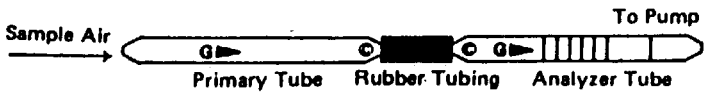
1. Arrange a graduated 100 ml soap film flow meter in a volume test mode.
2. Insert a fresh Gastec tube into the pump. The tube must be broken at both ends (ready for use).
3. Attach the Gastec tube to top of soap film flow meter with rubber hose. Make sure there are no leaks.
4. Pull pump handle out full to lock at one stroke in normal sampling manner.
5. Wait until the bubble stops moving and read the volume evacuated.
6. If the volume evacuated is other than 100 ± 5 ml, proceed to lubrication instruction and retest.

6. Operating Procedure for Gastec Precision Gas Detector System

Sampling & Measurement Procedure



1. Break tips off a fresh detector tube by bending each tube end in the tube tip breaker of the pump.
2. Insert tube securely into pump inlet with arrow on tube pointing toward pump.
3. For twin tubes, connect ⓒ marked ends with rubber tubing after breaking each end. Insert analyzer tube into pump with arrows on tubes pointing toward pump. See figure below.



4. Make certain pump handle is all the way in. Align guide marks on pump body and handle.
5. Pull handle out to desired stroke volume. Handle can be locked on either 1/2 pump stroke (50 ml) or 1 pump stroke (100 ml).
6. Read concentration at the interface of stained-to-unstained reagent when staining stops. Unlock handle by making 1/4 turn and return it to starting position.
7. In case more pump strokes are indicated in the instruction sheet included in each box of tubes, take additional sample by repeating pump strokes without removing tube.

Correction for Temperature, Humidity or Pressure:

Calibration of the Gastec detector tubes is based on a tube temperature of 20°C

(68° F), approximately 50% relative humidity, and normal atmospheric pressure.

1. No correction is normally required for tube temperature of 20° – 40° C (32° – 104° F) and for relative humidity range of 20 – 90%.
2. Where the detecting reagent is abnormally sensitive to temperature or humidity, a correction table or chart is provided in each box of tubes. In this case, the tube reading must be corrected using correction table or chart.
3. Tube reading is proportional to absolute pressure. To correct for pressure, multiply by

$$\frac{760}{\text{Atmospheric Pressure (mmHg)}}$$

How to Use Counter Ring:
In case numerous pump strokes are required, use the counter ring of the pump.
(1) Turn the counter ring manually counterclockwise after each pump stroke.
(2) The operator may thus keep count of the number of pump strokes.

For more details see the instruction sheet included in each box of detector tubes.
Detector tube should be stored in accordance with instructions on each box.

7. Different Methods of Sampling

1. Effect of sampling time and related data

Concentration of contaminants and chemical substances in the air is always fluctuating. On the other hand, sampling is usually conducted within certain time and frequency limits. Therefore, a deviation naturally exists between the average environmental concentration and results obtained on the basis of "grab" samples.

For example, Fig. 2 below shows time-lapse variation of dust concentration which was observed by using Tyndallometer readings plotted in 30-second intervals. This test was conducted at a raw material mixing plant in the glass mill.

The variation of concentration during the hour was up to 60 times the average concentration of dust. This idea can be adapted to the gas concentration in air of toxic substances as follows:

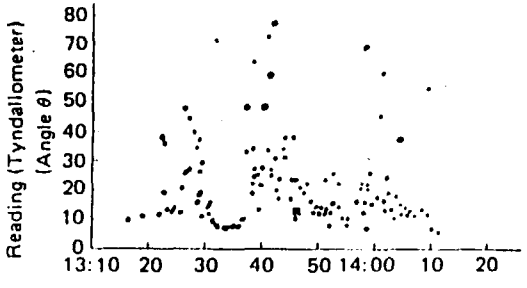


Fig. 2 Time-lapse Variation of Dust Concentration
(Place: Raw material mixing plant in glass mill)
by Tyndallometer

The variation of the measured concentration is largely dependent on the sampling time and position. Large differences occur when the operation is intermittent. If the sampling time is short and the quantity of sample gas or vapor is relatively small, large variations will result between instantaneous concentration and average concentration. Under extremely adverse conditions, there can be as much as ten fold difference in detector tube readings taken at different times in the same location.

2. Tanks, Manholes, or Closed Places

It is dangerous to enter a manhole directly without a pre-check. Before entering any such closed place, the workman must first check the gas level by using an extension hose in at least three levels, that is the higher, middle, and lower level of the closed area. The reason for this is as follows:

$$\text{Specific Gravity of Gas} = \frac{\text{Molecular Weight of Gas}}{\text{29 (Average MW of Air)}}$$

If the total fraction is greater than 1.0, it means the gas will rise to the top. However, if the total fraction equals more than 1.0, it means the gas concentration is increasing at the lower level.

3. Leak-check from Pipeline

If an air flow or a breeze is observed prior to taking the measurement, measuring points must be moved away from the direction of the air flow.

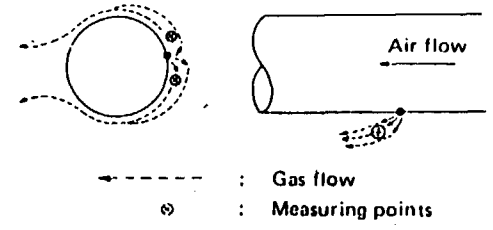


Fig. 3

Caution: If the measurement is conducted at a point some distance from the pipeline, the gas emitted will be diluted very rapidly, and the true concentration of the gas cannot accurately be detected. Therefore, measurement must be conducted at a point as close as possible to the suspected leak.

4. Measurement in a Chemical Plant, Gas Concentration in Pipelines

It is sometimes necessary to collect the gas sample temporarily in a sampling bag or a bottle as shown in the two examples below. Measurement of the concentration is made by passing a sample through an appropriate Gastec tube.

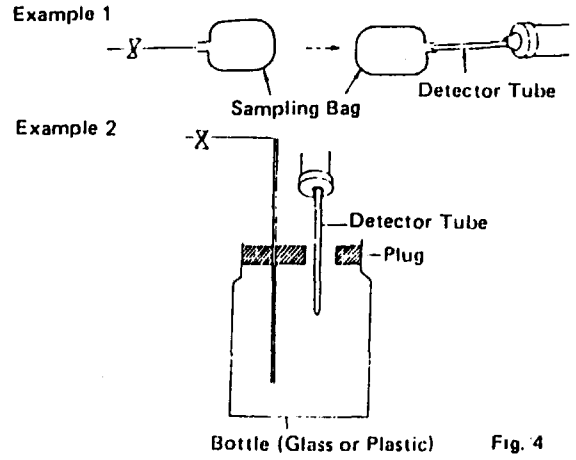


Fig. 4

5. Measurement and Threshold Limit Value of Liquid Mixture

A rapid reliable method for measuring concentration of industrial solvents in air is available using the Gastec Precision Gas Detector System.

5-1 Elements Evaporating from Solvent

Industrial solvents are liquids but have an appreciable vapor pressure at room temperature. The composition of the vapor arising from the solvent can be quite different from the composition of the solvent itself.

Take, as an example, a lacquer thinner mixture of 66% of toluene, 14% each of ethyl acetate and butyl acetate, 4% of butyl alcohol and 2% of cellsolve. At the early stage of its evaporation, the lower-boiling point metarial vaporize.

If a coating plant uses the above thinner, the thinner will vaporize according to the chart below.

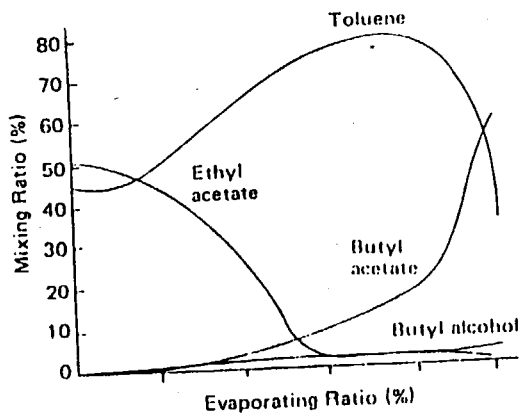


Fig. 5 Evaporation from Mixing Solvent

The ratio of vaporized components is greatly dependent upon the working environment even if the same solvents are used. Therefore we must carefully consider the environmental assessment of exposure to complex mixtures. Many lacquer thinner manufacturers keep the ratio of materials secret. However, OSHA has legislated that each producer must publish the principal components and hazardous properties of all proprietary solvents in Material Safety Data Sheets.

5-2 Selecting Measuring Apparatus

One of the best methods used in measuring each component of a solvent mixture is by means of gas chromatography. However, a simpler analysis using detector tubes is usually preferred for daily environmental measurement. Use of detector tubes and selection of tubes is greatly dependent on the individual case. It is recommended that a specialist be consulted for correct interpretation of the results. Items such as interferences and reaction rates have a major effect on the accuracy and precision of the measurement.

5-3 Threshold Limit Value for Mixtures

When two or more hazardous substances are present, their combined effect, rather than that of either individually, would be given primary consideration. In the absence of information to the contrary, the effects of the different hazards should be considered as additive. That is, if the sum of the following fractions,

$$\frac{C_1}{T_1} + \frac{C_2}{T_2} + \cdots\cdots \frac{C_n}{T_n} = 1$$

exceeds one, then the threshold limit of the mixture should be considered as being exceeded C_1 indicates the observed atmospheric concentration, and T_1 the corresponding threshold limit (see example below).

It is essential that the atmosphere be analyzed both qualitatively and quantitatively for each component present, in order to evaluate compliance or noncompliance with this calculated TLV.

$$\frac{C_1}{T_1} + \frac{C_2}{T_2} + \cdots\cdots \frac{C_3}{T_3} + \cdots\cdots = 1$$

Example: Air contains 5 ppm of carbontetrachloride (TLV = 10 ppm) 20 ppm of 1, 2-dichloroethane (TLV = 50 ppm) and 10 ppm of 1, 2-dibromoethane (TLV = 20 ppm)

Atmospheric concentration of mixture = 5 + 20 + 10 = 35 ppm of mixture

$$\frac{5}{10} + \frac{20}{50} + \frac{10}{20} = \frac{25+20+25}{50} = 1.4$$

Threshold limit is exceeded. Furthermore, the TLV of this mixture may be calculated by reducing the fraction to 1.0; i.e.

$$\text{TLV of mixture} = \frac{35}{1.4} = 25 \text{ ppm}$$

When a given operation or process characteristically emits a number of harmful dusts, fumes, vapors, or gases, it will frequently be only feasible to attempt to evaluate the hazard by measurement of a single substance. In such cases, the threshold limit used for this substance should be reduced by a suitable factor, the magnitude of which will depend on the number, toxicity and relative quantity of the other contaminants ordinarily present.

Examples of processes which are typically associated with two or more harmful atmospheric contaminants are welding, automobile repair, blasting, painting, lacquering, certain foundry operations, diesel exhausts, etc.

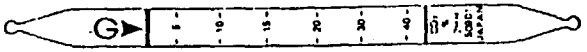
II Gastec Detector Tubes

These definitions apply to each of the following pages.

1. Performance of the Tube (n: Number of Pump Strokes)
2. Detecting Principle of the Gas
3. Interferences of the Tube
4. Method of Standard Gas and Analysis
   - 4.1 Standard Gas and Analysis
   - 4.2 Method of Analysis
5. Dangerous and Hazardous Properties
   - 5.1 T.L.V.-T.W.A.: The timeweighted average concentration for a normal 8-hour workday or 40-hour workweek, to which nearly all workers may be repeatedly exposed, day after day, without adverse effect.
     Threshold Limit Value-Time Weighted Average for 1985 adopted by American Conference of Governmental Industrial Hygienists.
   - 5-2 T.L.V.-STEL: The maximum concentration to which workers can be exposed for a period up to 15 minutes continuously without suffering from 1) intolerable irritation, 2) chronic or irreversible tissue change, or 3) narcosis of sufficient degree to increase accident proneness, impair self-rescue, or materially reduce work efficiency, provided that no more than four excursions per day are permitted, with at least 60 minutes between exposure periods, and provided that the daily TLV-TWA also is not exceeded.
     Threshold Limit Value-Short Term Exposure Limit for 1985 adopted by American Conference of Governmental Industrial Hygienists.
   - 5-3 F.L.: Flammable Limits In Air (lower and upper limits)
6. Physical Constants
   - 6-1 C.F.: Chemical Formula
   - 6-2 M.W.: Molecular Weight
   - 6-3 S.G.: Specific Gravity, Gas @20°C (68°F), NTP 1 atm unless otherwise stated
   - 6-4 M.P.: Melting Point
   - 6-5 B.P.: Boiling Point
   - 6-6 AIT: Autoignition Temperature
   - 6-7 V.P.: Vapor Pressure (mmHg)
7. Application for other gases — Other gases for with the some detector tube may be used to detect.

CARBON DIOXIDE EXTRA HIGH RANGE TUBE — No. 2HH

1. Performance



Calibration Scale	5 - 40% (n=1/2)	Color Change	White - Purple
Measuring Range	2.5 - 5% (n=1) 5 - 40% (n=1/2)	Sampling Time	1 min./pump stroke
Detecting Limit	0.5% (n=1)	Shelf Life	3 years

2. Detection Principle

Carbon dioxide reacts with hydrazine to form carbonic acid monohydrazid, which discolors redox indicator (crystal violet).

$$CO_2 + N_2H_4 \text{ — } NH_2 \cdot NH \cdot COOH$$

3. INTERFERENCES:

Other gases and vapors do not affect the tube reading

4. 4.1 Standard Gas Generation Method: Cylinder bottle gas & dynamic gas flow system

4.2 Method of Analysis: Gas chromatography (TCD)

5. Dangerous & Hazardous Properties

	Concentration	Phisical Effects
T.L.V.: 5000 ppm	2.5%	No effect for a few hours
STEL: 15000 ppm	10%	Can endure for a few minutes
F.L.:	12 – 15%	May cause death in exposures for several hours

6. Physical Constants

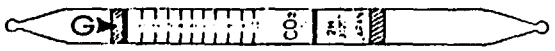
C.F.: CO_2 M.W.: 44.01 S.G.: 1.53
M.P.: −56.6°C B.P.:
AIT: V.P.:

7. Application for other gases

8510

CARBON DIOXIDE HIGH RANGE TUBE — No. 2H

1. Performance



Calibration Scale	1.0 - 10% (n=1)	Color Change	White - Purple
Measuring Range	0.5 - 1.0% (n=2) 1.0 - 10% (n=1) 10 - 20% (n=1/2)	Sampling Time	1 min./pump stroke
Detecting Limit	0.1% (n=2)	Shelf Life	3 years

2. Detection Principle

Carbon dioxide reacts with hydrazine to form carbonic acid monohydrazide, which discolors redox indicator (crystal violet).

$$CO_2 + N_2H_4 \rightarrow NH_2 \cdot NH \cdot COOH$$

3. Interferences:
Other gases and Vapors do not affect the tube reading.

4. 4.1 Standard Gas Generation Method: Cylinder bottle gas & dynamic gas flow system

4.2 Method of Analysis: Gas chromatography (TCD)

5. Dangerous & Hazardous Properties

	Concentration	Physical Effects
T.L.V. = 5,000 ppm	2.5%	No effect for a few hours
STEL = 15,000 ppm	10%	Can endure for a few minutes
F.L. =	12 – 15%	May cause death in exposures for several hours

6. Physical Constants

C.F.: CO_2 M.W.: 44.01 S.G.: 1.53 M.P.: −56.6°C B.P.: −78.5°C
AIT: V.P.:

7. Application for other gases

8301

CARBON DIOXIDE LOW RANGE TUBE

No. 2L

1. **Performance**

Calibration Scale	0.25 – 3.0% (n=1)	Color Change	White - Purple
Measuring Range	0.13 – 0.25% (n=2) 0.25 – 3.0% (n=1) 3.0 – 6.0% (n=½)	Sampling Time	2 minutes/pump stroke
Detecting Limit	0.025% (n=2)	Shelf Life	3 years

2. **Detection Principle**

Carbon dioxide reacts with hydrazine to form carbonic acid monohydrazide, which discolors redox indicator (crystal violet).

$$CO_2 + N_2H_4 \longrightarrow NH_2 \cdot NH \cdot COOH$$

3. Interferences:
Other gases and vapors do not affect the tube reading.

4. 4.1 Standard Gas Generation Method: Cylinder bottle gas & dynamic gas flow system
4.2 Method of Analysis: Gas chromatography (TCD)

5. **Dangerous & Hazardous Properties**

T.L.V. = 5000 ppm
STEL = 15000 ppm
F.L. =

Concentration	Physical Effects
2.5%	No effect for a few hours
10%	Can endure for a few minutes
12 – 15%	May cause death in exposures for several hours

6. **Physical Constants**

C.F.: CO_2 M.W.: 44.01 S.G.: 1.53 M.P.: -56.6°C B.P.:
AIT: V.P.:

7. Application for other gases

8201

CARBON DIOXIDE EXTRA LOW RANGE TUBE

No. 2LL

1. **Performance**

Calibration Scale	300-5000 ppm (n=1)	Color Change	White - Purple
Measuring Range	100 - 300 ppm (n=3) 300-5000 ppm (n=1) 4600-11500ppm(n=1/2)	Sampling Time	2 min./pump stroke
Detecting Limit	30 ppm (n=1)	Shelf Life	3 years

2. **Detection Principle**

Carbon dioxide reacts with hydrazine to form carbonic acid monohydrazide, which discolors redox indicator (crystal violet).

$$CO_2 + N_2H_4 \rightarrow NH_2 \cdot NH \cdot COOH$$

3.

Interferences	Concentration	Result	Comment
Ammonia	Up to 1,000 ppm	No effect	At more than 1,000 ppm gives minus error
Carbon monoxide	Up to 500 ppm	"	
Sulfur dioxide	Up to 30 ppm	"	
Nitrogen dioxide	Up to 30 ppm	"	
Chlorine	Up to 20 ppm	"	

4. 4.1 Standard Gas Generation Methods: Cylinder bottle gas & dynamic gas flow system
4.2 Method of Analysis: Gas chromatography (TCD)

5. **Dangerous & Hazardous Properties**

T.L.V. = 5000 ppm
STEL = 15000 ppm
F.L. =

Concentration	Physical Effects
2.5%	No effect for a few hours
10%	Can endure for a few minutes
12 – 15%	May cause death in exposures for several hours

6. **Physical Constants**

C.F.: CO_2 M.W.: 44.01 S.G.: 1.53 M.P.: -56.6°C B.P.:
AIT: V.P.:

7. Application for other gases

7803

APPENDIX D

LABORATORY CUSTODY SOPs

APPENDIX D-1

ETC

TITLE ETC-Edison Log-In Procedure	Doc.#ED210003
	Revision #3
	Effective
Approval: Lab Manager [signature] 11/2/90	Date 11/1/90
QA Manager [signature] 11/2/90	

1.0 SCOPE AND APPLICATION

1.1 Procedure is intended to assure proper receipt of samples and entry of sample receipt data into lab management system. This process is referred to as "logging in/log-in of samples".

1.2 Samples are received daily (Monday - Saturday) from both commercial carriers and clients. Samples may be received in either ETC Shuttles or in client provided containers. Special arrangements should be made for ETC Saturday receipt situations.

1.3 Upon receipt ETC must maintain custody and must maintain samples at a temperature of 4° +/- 2°C.

1.4 All receipts must be documented in a "loglink" log book. At a minimum the following information is required:

Carrier or courier identity
Client identity
Number of Shuttles (or client provided containers)
ETC sample number(s) received
Date of receipt
If a problem note(s) was required (Yes or No)

1.5 All deficiencies are documented on an "ETC Problem Note" or equivalent (Appendix 9).

1.6 Samples are logged in on an ETC "log-in" form which documents the number and type of bottles received, all order information, comments regarding the samples and any other information requested by the individual work group managers. Log-in forms may be generated through the ETC data base or manually.

1.7 An additional "Log-in form" is used for USEPA-CLP samples, which documents additional pieces of specific USEPA-CLP information for the case file. This form is used with and replaces in certain instances the standard problem note form.

2.0 SAFETY

2.1 Read ETC Safety Manual before proceeding.

2.2 Wear the following while working with any sample

2.2.1 Lab coat

2.2.2 Safety-shield glasses

2.2.3 Gloves, Solvex (Baxter G7197 or equivalent)

2.3 Wear ETC provided OSHA approved respirators as required.

2.4 Use ventilation hoods as required.

3.0 APPARATUS AND MATERIAL

3.1 Pliers

3.2 pH indicator strips, wide range (S/P #P1119-1A, or equivalent)

3.3 Capillary pipette (Kimble #71900, or equivalent)

3.4 Temperature monitoring device, calibrated

3.5 Clean work surface

3.6 Terminal and printer

3.7 Black ball point pen

4.0 PROCEDURE

CAUTION: DO NOT write on any custody document except as indicated on attachments. Use black ball point pens ONLY. These are legal documents which become part of the final report provided to the clients. Do not write on ETC "CC2" custody form.

4.1 Opening ETC Shuttles

4.1.1 Segregate Shuttles and client provided containers by workgroup. Determine work group priority. Notify supervisor if shipping containers cannot be assigned to a work group.

4.1.2 Reconcile number of Shuttles received with the corresponding airbill. If discrepancies exist, alert the Technical Project Manager (TPM) so that proper actions may be taken.

4.1.3 Note status of seal integrity. Open Shuttle by breaking seal with pliers. Remove all custody documents (CC1, CC2, NJDEP and any others provided by clients, Appendix 1, 2, 3). Document time and date of Shuttle opening, Shuttle identification number and

custody seal number on custody form(s). If custody seal was not present write "NONE". Notify TPM and document on Problem Note.

4.1.4 Open unlabeled Shuttles first.

4.1.5 Insert temperature monitor into the Shuttle between the sleeve and the wall. Close the Shuttle. Allow reading to equilibrate.

4.1.6 Review custody form(s) for completeness (signature, date, seal number, etc.). Notify the TPM of all the comments made by the samplers.

4.1.7 Document integrity status of seal on custody form(s).

4.1.8 Reopen Shuttle if necessary to read temperature. Record measurement on custody form(s). Notify TPM if temperature measurement is NOT between 2-10°C and document on Problem Note.

4.1.9 Carefully remove bottles from Shuttle. Match ETC job numbers against information on custody form. Line up bottles by job number. Document nonconformances on Problem Note.

4.1.10 Check and record the condition of each bottle on the custody form(s). Document if headspace is present in TOX and VOA bottles; if bottles are empty or broken, etc. Notify TPM and document nonconformances on Problem Note.

4.1.11 Check pH of all samples which were to be preserved. DO NOT CHECK preserved VOA samples. For each sample remove a small aliquot using a new capillary pipet. Dispense aliquot on pH paper. Acid preserved samples should have a pH <2 and basic preserved samples should have a pH >12. Preservatives that should have been added at the time of sampling are on the CC1 custody form. Note observations on the custody form(s) by placing a check mark (" ") next to the preservative which was to be added. Notify TPM and document nonconformances on Problem Note.

4.1.12 Sign and date custody form(s) and air bills. Bring custody form(s), air bill, any other documentation and Problem Notes to TPM in log link jacket.

4.1.13 If shuttles are received empty.

1. Remove the contents for cleaning.
2. Complete custody form(s) as in Steps 4.1.3, 4.1.7, and 4.1.12.
3. Bring forms to TPM for follow-up action (drywell, cancellation, etc.).

4.2 Opening non-ETC Sample Shipping Containers

4.2.1 For each client identified sample, have TPM assign an ETC job number. Verify that client ID number(s) match custody information. Document discrepancies on a Problem Note.

4.2.2 Label client bottles with ETC Job number, analysis required, bottle number (for example, if 3 aliquots of one sample are received, label 1 of 3, 2 of 3, 3 of 3).

4.2.3 Initiate an ETC "Chain of Custody" form and any others required by clients (Appendix 5).

4.2.4 Proceed with Step 4.1.5.

4.2.5 Refer to Section 4.6 for receipt of USEPA CLP samples.

4.3 Receipt of Client Deliveries

4.3.1 Document receipt in "ETC Client Delivery" logbook. At a minimum record client identity, address and sample description. Logbook is shared by all work groups.

4.3.2 Request that client sign log book as the relinquisher.

4.3.3 Sign as receptor. Record date and time in the appropriate column.

4.3.4 Complete an ETC "Client Chain of Custody" and any forms required by the client (Appendix 6, 4). Provide a copy to the client.

4.3.5 Proceed with Step 4.1.5 or 4.2 as appropriate.

4.4 Generate a CSLIST (optional if automated log-in is unavailable). If automated log-in is available, go to Step 4.5.

4.4.1 Log onto SYSA and access FT CSLIST.

4.4.2 Enter ETC job numbers as requested.

4.4.3 Check A-types on resultant CSLIST against the custody form(s) and bottles received (Appendix 7).

4.4.4 If a loglink number is present, add the new receipt to that link. (NOTE: presence indicates that the sample had previously been received or may be indicative of sampling problems.) Document on Problem Note and describe cause if known.

4.5 Generation of a log-in form (Appendix 8).

4.5.1 Log onto SYSA and access TR ELOGN.

4.5.2 Generate a separate log-in for each individual client, facility and turnaround time. Log-ins are referenced by the assigned loglink number (Step 1.4). Problem Notes must be initiated for all discrepancies.

4.5.3 Record air bill number. If air bill was not received enter "NONE".

4.5.4 Record matrix of each sample.

4.5.5 Record the analyses requested.

4.5.6 Assure that client name matches log-in.

4.5.7 Verify that actual sample matrices match those documented on the log-in. Enter the actual matrix in the appropriate area on the log-in. Use best judgement. Record discrepancies on Problem Note.

4.5.8 Record number of bottles received and analyses required.

4.5.9 Record presence or absence of client custody form(s), EPA or state traffic reports and/or SAS packing lists in comment section.

4.5.10 Document if samples are aliquotted. Aliquotting is necessary if sample bottles are received broken or additional analyses are requested after Shuttle

shipment. Notify TPM before proceeding.

4.5.11 Summarize all problems on Problem Notes in the login comment section.

4.5.12 Secure ETC "Spec" Sheet(s) for sample(s). Record number on log-in.

4.5.13 Review log-in form upon completion. Sign log-in.

4.5.14 Color code (dot) bottle caps with the assigned loglink number.

4.5.15 Samples to be sent to subcontract labs.

1. Identify samples

2. Initiate a subcontract "Request for Analysis" form. This form serves as a custody document. Refer to Appendix 10.

3. Bring a copy of the "Request for Analysis" form to the TPM or designate.

4. Pack samples in an ETC Shuttle or prepare for pickup as appropriate. Include the original "Request for Analysis" form.

4.5.16 Place all samples in designated cold storage area(s).

4.5.17 Link samples into the system by logging on SYSA and accessing TR LOGIN. Enter the following when requested:

Operator ID: [Type first and last name initial and last four digits of social security number]

Edit All N/Y: [N] <R>
R-Date N/Y: [Y] <R>
Due-Date N/Y: [Y] <R>
F-Code N/Y: [N] <R>
Next Loglink: [Type in loglink number] <R>
Next Jobnum: [type in job number] <R>
Red'd Date: [Type in accurate sample receipt (YYMMDD)] <R>
Due-Date: [type in due date from the top left hand corner of the screen] <R>
Up-Date: [Y]

4.5.18 If samples are from NJDEP, initiate "DEP-077" form (Internal Chain of Custody). Refer to SOP SMS01600.ED.

4.5.19 Attach ETC "Spec" sheets and NJDEP "DEP-077" form (s) to log-in. Circulate original log-in and copies as directed by each work group. Hand deliver all EMERG turnaround log-ins directly to the responsible individuals for each analytical cost center. Copies of all log-ins must go to Planning and Project Services in addition to designated work group personnel.

4.6 Receipt of USEPA CLP Samples

4.6.1 A USEPA case is initiated by a telephone call from the Sample Management Office (SMO) to the designated ETC/Edison contact.

4.6.2 The information given by the SMO follows: contract #, bid lot (if applicable), case #, region, ship date(s), number of samples, matrix, level , and parameters.

4.6.3 The work group designated to analyze USEPA CLP samples will be notified; the original documentation of the SMO telephone contact will be retained for inclusion in the case file.

4.6.4 USEPA samples are shipped by overnight courier. At the time of courier delivery, the sample custodian verifies that the number of containers recieved matches the information on the airbill. The sample custodian signs the airbill with the date and time of delivery. The airbill is retained for inclusion in the case file.

4.6.5 Proceed with Sections 4.1.5 through 4.1.13, 4.2.1 through 4.2.3, Sections 4.4 and 4.5, and the following Sections 4.6.6 through 4.6.12.

4.6.6 The unopened ice chests are inspected for the presence or absence of intact custody seal(s), intact strapping tape, or evidence of damage. This information is recorded on the USEPA Traffic Report (TR) and othe documentation.

4.6.7 All samples should be listed on the TR present in the

cooler(s). The TR is completed.

4.6.8 The "USEPA Sample Log-In" form is utilized to document additional pieces of specific information for the case.

4.6.9 If there are any questions or discrepancies noted on the receipt of the samples, the SMO is notified immediately by telephone by the designated ETC/EDison contact or technical project manager. A record of the clarification (which may be attached to the USEPA SAmple Log-In Form) is retained in the case file.

4.6.10 The receipt time and date stated on the airbill is the Verified Time of Sample Receipt (VTSR); all holding times start from this date.

4.6.11 The completed TR is returned to the SMO within 3 calendar days following receipt of the last sample in the sample delivery group. A copy of the TR is retained for the case file.

4.6.12 The empty ice chest is shipped back to the return address indicated within 14 days of case reciept at the facility.

5.0 SUPPLEMENTAL DOCUMENTS

5.1 SMS01600.ED - SOP for NJDEP Sample/Extract Internal Custody

5.2 GES00600.ED - SOP for Assignment of Log-Link Numbers at Edison.

6.0 REFERENCES

- NJDEP Contract X-408.
- USEPA CLP SOW for Organics, 2/88.

7.0 SUBSTANTIVE REVISIONS

- Revision 3 includes detail regarding receipt of USEPA CLP samples.

APPENDICES

Appendix 1 - ETC "CC1" form
Appendix 2 - ETC "CC2" form
Appendix 3 - NJDEP "DEP-060" form
Appendix 4 - NJDEP "DEP-009" form
Appendix 5 - ETC "Chain of Custody" form for non-ETC sample containers
Appendix 6 - ETC "Client Chain of Custody" for receipt of client deliveries
Appendix 7 - CSLIST report
Appendix 8 - ETC "Log-In" form
Appendix 9 - ETC "Problem Note"
Appendix 10 - ETC subcontract "Request for Analysis"
Appendix 11 - USEPA Traffic Report
Appendix 12 - ETC "USEPA Sample Log-In" Form.

APPENDICES

APPENDIX 1

ETC Environmental Testing and Certification Corp.

CHAIN OF CUSTODY FORM (CC1)

ORIGINAL

Seal No. 175719 ETC Job # HA3064

Date Sealed 90/09/18 By: AS

Company: X Attn.: X

Facility/Site: X Phone: X

Address: X

SAMPLE IDENTIFICATION

Facility: [redacted] (Facility/Site Code) (Optional Sample Point Descriptions)

Sample Point: [X] - DUPLICATE | 90/09/26 | 1415 | | |

Source Code (from below): X — Your Sample Point ID (left justify): DUPLICATE — Start Date (YY/MM/DD): 90/09/26 — Start Time (2400 hr clock): 1415 — Elapsed Hours (composite):

10/4/90

Source Codes:
Well (W) Outfall (O) Bottom Sediment (B) Surface Impoundment (I) Leachate Collection Sys. (C) Other (X)
Soil (S) River/Stream (R) Generation Point (G) Treatment Facility (T) Lake/Ocean (L) Specify

SHUTTLE CONTENTS

BOTTLE				ANALYSIS	SAMPLER		LAB
Io	Type	Size	Preserv.		Filt. (Y/N)	Observations	Observations
2	UVOA	40	NONE	HPT/VOLATILES	N		✓
2	VOA	40	HCL	PT/VOLATILES	N		✓
1	VS	40	HCL	VOA SCREEN	N		✓ (4.1.10)
1	UTB	40	GC/MS	UNPRES TB	N		✓ (4.1.11)
1	TB	40	GC/MS	TRIP BLANK	N		✓
4	EXT	1000	NONE	Extractables	N		✓
1	MET	1000	✓ HNO3	METALS	N		✓
2	SULF	125	✓ Z/NAOH	SULFIDES	N		✓
4.1.11							

CHAIN OF CUSTODY CHRONICLE

1. Shuttle Opened By: (print) SAMPLER Date: 9/19/90 Time: 1500
Signature: Sampler Seal #: 175719 Intact: yes

2. I have received these materials in good condition from the above person.
Name: Signature: SEAL # MUST MATCH
Date: Time: Remarks:

3. I have received these materials in good condition from the above person.
Name: Signature:
Date: Time: Remarks:

4. Shuttle Sealed By: (print) SAMPLER Date: 9/26/90 Time: 1435
Signature: Sampler Seal #: 0175720 Intact: yes

LAB USE ONLY Opened By: (4.1.12) Date: (4.1.3) Time: (4.1.3)
SHUTTLE # (4.1.5) TEMP. °C (4.1.8) SEAL # 175720 COND. (4.1.7)

APPENDIX 2

ETC

FIELD PARAMETER FORM (CC2)

Form 0002
Sample Management
12/89

ETC JOB # HA3064

Sample Point W DUPLICATE
Source Code — Sample Point I.D.

FIELD PROCEDURES

PURGE DATE (YY MM DD)	START PURGE (2400 Hr Clock)	ELAPSED HRS	WATER VOL IN CASING (Gallons)	VOLUME PURGED (Gallons)
900926	0905	5	4.94	450

SAMPLING METHOD: Stainless Steel Bailer

Sampler Type [E] A-Submersible Pump, B-ISCO, C-Bladder Pump, D-Dipper/Bottle, E-Bailer, F-Scoop/Shovel, X-Other _____ (SPECIFY OTHER)

Sampler Material [B] A-Teflon, B-Metal, C-PVC, D-Plastic, X-Other _____ (SPECIFY OTHER)

Tubing Material [ ] A-Teflon, B-Tygon, C-Polyethylene, D-Silicon, X-Other _____ (SPECIFY OTHER)

Sample Composited [Y/N] NO
Procedure/Proportions

FIELD MEASUREMENTS

Well Elevation (ft/msl) [ ]
Depth to Ground water (ft) [ ]
Groundwater Elevation (ft msl) [ ]
Well Depth (ft) [ ]
Sample Depth (non-well) (ft) [ ]

	ph	(STD)		spec. cond.	um/cm at 25° C	(other parameter)	value	units
1st			1st					
2nd			2nd					
3rd			3rd					
4th			4th					

Sample Temp [ ] (°C)
Turbidity [ ] NTU

FIELD COMMENTS

Sample Appearance: cloudy
Weather Conditions:
Other:

FILTERING: Use Chain of Custody (CC1) to indicate which bottles were filtered

Sampler: [redacted] (Print) Employer: [redacted]

I certify that sampling procedures were in accordance with applicable EPA state and corporate protocols.

9/26/90 [redacted]
(Date) (Signature)

ORIGINAL

APPENDIX 3

Form DEP-060
10/86

NEW JERSEY DEPARTMENT OF ENVIRONMENTAL PROTECTION

CHAIN OF CUSTODY RECORD WITH SHUTTLE

Use One Form for Each Sample — *Shaded Areas for DEP Field Use Only*

NAME OF UNIT AND ADDRESS: NJDEP

Name of Person(s) Preparing Container(s) for Sample Shuttle

Name [redacted] Title Sample Manager Agency ETC

Name Title Agency

Date above-mentioned Person(s) Placed Container(s) in Sample Shuttle: 90/10/16 | Military Time: 1520 | Laboratory Affixed Sample Shuttle Seal No. 0185429

Name of Person(s) Breaking Laboratory Seal on Sample Shuttle and Taking Field Sample

Name [redacted] Title Sr Env Specialist

Name [redacted] Title Env. Specialist

Name Title

Date Laboratory Sample Shuttle Seal No. 0185429 Broken 10/17/90 | Time (Mil) Seal Broken and Sampling Begun 0855

FIELD SAMPLE NUMBER	CONTAINER NUMBER(S)	VOLUME OF CONTAINERS	DESCRIPTION OF SAMPLE
BTR-042	2	40-ml VOA	
	2	500 ml EXT	
	2	125 ml	
	1	250 ml	
	1	40 ml TRIPBLANK	

LAB CONTROL NO.
HA3299 - Prior. Poll.
HA3302 - EPTOX

Date Sampling Completed & Field Seal Affixed to Sample Shuttle: 10/17/90 | Military Time: 1100 | Field Affixed Sample Shuttle Seal No. 0185428

Laboratory Person Breaking Field Seal on Sample Shuttle & Accepting Responsibility for Sample

Name (4.1.12) Title (4.1.12)

(4.1.3) Field Sample Seal No. 185428 | (4.1.3) Date Broken 10/17/90 | (4.1.3) Military Time Seal Broken 1430

SAMPLE RELINQUISHED BY	SAMPLE RECEIVED BY	DATE	TIME	REASON FOR CHANGE OF CUSTODY
Laurie T. Seekinger	To ETC Shuttle	10/17/90	1100	
	[redacted]	10/17/90	~~2330~~ 1430	
	(4.1.12)	(4.1.3)	(4.1.3)	

COPIES: *Gold - Container/Shuttle Preparation Receipt* *Pink - Field Sampler Receipt* *Yellow - Analyst Chain of Custody* *White - Sample Custodians Chain of Custody*

APPENDIX 4

Form DEP-009
10/86

STATE OF NEW JERSEY
DEPARTMENT OF ENVIRONMENTAL PROTECTION
TRENTON, NEW JERSEY 08625

CHAIN OF CUSTODY WITHOUT SHUTTLE

I.D. # _____

NAME OF UNIT AND ADDRESS:

CLIENT NAME AND ADDRESS

SAMPLE NUMBER	Number of Containers	DESCRIPTION OF SAMPLES
CLIENT SAMPLE POINT ID	AS REQUIRED	MATRIX, DESCRIPTION OF CONTAINERS ETR JOB #

PERSON ASSUMING RESPONSIBILITY FOR SAMPLE: | TIME | DATE

SAMPLE NUMBER	RELINQUISHED BY:	RECEIVED BY:	TIME	DATE	REASON FOR CHANGE OF CUSTODY
CLIENT	CLIENT	ETR	AR	AR	DELIVERY TO LAB
SAMPLE	SIGNATURE	SIGNATURE			
POINT ID					

APPENDIX 5

ETC

100614

CHAIN OF CUSTODY

Company: NJDEP Job # _____

Address: TRENTON, NJ

Attention: _____

Sample Description:

CUSTOMER ID	DESCRIPTION	ETC #
Field Blank	Field Blank	#A3304
	(1) 1L Metal (1) 125CY (1) 125 Phenol	
	(2) 1L EXTBL (2) 40ml VOA	
Trip Blank	(1) 40ml. TB	#A3301

Sample(s) Relinquished by: (CLIENT SIGNATURE / PRINTED NAME)

Time: (AS REQUIRED) Date: (AS REQUIRED)

Sample(s) Received by: (SAMPLE MANAGER)

Time: 230 Date: 10/17/96

721
178928

APPENDIX 6

ETC

CLIENT CHAIN OF CUSTODY

COMPANY: ETC Corporation
284 Raritan Center Parkway
Edison, NJ 08818

Samples Relinquished by: _____

Samples Received by: _____

Time: _____

Number of Shuttles: _____

APPENDIX 7

(4.4.4)

JOB#: BD4445 XRND: NORM COMPANY:
LOG#: FACILITY:

/PROGRAM ELEMENT 2a/ /SPECVOA/DM/RP--REPORT ONLY--BENZENE, METHYLENE CHLORIDE,/ /TETRACHLOROETHYLENE, TRICHLOROETHYLENE, TOLUENE,/ /1,1,1 TRICHLOROETHANE, MEK AND XYLENES ON QR01 AND/ /DM-1HC, KEN BAKER TO REVIEW PRIOR TO MAILING/ /FIELD/MISC.-STICKUP/

ABRP	ETCL/
DM-1HC	ETCL/
DMPKG	ETCL/
SPECDM	ETCL/
SPECRP	ETCL/
FIELD/TEMP	ETCL/
FIELD/DTGW	ETCL/
FIELD/GWE	ETCL/
FIELD/MISC.	ETCL/
FIELD/ELEV.	ETCL/
FIELD/WELD	ETCL/
1PP/VOA	ETCL/
SPECVOA	ETCL/
1SU/XVOA	ETCL/
XVOA2	ETCL/
XVOA17	ETCL/
SUBOTTL	ETCL/GCLL
1GWP3/Q/FIELD	ETCL/
1GWP2	ETCL/
FIELD/PH	ETCL/
FIELD/SCOND	ETCL/
1TOC4	ETCL/
1TOX4	ETCL/
FIELD/DENTRY	ETCL/
1CHLOR	ETCL/GCLL
1GW/MET/P2	ETCL/GCLL
1PHENOLS	ETCL/GCLL
1SO4	ETCL/GCLL

APPENDIX 8

ETC LOGIN SHEET

(4.5.11)

Company Name
(4.5.6)

Loglink
(4.5.2)

Facility
(4.5.2)

T/A Date received
(4.5.2)

Shipper # of shuttles
(4.5.3)

Airbill #
(4.5.3)

Revision

10/9, - REVISED A-TYPE, AND COMMENT FOR HA3062, 64

Order Entry Comments

HA3062-SPECVOA: 7 DAY HOLDING TIME FOR HPT/VOLATILES.
HA3062-SPECVOA/ACID/BN/GC/AA/CV: TIER I DELIVERABLES REQ'D.
HA3062-SPECSUB: RLEASE PROVIDE ALL RAW DATA AND QC INFORMATION
HA3062-WITH DATA PACKAGE.
HA3064-SPECVOA: 7 DAY HOLDING TIME FOR HPT/VOLATILES.
HA3064-SPECVOA/ACID/BN/GC/AA/CV: TIER I DELIVERABLES REQ'D.
HA3064-SPECSUB: PLEASE PROVIDE ALL RAW DATA AND QC INFORMATION
HA3064-WITH DATA PACKAGE.
HA3062, 3064, - SPECCV.: PLEASE ALIQUOT FROM EXTRACTABLE BOTTLE FOR PH/SCOND.

(4.5.4)

Sample Mgmt Comments

(4.5.9)
(4.5.10)
(4.5.11)

Cr+6 transferred to sub-lab from field.

Problem note #: Yes

(4.5.5)

Shuttle #	Temp	Sample Id	Sample Date	Job#				
999	7	BF99			1AP9/HPTVOA	AP05/	1AP9/PEST(EC)	AP05/
103	6		09/26/90	HA3062	1AP9/PEST(FPD)	AP05/	1AP9/PCB(EC)	AP05/
					1AP9/MET	AP05/	1AP9/SULFIDE	AP05/CHYN
					1AP9/ACID	AP05/	1AP9/BN	AP05/
					1AP9/CY	AP05/	1AP9/HCP	AP05/
					1AP9/PTVOA	AP05/	1AP9/HERB(EC)	AP05/
					1CR(+6)	AP05/CHYN	SPECVOA	AP05/
					SPECVOA	AP05/	SPECACID	AP05/
					SPECBN	AP05/	SPECGC	AP05/
					SPECAA	AP05/	SPECCV	AP05/
					SPECSUB	AP05/CHYN	1 PH	
					(4.5.15)		1 SCOND	*
							SPECCV	
773	9	DUPLICATE			See Job HA3062			
348	8		09/26/90	HA3064	*			

(4.5.8)

Sample Type			
H2O	~~CR+6(1)~~ PH 500	CY/T(2) 500	Extractabl(8) 1000
	HPT/VOLATI(2) 40	METALS(1) 1000	PT/VOLATIL(2) 40
	SULFIDES(2) 125	TRIP BLANK(1) 40	UNPRES TB(1) 40
	VOA SCREEN(1) 40		
H2O	~~CR+6(1)~~ PH 500	CY/T(2) 500	Extractabl(8) 1000
	HPT/VOLATI(2) 40	METALS(1) 1000	PT/VOLATIL(2) 40
	SULFIDES(2) 125	TRIP BLANK(1) 40	UNPRES TB(1) 40
	VOA SCREEN(1) 40		

APPENDIX 9

100646

PROBLEM LOG

TODAY'S DATE 9/27/901

COMPANY NAME

FACILITY-CODE

PS REP

DATE SUBMITTED 9/27

PROBLEM DESCRIPTION	CHECK	JOB NUMBER/A-TYPES
1. SMPLE PT - UNAPPROVED/OMITTED	✓	LINKED NOT UPDATED
2. SOURCE CODE - OMITTED		fac. code/sample pts. approved.
3. START DATE - OMITTED		Need Chyon C of C
4. SMPL BOTLS REC'D EMPTY A-TYPES		[illegible] 9/27
5. INCOMPLETE SPECS		
6. MISMATCHING JOB# S (CC1VSC2)		
7. METALS COMMENTS		
8. MISMATCHING SMPLE ID'S (CC1VSCC2)		
9. MISMATCHING FAC-CODES (CC1VSORDER)		
10. REC'D EMPTY (ALL) DRYWELL/OTHER		
11. FIELD DATA-OMITTED/INCOMPLETE		

MISSING:

A.FIELD/PH	F.FIELD/ELEV
B.FIELD/SCOND	G.FIELD/WELD
C.FIELD/TEMP	H.FIELD/SAMPO
D.FIELD/DTGW	I.FIELD/MISC.
E.FIELD/GWE	J.OTHER

12. LOCATION CODES

13. OTHER

REVIEWED BY: [signature] DATE. 9/27/90

RESOLVED BY: [signature] DATE. 10/4/90

JACKET COMPLETED BY: DATE.

APPENDIX 10

OHM Corporation
Environmental Testing and Consultation Corp.

Request for Subcontract Analysis and Sample Chain-of-Custody

Name of Subcontract Laboratory: _____

Loglink(s): 100646 Workgroup: APD5

Matrix: H_2O Sampling date(s): 9/26/90

ETC Sample ID Numbers: HA3062 (2)
HA3064 (2)

Turnaround is 14 days unless otherwise indicated. _____
(If deadline can not be met, contact ETC Subcontract Department - (201)225-6700
Send report and invoice to ETC Corp. P.O.Box 7808, Edison, NJ 08818-7808
Attention: Subcontract Department

The following analyses are requested:

ACIDITY	CY/T	SOLIDS/SETL
ALKA	FLUORIDE	SOLIDS/TS
NH3	FORM/UV	SOLIDS/TV
NH3/D	HARD	SO4
ASBESTOS/EM	NO3	✓ SULFIDE
ASBESTOS/LM	NO2	SO3
BICARB	N2/ORG	SURFAC
BOD	N2/TK	TOX
BROM	ODOR	TOX2
BTU	O+G/GRAV	TOX4
CARB	O+G/IR	TOC2
CA/CACO3	PETHY/GRAV	TOC4
CHLOR	PETHY/IR	TURB
CL2/T	PHENOLS	DW1/ABO
CL2/D	PHENOLS/DL	DW1/RA226
CL2/R	PO4/T	DW2/RA228
COD	PO4/ORG	AP9/SULFIDE
COLI/F	PO4/ORT	AP9/CY
COLI/T	PHOS	COMPCV
COLOR/S	CY/REACT	FILT
COLOR/A	SULFIDE/REACT	HOMO
CR(+6)	SOLIDS/T	ECRA/PETHYIR
CY(CHLOR)	SOLIDS/TD	

Others: spec sub please provide all Raw DATA + QC information with data package

Chain-of-custody (complete appropriate section)

Option A: Courier pickup at ETC
Sample(s) relinquished by ETC: X
Time: 200 Date: 9/27/90
Sample(s) received by: X
Time: 1430 Date: 9/27/90

Option B: Sending sample from ETC
Shuttle sealed at ETC by: _____
Date: _____ Time: _____ Seal Number: _____
Shuttle opened by: _____
Date: _____ Time: _____ Seal Number: _____
Seal intact? yes / no Shuttle contents in good condition? yes / no

Comments: _____

Sub form 0007.01

(4.5.15)

APPENDIX 11

EPA

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Organic Traffic Report
(For CLP Use Only)

Case Number | RAS No.

1. Sample Description *(Enter in Column A)*

1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) *(Specify)*

2. Region Number | Sampling Co.

Sampler (Name)

3. Ship To:

4. Date Shipped | Airbill Number

Carrier

Triple volume required for matrix spike/duplicate aqueous sample.

Ship medium and high concentration samples in paint cans.

See reverse for additional instructions.

5. Date Received | Received by

Laboratory Contract Number | Unit Price

6. Transfer to | Date Received

Received by

Contract Number | Price

CLP Sample Number (From labels)	(A) Sample Description (From box 1)	(B) Concentration L=low M=med H=high	(C) RAS Analysis VOA	BNA	Pest/PCB	(D) Special Handling	(E) Station Location	(F) Date/Time of Sample Collection	(G) Corresponding CLP Inorganic Sample Number	(H) Sample Condition on Receipt	(I) High Conc. Phases (Check below) Solid	Water—MIS Liq.	Non Water—MIS Liq.

FIGURE 2.

APPENDIX 12

ETC

USEPA SAMPLE LOG-IN

Case #: _____

Received Date: _____

Sample Manager/Date: _____/_____

DIRECTIONS: Circle the appropriate answer and complete all blanks.

1. EPA Chain of Custody Form is present for all samples. YES NO
2. Airbills present for all containers received. YES NO
3. Traffic reports/SAS packing list present. YES NO
4. Custody seals present on shipping containers. YES NO
5. Custody seals present on sample containers. YES NO
6. Sample tags present for all samples. YES NO
7. List any sample ID tags not included on chain of custody records:

   _____ _____ _____ _____

   _____ _____ _____ _____

8. Condition of shipping containers: INTACT NOT INTACT
9. Condition of sample containers: INTACT NOT INTACT
10. Receiving documents are in agreement. YES NO
11. Problem numbers discussed with Sample Management Officer:

    _____

    _____

    _____

    _____

SMO Contacted By: _____

Name of SMO Contact: _____

Date: _____

Rev. 10/90

TITLE Internal Sample Tracking	Doc.#ED222000
Approval: Lab Manager Gregory D. Morrison QA Manager Lee B. Randolph 11/2/90	Revision # 0 Effective Date 11/1/90

1.0 SCOPE AND APPLICATION

1.1 The purpose of this SOP is to describe the mechanisms for internal sample tracking at ETC/EDISON from receipt to final analysis and to include examples of laboratory documents.

2.0 SUMMARY

2.1 Sample tracking begins at time of receipt, when a hardcopy login form is generated and the sample is linked into ETC's computer system for scheduling and tracking. Sample are placed in a secured storage area by the sample manager as part of the log-in process. The samples are accessed by authorized laboratory personnel, utilizing laboratory chronicles to document changes in custody.

2.2 Designated schedulers utilize log-in Forms and computer-generated reports to prepare analytical batches, which are processed by appropriate laboratory personnel. As work progresses on each sample, changes in status are routed through schedulers, who update the computer system. Information from the computer system is available in both hardcopy and screen versions, and is used by ETC personnel to track the status of samples in the laboratory.

2.3 When all analytical work has been completed on a sample, the data batch is routed to the scheduler or other designated person within each work area and the computer system is updated.

2.4 Any remaining sample and/or extract is held in a secured storage area until at least 30 days after the issuance of the technical report. At the end of 30 days, the sample is disposed of. ETC retains samples and/or extracts samples for longer periods of time in those cases where it is requested by the client or is contractually required.

3.0 Safety

3.1 All appropriate safety precautions as described in the ETC Safety Manual should be used when handling samples.

4.0 APPARATUS AND MATERIALS

4.1 Computer terminal with access to

4.1.1 AMSTAT

4.1.2 CSO5AM

4.2 Laboratory Chronicles

5.0 PROCEDURES

5.1 Samples are received at ETC in the sample management area. The sample manager logs the sample in utilizing the procedures described in SOP# ED21003 ETC/EDISON Log-in Procedure. At the time of login, a hardcopy log-in form is generated and circulated to appropriate personnel, including group schedulers, and the samples are linked into ETC's computer system. The samples are placed in a secured storage area.

5.2 The schedulers utilize the log-in forms and other computer generated reports to prepare and schedule analytical batches. The batch sheets are routed to appropriate personnel for processing. The samples on the batch sheets are retrieved by analysts and/or technicians from the secured storage areas. Changes in custody are documented by the use of laboratory chronicles, as described below:

5.2.1 Sample Preparation Chronicle

The analyst who signs under "set-up" claims custody of the sample from sample storage to the laboratory. The verification of the supervisor's signature represents the completion of analysis and subsequent transfer of the remaining sample back to sample storage, and of the extract to the extract storage refrigerator.

5.2.2 GC/MS Laboratory Chronicle

- Volatiles - The analyst who signs the analysis chronicle claims custody of the sample from sample storage to the laboratory. The supervisor's signature represents the completion of analysis and transfer of the sample from the laboratory back to the volatile refrigerator.

- Extractables - The analyst who signs the analysis chronicle claims custody of the extract from the extract refrigerator to the laboratory. The supervisor's signature represents the completion of analysis and the transfer of the remaining extract back to extract storage.

5.2.3 GC and HPLC Analysis Chronicle

The signature of the analyst represents custody of the extract in the laboratory. The supervisor's signature represents the completion of analysis and transfer of remaining extract back to extract storage.

5.2.4 Metals Laboratory Chronicle

The chemist who signs for preparation is responsible for the transfer of the sample from initial storage to the laboratory. The supervisor's signature represents the completion of sample analysis and transfer of remaining sample back to initial sample storage.

5.2.5 Conventionals Laboratory Chronicle

The analyst who signs on the analysis log form claims custody of the sample from initial storage to the laboratory. The signature of the supervisor represents completion of analysis and transfer of remaining sample back to storage.

5.2.6 Percent Moisture Worksheet

The Form may be used independent of the Sample Preparation Chronicle . The analyst who signs on the form claims custody of the sample from initial storage to the laboratory. The signature of the supervisor represents

completion of analysis and transfer of remaining sample back to storage.

5.3 As the sample progresses through the laboratory, changes in status are routed through group schedulers, who update the computer system as needed.

5.3.1 When sample preparation re-work is required for semi-volatile organic analysis, a "Rework Required Form for Sample Preparation" is initiated and the sample is rescheduled for the re-extraction or other rework specified.

5.4 When all analytical work has been completed on a sample, the data batch is submitted to the group scheduler or designate. This person updates the computer system, and transfers the batch on to the Report Production area for processing into technical reports. Issued technical reports are stored in a secured area on-site for a period of up to 1 year then transferred to an off-site archive.

5.4.1 USEPA CLP Samples technical reports are purged with the case file as contractually required.

5.5 Any remaining samples and/or extracts are stored in secured areas for at least 30 days after the issuance of technical reports, or for as long as requested by the client or contractually required. After this period, the sample is disposed of as described in SOP# SMS00501.ED.

6.0 QUALITY CONTROL

7.0 APPENDICES

7.1 References

7.2 Supplemental Document

7.3 Quality Control Criteria

APPENDIX

7.0 APPENDICES

7.1 References

7.1.1 ED21003 - SOP for ETC/EDISON Log-In Procedure

7.1.2 SMS00501.ED - SOP for Sample Disposal at EDISON.

7.2 Supplemental Documents

7.2.1 ETC login form

7.2.2 Copy of page from CS05 showing various status' of samples

7.2.3 Copies of laboratory chronicles

7.2.4 Copy of status change request form from the laboratory

7.2.5 Copy of Sample Preparation Rework Form

7.2.6 Copy of computer generated GC/MS schedule

7.3 Quality Control Limits

7.3.1 Not Applicable

SUPPLEMENTAL DOCUMENTS

ETC LOGIN FORM

ETC LOGIN SHEET

Company Name

Loglink

Facility

T/A Date received

Shipper # of shuttles

Airbill #

Revision

Order Entry Comments

Sample Mgmt Comments

Problem note #:_____

Shuttle #	Temp	Sample Id	Sample Date	Job#		Sample Type				

COPY OF PAGE FROM CS05 SHOWING VARIOUS STATUS' OF SAMPLES

ALL atypes, ALL costcenter(s)
Sorted by: DUEDATE, ACCOUNT, JOBNUMBER

Lab Sample Control Report (CS05A0)

Data last updated: 90/11/02 at 05:41

Job No. <Bkjob> Facility	LogLnk JobLnk SmpPnt	Xrnd Team	DueDate RecDate Price	Hold Date	Src/Sent Location	Atype	QaBatch	SP	MS	GC	AA	CV	PR	DM
Acctid: [redacted]														
CA5090-02	100704	DA14	90/11/09	00/00/00	CLP1/	2IFB/PST/PCB		****	****				****	
	35507	WS/UA	(90/10/26)	90/11/04	CLP1/	2IFB/PST	QG70315	D1031		H1031			NEED	
[redacted]	SR-28(1-2)		[redacted]	00/00/00	CLP1/	IFB/PH/M	QG70315	D1031						
				90/11/04	CLP1/	2IFB/PCB	QG70315	D1031		H1031			NEED	
				00/00/00	CLP1/	SPECGC	QG70315			H1031			NEED	
				00/00/00	CLP1/	SPECRP							NEED	
/SPECGC/RP: REPORT ONLY PCB'S./ /** 14-DAY VERBAL, 21-DAY HARDCOPY./** [redacted]														
CA5096-03	100704	DA14	90/11/09	00/00/00	CLP1/	1IFB/PST/PCB		****		****			****	
	35507	WS/UA	(90/10/26)	90/10/29	CLP1/	1IFB/PCB	QG70316	D1031		H1031			NEED	
[redacted]	FIELDBLANK		[redacted]	90/10/29	CLP1/	1IFB/PST	QG70316	D1031		H1031			NEED	
				00/00/00	CLP1/	SPECGC	QG70316			H1031			NEED	
				00/00/00	CLP1/	SPECRP							NEED	
/SPECGC/RP: REPORT ONLY PCB'S./ /** 14-DAY VERBAL, 21-DAY HARDCOPY./** [redacted]														
CA5097-03	100704	DA14	90/11/09	00/00/00	CLP1/	1IFB/PST/PCB		****		****			****	
	35507	WS/UA	(90/10/26)	90/10/30	CLP1/	1IFB/PCB	QG70316	D1031		H1031			NEED	
[redacted]	FIELDBLANK		[redacted]	90/10/30	CLP1/	1IFB/PST	QG70316	D1031		H1031			NEED	
				00/00/00	CLP1/	SPECGC	QG70316			H1031			NEED	
				00/00/00	CLP1/	SPECRP							NEED	
/SPECGC/RP: REPORT ONLY PCB'S./ /** 14-DAY VERBAL, 21-DAY HARDCOPY./** [redacted]														
---------- CWMRESOLVE Facility Total: 3 [redacted] ----------														
Account Daily Total: 3 [redacted]														
Daily Totals for 90/11/09			Jobcount: 3		[redacted]			0	0	9	0	0	12	0

A-acquire, C-cancel, D-done, H-here, B-BR, R-redo, S-sent, W-work in process

COPIES OF LABORATORY CHRONICLES

ETC CORP. LAB CHRONICLE: Sample Preparation	QC BATCH NO:

JOB NO.	LOGLINK	Ver. Surr.	SAMPLE AMT ml gm	MOIST %	DRY WGHT gm	EXTRACT VOL (ml)			Ver. Spike	REPEAT		ATYPES
										ORIGINAL JOB NO.	ORIGINAL QS. NO.	
QS	VERIFY SPIKE											
S1												
S2												
MS1												
MSD1												
MS2												
MSD2												

COMMENTS:

PROCEDURE	SIGNATURE	DATE
☐ CONTINUOUS		
☐ SEP. FUNNEL		
☐ SOXHLET		
☐ SONIFICATION		
☐ DILUTION		
☐ DERIVATION		
☐ CLEANUP		
☐ SETUP		
☐ CONCENTRATION		
☐ SUPERVISOR		

SPIKE	VOL ml	CONC ug/ml	LOT NUMBER
SURROGATES	**VOL ml**	**CONC ug/ml**	**LOT NUMBER**

MATRIX
- ☐ 1 WATER
- ☐ 2 SOIL
- ☐ 3 COMPLEX
- ☐ 4 ORGANIC LIQ.

UPDATES
- ☐ QS
- ☐ WIP
- ☐ DONE

EHD: ____________

ETC CORP.

PERCENT MOISTURE WORKSHEET

$$\%M = \frac{\text{WET SOIL WEIGHT} - \text{DRY SOIL WEIGHT}}{\text{WET SOIL WEIGHT}} \times 100$$

$$\text{DRY WEIGHT} = 1 - \frac{\%M}{100} \times \text{WET SOIL WEIGHT}$$

$$\%\text{SOLIDS} = (1 - \%M) \times 100$$

QC BATCH: _____

SAMPLE NUMBER	LOG LINK	CRUCIBLE WEIGHT (g)	CRUCIBLE & WET SOIL WEIGHT (g)	WET SOIL WEIGHT (g)	CRUCIBLE & DRY SOIL WEIGHT (g)	DRY SOIL WEIGHT (g)	PERCENT MOISTURE (%)	PERCENT SOLIDS (%)
-D								

COMMENTS: _____

PERCENT MOISTURE SET-UP: _____ DATE: _____

PERCENT MOISTURE COMPLETION: _____ DATE: _____

SUPERVISOR SIGNATURE: _____ DATE: _____

ETC Corp. LABORATORY CHRONICLE : GC-MS Department

Date_____ Instr._____

A-Type_____
Tune file_____
Seq. file_____ ,_____
Method file_____
ID file _____
CB file _____
Analyst_____
Reviewed by/Date_____

Batch #'s _____

__ Standards Updated_____
Date_____By_____

Tape # _____Inj.___ul

Standard	Conc ppm	Lot No.	Lot Vol.

R	NAME	DATA File	init. ml/g	ALS #	Dil	Inj. Time	Comments	+ P

R : redo , P : Aroclor search , + : Plus search

ETC Corp. LABORATORY CHRONICLE : GC Department 57 Page___of___

Date_____ Instr._____

A-Type_____
Column_____
Col. Lot_____
Calib.File_____
Seq. File_____ ,_____
Method file_____
ID file_____
Configu. file_____
Analyst_____
Reviewed by/Date_____
Batch #'s

_____

_____Standard Updated_____
Date_____By_____

Tape #_____ Inj.___ul

Standard	Conc. ppm	Lot No.	Standard	Conc. ppm	Lot No.

R	NAME	Data File Detec.1	Detec.2	init. ml/g	ALS #	Dil	Comments

R : redo

ETC Corp. LABORATORY CHRONICLE : LC Department 3 Page___of___

Date_____ Instr._____

A-Type_____

Seq. file_____ ,_____
Method file_____
ID file _____
CB file _____
Analyst_____
Reviewed by/Date_____

Batch #'s _____

__ Standards Updated_____
Date_____By_____

Tape # _____Inj.___ul

Standard	Conc ppm	Lot No:

R	NAME	DATA File	init. ml/g	ALS #	Dil	Comments

ETC

ETC LOGLINK _____

ETC CORP.

METALS DEPARTMENT
LABORATORY CHRONICLE

SAMPLES

_____

_____

_____

_____

	CHEMIST	DATE
Hg Prep		
AA Prep		
ICAP Prep		
Boron Prep		

SUPERVISOR SIGNATURE _____ DATE _____

ETC

TOTAL PHENOLICS

QW-Batch: _____ Method Ref: EPA 420.2, SW-846 9066 Page _____ of ____

QC-Batch: _____ Colorimetric, Auto-4AAP, w/Distillation Date: _____

Verified: _____ MDL: 0.050 mg/l Time: _____

Instrument: _____ Matrix: Aqueous Analyst: _____

Seq #	ETC Job #	pH Int.	Analyzed mg/l	Dilution Factor	Reported mg/l	Comments Calculations

COPY OF STATUS CHANGE REQUEST FORM FROM THE LABORATORY

ETC CORPORATION

CLP1 UPDATE FORM

BATCH #: _____ DATE: _____

SAMPLE #'S OR ATTACHED CHRONICLE: _____

_____

COST CENTER:
SP [ ] MS [ ] GC [ ] AA [ ] CV [ ] PR [ ]

STATUS: HERE [ ] ACQ [ ] WIP [ ] DONE [ ] BATCH [ ]

ATYPE:

IFB/VOA, IFB/VO+1	[ ]	ECRA/VOA, ECRA/VO+15	[ ]
IFB/BNA, IFB/BNA+2 OR 20	[ ]	ECRA/BNA, ECRA/BNA+25	[ ]
IFB/PST	[ ]	ECRA/PST(GC)	[ ]
IFB/PCB	[ ]	ECRA/PCB(GC)	[ ]
IFB/MET	[ ]	ECRA/MET	[ ]
IFB/CVAP	[ ]	ECRA/CN	[ ]
IFB/FLAME	[ ]	ECRA/PN	[ ]
IFB/FURNACE	[ ]	NJTSK/VOA, NJTSK/VO+15	[ ]
IFB/ICAP	[ ]	NJTSK/BN, NJTSK/BN+15	[ ]
IFB/CN	[ ]	NJTSK/ACID, NJTSK/AC+1	[ ]
PP/VOA	[ ]	NJTSK/PST(GC)	[ ]
PP/BN	[ ]	NJTSK/PCB(GC)	[ ]
PP/ACID	[ ]	NJTSK/MET	[ ]
PP/PST	[ ]	NJTSK/CN OR NJTSK/CY	[ ]
PP/PCB	[ ]	NJTSK/PN	[ ]
O&PST/PCB	[ ]	AP__/PTVOA	[ ]
PP/MET	[ ]	AP__/HPTVOA	[ ]
RC/MET	[ ]	AP__/ACID	[ ]
EPTX/MET	[ ]	AP__/BN	[ ]
TCLP/MET	[ ]	AP__/HCP	[ ]
TAL/MET	[ ]	AP__/PST(EC)	[ ]
M601	[ ]	AP__/PCB(EC)	[ ]
M602	[ ]	AP__/PST(FPD)	[ ]
M618	[ ]	AP__/HERB	[ ]
SPEC___	[ ]	AP__/MET	[ ]

OTHER: _____

SUBMITTED BY: _____

UPDATED BY: _____ DATE: _____

COPY OF SAMPLE PREPARATION REWORK FORM

ETC

CLP 1 WORK GROUP

REWORK REQUIRED FORM FOR SAMPLE PREPARATION
Q__ BATCH NO._____

SAMPLE NO.	FRACTION	REASON (Refer to Key)

KEY:

(1) LOW/NO PEST SURR
(2) LOW/NO BN SURR
(3) LOW/NO ACID SURR
(4) HIGH PEST SURR
(5) HIGH BN SURR
(6) HIGH ACID SURR
(7) MS,MSD DO NOT MATCH
(8) BIPHASIC
(9) NEEDS DILUTION
[] (1:5), [] (1:10)
[] ( )(WRITE IN)
(10) CONTAINS PRECIPITATE
(11) EVAPORATED
(12) BLANK CONTAM. WITH_____
(13) NEEDS TBA SULFITE CLEAN-UP
(14) NEEDS COPPER CLEAN-UP
(15) LOW/NO SPIKE
(16) HIGH SPIKE
OTHER_____
_____

REQUESTOR:_____ DATE:_____
AUTHORIZED BY:_____(MANAGER) DATE:_____
QA OFFICER:_____ DATE:_____

COPY OF COMPUTER GENERATED GC/MS SCHEDULE

Current date: 90/09/24 *** MS SCHEDULING SHEET *** Page: 1

Sequence:

Batch#: MS5310
Sched: 90/09/24
Category: VOA

10/1

Job#	Scrn	Amt	Fraction	Loglnk	Hold	Xrnd	Batch#	Acq	Wip	Comments
CA4910			1NJTSK4/VO+15	100629	09/30	DA14	QV7035	✓	✓	SPECVOA: VERBAL DATA
			1NJTSK4/VOA	100629	09/30	DA14	↓	✓	✓	DUE 14 DAYS VTSR.
			SPECVOA	100629	00/00	DA14		✓	✓	HARDCPY DUE 21 DAYS VTSR.

END OF BATCH

NO INDEP INTERNAL CHAIN OF CUSTODIES

Start date: [illegible] Signature: 9/25

Completion date: [illegible] Signature: 9/20

APPENDIX D-2

ENSECO

STANDARD
OPERATING
PROCEDURE

Subject or Title: SAMPLE LOG-IN		Page 1 of 3
SOP No.: LP-RMA-0003	Revision No.: 1.0	Effective Date: Feb. 27, 1992
Supersedes: Original		

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1. Purpose:

   To create analyses records in the laboratory computer for notification to lab analysts.

2. Policies:

   Log-in must be completed within 24 hours of authorization. Authorization occurs after all discrepancies are resolved or in some instances approved addendas are received.

Prepared by: Cindy Patterson	Date: February 27, 1992
Management Approval:	Date: 2-27-92
QA Officer Approval:	Date: 02/28/92

3. Safety:

Always wear gloves, safety glasses and lab coats when handling samples.

4. Procedure:

As log-in proceeds fill out the LOG-IN checklist (Figure 1) and address all the issues on this form.

4.1 Once all discrepancies (as described in the sample receipt SOP LP-RMA-0005) have been resolved as best possible, go to the second page of the project screen in LIMS. Make sure that the correct information regarding turn around time, due date, date of receipt are present. Accept the samples by using the sample delivery acceptance soft key. After each soft key, a "do" or saving key must be used to save the changes. Samples cannot be logged in unless this step is performed.

4.2 Determine group codes for each sample. Samples with the same analytical requests and same matrix type should be grouped together in one group code. Check with the program administrator regarding any special instructions for the samples.

4.3 In LIMS, go to "Group Code"by using the "Group Code" soft key. Add a group code by pressing the "Add Group Code" soft key. In the group code the LIMS sample numbers, matrix type and total sample number fields should be completed.

4.4 Next go to "Test List" using the "Test List" soft key. Use the "Add test list" soft key and add the test codes that are applicable to the client request for each sample as listed on the chain of custody and/or other supporting documentation.

4.5 Create group codes and add the requested tests for all samples with similar sample matrices and analytical requests.

4.6 Add any applicable special instructions for the corresponding groupcode in which the sample(s) is assigned. To input special instructions, the following sequence of soft keys must be used. If others are used, the special instructions will not appear. "Group Code", "Test List", "More Functions", "More Functions", "Group Instruc.", "Add Instruc." If special instructions are long, additional sequences may be necessary. Make sure a note that there are additional sequences is added.

4.7 Go to the sample list by using the "Sample List" soft key. Add each sample, (use "Add Sample" soft key) filling out all pertinent information such as; sampling date and time, group code, bottle types received. Fill out all fields.

4.8 Print the sample list, and a group code report using a control"R" at the sample list screen and group code screen. The paper work should be placed in the project folder. A level 3 checklist (Figure 2) should already be in the the project folder. Abort from this data base and go into "Project reports". Go to acceptance letter and print the acceptance letter by using the appropriate soft key. By using the appropriate soft keys print the sample description and request list paper work. This paper work should also be place into the project folder.

4.9 Fill out a chain of custody (Figure 3) for subcontracted work if necessary. A purchase order (Figure 4) must also be filled out. For subcontracting to another Enseco facility fill out the Interlaboratory Analysis Request form (Figure 5).

4.10 Perform any compositing, filtering or splitting as necessary. Create any additional preserved bottles if necessary.

4.11 Put samples in the proper locations. Volatiles are placed in separate refrigerators. Waters for organic prep are placed in refrigerators near the Organic prep labs. All other samples are placed by team identification in the walk in refrigerator.

4.12 Place project folder in the appropriate location for the team.

5. Responsibilities:

Program Administrator is responsible for reviewing that the log in has been performed correctly based upon the client's requirements and that sample receiving personnel are notified of log in errors. The Program Administrator is responsible for confirming that errors have been corrected. Sample receiving personnel are responsible for transferring the information received with the samples to the laboratory LIMS system and ensuring that a hard copy of this information is placed into the project folder.

6. Definitions:

Special instructions - Typed instructions in LIMS to the teams and analysts that are necessary to complete the work and can not be indicated by using one of the test codes assigned to the samples.

FIGURE 1

RMAL Industrial Login Review Revision 3.0 February 27, 1992

Project #: _____ Dup'd from project #: _____

Set-up By: _____ Dup'd Group Codes: _____

Logged By: _____ Date: _____

SAMPLE CONTROL REVIEW

	Y	N	INITIALS
1. Chain of Custody filled out correctly:	___	___	_____
2. Short holding time worksheet correct:	___	___	_____
3. Sample bottle/type correct:	___	___	_____
4. Overflow sample storage in special instructions:	___	___	_____
5. All login paperwork included and correct: Sample list, group code report & acceptance letter	___	___	_____
6. Trip blanks, equipment blanks, and field blanks have correct aliquot designation:	___	___	_____
7. Sample disc., request list, and acceptance letter in folder:	___	___	_____

8. Comments: Include action taken to resolve discrepancies. Include hardcopy of VAX mail, or extra paper, if more space is needed. Sign and date.

_____

_____

PROJECT ADMINISTRATOR REVIEW:

	Y	N	INITIALS	DATE
Report input sheet:	___	___	_____	_____
Invoice information:	___	___	_____	_____
All discrepancies resolved:	___	___	_____	_____
Sample and test matrices correct:	___	___	_____	_____
Sub paper work correct:	___	___	_____	_____
Clear picture of sub samples in folder	___	___	_____	_____
Special Instructions in LIMS:	___	___	_____	_____
Modified component lists checked:	___	___	_____	_____
Project due, TAT, received & collected dates OK:	___	___	_____	_____
Log released:	___	___	_____	_____

_____

_____

_____

RMAL Industrial Sample Checklist Revision 3.0 February 27, 1992

Project #:_____ Date/Time Received:_____

Company Name & Sampling Site:_____

*Cooler #(s):_____ _____ _____ _____

Temperatures:_____ _____ _____ _____

* Place copy of airbill inside all non-RMAL coolers. Describe here.

==========

UNPACKING & LABELING CHECK POINTS:	Y	N	INITIALS
1. Radiation Checked; (record reading if > 15 mr):	___	___	_____
2. Cooler seals Intact:	___	___	_____
3. Chain of Custody Present:	___	___	_____
4. Bottles broken or leaking (comment if Y): **-photograph broken bottles-**	___	___	_____
5. Containers labeled (comment if N):	___	___	_____
6. pH of samples taken: -any discrepancies between pH and bottle type? (list below)_____	___	___	_____
7. Chain of Custody signed with date, time & lab:	___	___	_____
8. CoC agrees with bottle count (comment if N):	___	___	_____
9. CoC agrees with labels (comment if N):	___	___	_____
10. VOA samples filled completely (comment if N):	___	___	_____
11. VOA samples preserved:	___	___	_____
12. Sediment present in "D" bottles:	___	___	_____
13. Short holding times:	___	___	_____
14. Matrix QC verified:	___	___	_____
15. Multi phase samples present (comment if Y): **-photograph multiphase samples-**	___	___	_____
16. Clear picture taken & labeled:	___	___	_____

Comments: include action taken to resolve discrepancies/problems. Include a hard copy of VAX mail or extra paper if more space is needed.

_____

_____

_____

Sign and Date:_____

FIGURE 2

Level 3 Checklist

Client:_____ Project #:_____

Report Writing Checklist	Organics Yes	Organics No	Inorganics Yes	Inorganics No
1. Are departmental camera ready report cover sheets available and complete? Have anomalies been documented?	___	___	___	___
2. Have holding times been met? If no, is the holding time violation notification present?	___	___	___	___
3. Are project data sheets and QC reports complete and in a finished client report format?	___	___	___	___
4. Is the industrial report writing checklist available and complete?	___	___	___	___

Initials:_____ Date:_____

Review of Checklists, Set-up, and Project	Yes	No
1. Is the chain of custody properly signed?	___	___
2. Have all requested parameters been reported for each sample?	___	___
3. Are anomaly forms, out-of-control forms, and report writing checklists available and complete?	___	___
4. Has confirmation of final disposition of samples been generated?	___	___
5. Has the client been notified about anomalies and/or missed holding times?	___	___
6. Are ALL phone conversations documented and present in project folder? (Self sticking or gummed note pads are not appropriate.)	___	___
7. Is the report format consistent with the client's request?	___	___
8. Are the project data consistent with related measurements and parameters?	___	___
9. Has the client's overall quality objectives been met?	___	___
10. Was the quoted turnaround time met?	___	___
11. If work was subcontracted, are the results consistent with the client's request?	___	___

PA Initials:_____ Date:_____

BC4

ENSECO, INC.
RMAL Division
Project Billing-Audit Checklist

Project No. _____

Audit Step	Procedure	Yes	No
1. Account Number	If new account, has Accounting Department been notified and credit approval obtained	___	___
2. Prices	Prices have been verified (including discount and surcharges)-Check as applicable	___	___
	A. Enseco Price List _____		
	B. Quotation _____		
	C. Contract _____		
3. Quantities	Quantities billed verified for accuracy	___	___
4. Penalties	Was guaranteed TAT period negotiated with client, if yes, complete separate sheet?	___	___
5. Client Data	Proforma invoice has been verified to ensure invoice is complete and mailed to correct person/dept.		
	A. Client ID verified	___	___
	B. Address verified	___	___
	C. P.O. # is shown or, if verbal, name of contact	___	___
7. Sub Out	Was project or part of project sub out (check one)	___	___
	A. Sister Lab A/C 920 _____		
	B. Outside Lab A/C 160 _____		
	C. Data Delivered to RMAL on time? If no, elaborate below.	___	___
	If yes to above has invoice been charged to account 920 or 160 as appropriate?	___	___

Project has been audited and approved for billing.

PA Signature _____ Date _____

FIGURES

Enseco
A Corning Company

Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada, CO 80002
303/421-6611 FAX: 303/431-7171

CHAIN OF CUSTODY

ENSECO CLIENT			
PROJECT			
SAMPLING COMPANY			
SAMPLING SITE			
TEAM LEADER			

SAMPLE SAFE™ CONDITIONS

PACKED BY		SEAL NUMBER
SEAL INTACT UPON RECEIPT BY SAMPLING COMPANY		CONDITION OF CONTENTS
SEALED FOR SHIPPING BY		INITIAL CONTENTS TEMP. °C
SEAL NUMBER	SAMPLING STATUS ☐ Done ☐ Continuing Until	
SEAL INTACT UPON RECEIPT BY LAB. ☐ Yes ☐ No	CONTENTS TEMPERATURE UPON RECEIPT BY LAB. °C	

DATE	TIME	SAMPLE ID/DESCRIPTION	SAMPLE TYPE	# CONTAINERS	ANALYSIS PARAMETERS	REMARKS

CUSTODY TRANSFERS PRIOR TO SHIPPING

RELINQUISHED BY (SIGNED)	RECEIVED BY (SIGNED)	DATE	TIME

SHIPPING DETAILS

DELIVERED TO SHIPPER BY		
METHOD OF SHIPMENT		AIRBILL NUMBER
RECEIVED FOR LAB	SIGNED	DATE/TIME
ENSECO PROJECT NUMBER		

ENS—1133

White – CLIENT Pink – LAB

FIGURE 4

PURCHASE ORDER

NUMBER _____

Show this Purchase Order Number on all correspondence, invoices, shipping papers and packages.

Date

Rocky Mountain Analytical Laboratory

4955 Yarrow Street, Arvada, CO 80002 (303) 421-6611

A DIVISION OF
ENSECO
INCORPORATED

To _____ Contact _____

_____ Phone _____

_____ Date Required _____

Emp. No.	Dept.	Project	Quantity	Item#/Description	Price	Amount	Rec'd.	Date

Acknowledge promptly if you are unable to ship complete order by date specified.
Invoice in triplicate.

Signed by _____

INTERLABORATORY CHAIN OF CUSTODY

Enseco
A Corning Company

PAGE OF

SHIP TO:

ATTENTION:

ANALYTICAL REQUESTS

SEND RESULTS TO:

ATTENTION:

EXPORT ID							SAMPLE CONDITION UPON RECEIPT	COMMENTS
TEST PRICE								
SUBTOTAL								
DISCOUNT / SURCHARGE								
TOTAL								

WRITTEN RESULTS REQUIRED BY (DATE)

VERBAL/FAC RESULTS REQUIRED BY (DATE)

P.O. No.

Q.C. ☐ STANDARD ENSECO ☐ CLP PROTOCOL ☐ PROJECT SPECIFIC

SAMPLE DISPOSAL ☐ ENSECO ☐ RETURN TO CLIENT ☐ PHONE

DETECTION LIMITS ☐ COMMON PRODUCTS ☐ OTHER*

HOLDING TIMES ☐ ENSECO ☐ EPA-CLP ☐ TIER ☐ OTHER*

RAW DATA COPIES NEEDED ☐ YES ☐ NO

CUSTODY SEALS INTACT ☐ YES ☐ NO ☐ WET WEIGHT ☐ DRY WEIGHT

*SPECIAL INSTRUCTIONS

RELINQUISHED DATE / TIME

RECEIVED DATE / TIME

ENS -1109

IMPORT COPY

APPENDIX D-3

WARZYN

CHAIN-OF-CUSTODY
Superfund Level

Scope and Application: Chain-of-custody procedures are used to maintain and document sample possession. This particular procedure is applicable when full chain-of-custody procedures are required for enforcement driven investigations.

Reference: NEIC Policies and Procedures, May, 1978 (Revised March, 1986). EPA-330/9-78-001-R.
U.S. EPA Central Regional Laboratory (10/15/82).

Procedure: Due to the legal nature of enforcement driven investigations, possession of samples must be traceable from the time the samples are collected until introduced as evidence in legal proceedings or destroyed. To maintain and document sample possession, strict chain-of-custody procedures are followed.

A sample is under your custody if:

1) it is in your possession, or
2) it is in your view, after being in your possession, or
3) it was in your possession and you locked it up, or
4) it is in a designated secure area.

Field Custody:

1. As few people as possible should handle samples.

2. The field sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched to the appropriate laboratory.

3. Sample tags and labels shall be completed for each sample, using water proof ink (unless prohibited by weather conditions). For example: a log book notation would explain that a pencil was used to fill out the sample tag/label because a ball point pen would not function in freezing weather.

4. The field project coordinator determines whether proper custody procedures were followed during the field work and decides if additional samples are required.

Transfer of Custody and Shipment

1. Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment. The method of shipment, courier name, and other pertinent information is entered in the "Remarks" (#14) section of the custody record (see Figure 3). Shipping containers are "sealed" with numbered chain-of-custody seals. The seal numbers are also noted in the "Remarks" section of the custody record.

2. Samples are accompanied by a chain-of-custody record (see Figure 3). When transferring the possession of samples, field personnel will complete the information required for #1 through #11 on the chain-of-custody. Any remarks pertaining to shipping are included in #14. This information is required prior to the field personnel relinquishing custody of the samples. This record documents sample custody transfer from the sampler, to the mobile laboratory or a distant laboratory.

3. Each shipping container will be accompanied by a chain-of-custody record identifying the contents. The original record will accompany the shipment, and a copy will be retained by the field project coordinator.

Sample Receipt and Sample Log-In Procedures

Safety Precautions: All samples received should be considered hazardous and appropriate precautions should be taken when handling these samples. Under no circumstances should any personnel other than the sample custodian or project leader open coolers. If damage or leakage is noted, stay clear of the coolers and notify the sample custodian or project leader immediately. All samples will be opened in a hooded area!

Procedure:

1.0 The project leader will notify the sample custodian in writing of incoming samples.

2.0 The custodian will receive the samples and deliver them to a hood located in the analytical laboratory. The procedures described below will be followed by the sample custodian:

2.1 Examine the shipping container and record the following information on the project log form (Refer to Figure 1). A separate project log form is required for each shipping container.

- The presence/absence of custody seal on the shipping container.

- The condition of the custody seal (i.e., intact, not intact).

2.2 Open the shipping container in a hooded area, remove the enclosed sample documents, and record the following information on the project log form.

- The presence/absence of the chain-of-custody record(s).

- The presence/absence of airbills and/or bills of lading documenting shipment of the samples.

2.3 Remove the samples from the container and record the following information in the project log form.

- Condition of samples (intact, broken, leaking, cold, etc).

- The presence/absence of sample tags.

- Sample tag numbers. Compare these numbers with the chain-of-custody record(s) (Figure 3). If sample tag numbers do not match. Record this fact on both the chain-of-custody record and project log form and contact the data management supervisor so that the discrepancy can be resolved.

2.4 Compare the following documents to verify agreement of the information contained on them.

- Chain-of-custody records.

- Sample tags.

- Airbills or bills of lading.

2.5 Sign the Chain-of-Custody (Figure 3 - #12 and #13), project log form and airbills.

3.0 Login the samples (refer to the "Chain-of-Custody, Log-In and Tracking Procedures" SOP Parts 2-7):

3.1 Each sample is assigned a unique sequential laboratory number. The laboratory number is entered on the chain-of-custody (#15), sample labels and sample tags.

3.2 The sample custodian will remove the sample tags. All tags will be placed in sealed envelopes and placed in the appropriate project file.

4.0 Sample Storage:

4.1 Samples and extracts will be stored in a secure area designated for strict chain-of-custody samples.

4.2 Damaged samples will be disposed of in an appropriate manner and the method of disposal documented.

4.3 The laboratory and sample storage areas are secure with strict limited access by only laboratory personnel.

4.4 Whenever samples are removed from storage, the removal will be documented. All transfers of samples will be documented on the internal chain-of-custody records. (Refer to Figure 2).

4.5 Samples and extracts will be stored after completion of analysis in accordance with the contract or until sample custodian is instructed otherwise by the enforcement Project Officer.

4.6 The location of stored organic extracts will be recorded.

4.7 VOA samples will be stored separately from organic extracts.

4.8 Standards are stored separate from samples.

4.9 Samples requiring refrigeration are stored in the walk-in coolers. refrigerator. Metal samples are stored in the black cabinets in log-in. Metal digestates are stored on shelves in the metals lab.

5.0 Sample Security:

5.1 Samples will be stored in a secure area.

5.2 Access to the laboratory will be through a monitored area. Other outside-access doors to the laboratory will be kept locked.

5.3 Visitors will sign a visitors log (located at the reception area and will be escorted while in the laboratory area.

5.4 Refrigerators, freezers, and other sample storage areas are secure as they are located within the laboratory.

5.5 Only laboratory personnel have access to the laboratory.

5.6 Samples will remain in secure sample storage until removed for sample preparation or analysis. All transfers of samples into and out of storage will be documented on an internal chain-of-custody record. An example of an internal chain-of-custody record used for this procedure is attached. (See Figure 2).

5.7 These internal custody records will be maintained in the project file.

5.8 After a sample has been requested from storage by the analyst, the analyst is responsible for the custody of the samples. Each analyst must return the samples to the storage area before the end of the working day.

Internal Chain-of-Custody Procedure:

The following procedure for documentation of internal chain-of-custody for samples requiring continuous custody.

1. Samples requiring refrigeration will be stored in the walk-in coolers located in the sample entry area. Metal samples are stored in the black cabinets in log-in. Metals digestates are located in the metals lab. A list of sample numbers will be maintained by the sample custodian.

2. Only the laboratory personnel will have access to the walk-ins.

3. Samples will remain in the designated walk-ins until removed for sample preparation and/or analysis.

4. All transfer of samples into or out of the walk-ins will be documented on an internal chain-of-custody record (see Figure 2). These records are maintained by the sample custodian.

5. Once a sample is removed from a walk-in by the sample custodian, he/she will then give it to the analyst, who is then responsible for the custody of the sample. Each analyst must return samples to the walk-in before the end of the working day. Samples are not allowed to sit on the bench overnight.

6. The next morning, sample custodian will put samples back in their proper place on the shelves. Sample custodian is responsible for signing sample request sheet that samples were returned properly. Any remarks will also be noted by the sample custodian.

7. When sample analyses and necessary quality assurance checks have been completed by the laboratory or after a 3 month time period whichever is longer, the unused portion of the sample should be disposed of properly. All identifying tags, data sheets, and laboratory records shall be retained as part of the permanent documentation of the project. Tags and forms are never discarded!

Revision Date

9-26-87

8-18-88

3-16-90

Michael Linskens

Michael J. Linskens
Director, Technical Services

Kim Finner

Kim D. Finner
Laboratory Manager

[DMSOP]

FIGURE 1

WARZYN

Sample Custodian Signature: _____

Date: _____

(Circle Appropriate Response)

Custody Seal — present/absent; intact/not intact

Chain-of-Custody — present/absent

Sample Tags — present/absent

Sample Tag Numbers — listed/not listed on chain-of-custody

Project Number: _____

Airbill Number: _____

Date Rec'd	Time Rec'd	Chain-of Custody Record No.	Corresponding Sample Tag Nos.	Corresponding Assigned Nos.	pH <2	pH >9	pH >12	Does info. on Custody Records, Traffic and Sample Tags Agree	Remarks Condition of Samples

FIGURE 2

WARZYN SAMPLE REQUEST SHEET

Requested By: _____

Date/Time Submitted: _____

Need Samples By:

Date: _____

Time: _____

I am requesting:

Project No.	Sample No(s)	Parameter	Matrix	Remarks

I am returning: Date/Time:

Project No.	Sample No(s)	Remarks

FIGURE 3

CHAIN OF CUSTODY RECORD

University Research Park
P.O. Box 5385
Madison, Wisconsin 53705
(608) 273 0440

PROJ. NO. #1	PROJECT NAME # 2A	NO. OF CONTAINERS #8	REMARKS #9
	LOCATION: # 2B		
SAMPLERS: (Signature) #3			

LAB NO. #15	DATE #4	TIME #5	COMP.	#6 GRAB	#7 STATION LOCATION	NO. OF CONTAINERS #8							REMARKS #9

Relinquished by: (Signature)	Date / Time		Received by: (Signature)	Relinquished by: (Signature)	Date / Time		Received by: (Signature)
#10	#11A	#11B					
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) #12		Date / Time #13A	#13B	

Remarks #14

PROJECT MANAGER: #15

CHAIN-OF-CUSTODY, LOG-IN AND TRACKING PROCEDURES

Scope and Application: This procedure is applicable to the log-in of samples and addresses such areas as: sample custody, log-in, labeling and preservation of samples. This procedure begins as the samples enter the laboratory and follows the samples to their destruction.

Procedure:

1. Chain-of-Custody Procedure: Samples arriving in the laboratory are accompanied by some type of custody record. The most common type of record is that shown in Figure 1. Follow the instructions below for all levels of chain-of-custody.

   A. Superfund Level Chain-of-Custody:

      1. Refer to the "Chain-of-Custody - Superfund Level" SOP.

   B. Routine Chain-of-Custody:

      1. Samples arriving in the laboratory are accompanied by a chain-of-custody record (refer to Figure 1).

      2. Review the custody form with the samples received and see that the spaces marked #1 - #12 have been accurately completed by the sampler.

      3. Any discrepancies with the chain-of-custody record or the samples should be immediately brought to the attention of the customer service relations person so that the problems can be solved in an efficient manner.

      4. If samples were shipped, note the courier name in the remarks section (#15) of the chain-of-custody record.

      5. Record any other comments such as: temperature upon receipt, preservation (if done in lab), problems, etc, in the remarks section (#15).

      6. Fill in the lab numbers in the section marked #16 (refer to the log-in portion of this procedure for how to obtain lab numbers).

      7. Sign and date the chain-of-custody record (#13 - #14).

      8. Route the custody record to the data management office when log-in is complete.

NOTE: The laboratory is a secured area with strict limited access. Any samples arriving to the laboratory during "off hours" are stored in the laboratory refrigerator until they are logged in. Laboratory personnel are responsible for the care and custody of the samples during the analysis stage and until the samples are removed from the refrigerator and properly disposed of.

2. Log-In Procedure: Samples are logged in as follows (Refer to Figure 2):

A. Sample Information:

Care should be taken to document the condition of samples upon receipt. Anomalies such as broken or leaking bottles, expired holding times, improper labeling or preservation, and air bubbles in VOA vials should be noted on the chain-of-custody. It should also be noted whether the samples arrived cold.

B. Numbering Samples:

Number the samples on the chain-of-custody form. Record lab numbers used in the black lab book. If no chain-of-custody form accompanied the samples, the sampler must be contacted and one must be filled out. Blank forms are available in the log-in area.

NOTE:
- Sample preparation is required if the samples need to be filtered and/or preserved in the lab.
- If metals are requested, metal digestion is required for all matrices except filtered groundwaters or private well.
- Sample compositing is required if the lab is instructed to composite a series of samples received into one sample for analysis.

C. Subcontracted Work Required:

Refer to the subcontracting SOP for further instructions on subcontracting samples.

D. Special Accounting Notes:

If there is a discount or a special proposal for the sample analyses to be charged, record that information on the chain-of-custody.

E. Labeling Bottles:

1. Record the lab number on the sample bottle label and caps using a water-proof marker.

2. Check that the appropriate preservation and preparation has been circled.

3. If metals and total hardness are both required, using a red water-proof marker, place a dot on the bottle cap of that bottle to be used for those analyses.

F. Preserving Samples:

1. Samples which have been preserved in the field must be checked via pH paper in the laboratory to insure they were preserved to the correct pH. Check preserved samples as follows:

   a. Acid-preserved (HNO_3 or H_2SO_4) samples must be preserved to a pH <2. Using a pH stick, check the pH. If the pH is not <2, note this on the chain-of-custody, then add additional acid until a pH is obtained. IT IS CRITICAL THAT THE CORRECT TYPE OF ACID BE ADDED.

   b. Base-preserved NaOH samples must be preserved to a pH > 12. Using a pH stick, check the pH. If the pH is not > 12, note this on the chain-of-custody, then add additional 10N NaOH until a pH > 12 is obtained.

   c. Base-preserved NaOH with ZnAc samples must be preserved to a pH > 9. Using a pH stick, check the pH. If the pH is not > 9, note this on the chain-of-custody, then add additional 10N NaOH until a pH > 9 is obtained.

2. Samples which have not been preserved in the field need to be preserved in the laboratory. Record any preservation done in the lab on the field sheets or custody records. Refer to Figure 3 for preservation requirements and preserve accordingly. It is important that samples arrive in appropriate containers (see Figure 3). If they do not, contact the inorganic/organic supervisor. They will determine which analyses, if any, can be run.

3. It is important that samples be received and maintained cold for most analyses. If a sample was received warm, make note of this on the field sheet or custody record.

G. Computer Log-In:

Analysis requests are entered into the Laboratory Information Management System (LIMS). The LIMS database is used for scheduling of analytical work and to produce accounting invoices. Analysis requests are entered into the LIMS database as follows:

1. Turn on the computer terminal.

2. Enter: C CHICO (return).

3. Enter: ASCORA (return).

4. Enter: Password (return).

5. At the VMS $ prompt, enter LABMENU (return).

6. Enter: LOGIN (return).

7. Enter: Password (return).

8. The Log-In screen will appear as shown in Figure 2. Using information on the chain-of-custody, proceed as follows:

   a. Enter project number (return). Account number and name will be filled in automatically. If the information matches the chain-of-custody, press return.

   b. Sample date automatically defaults to yesterday's date. If this is correct, press return or over ride with correct date.

   c. Enter sample identification from station location on the chain-of-custody and press return.

   d. Enter proper matrix number and press return.

   e. Desc: This is used if sample identification is longer than the number of characters in the sample identification field. (Return to over ride or enter information and return.)

   f. Date received automatically defaults to today's date. If correct, press return or over ride with correct date.

   g. Days to process automatically defaults to 14 days. If this is correct (standard turnaround time) press return or if rush work, over ride with the number of days until due.

   h. Duedate: First date listed is report date, second date is the lab due date which can be changed if necessary.

i. Comments: Enter special tests or other information needed to analyze samples correctly and press return.

j. Manager: Filled in automatically; press return if correct or over ride.

k. Price/Cost: Return if correct or over ride.

l. % Markup/Discount: For accounting. Enter a negative number for a discount (i.e. -15 for 15%) and a positive number for markups (i.e. 100 for 2 x price). Check accounting sheets for projects with special pricing.

m. To enter parameters, press **Next Screen** (see Figure 2A).

n. Matrix automatically defaults to matrix entered above (return or override).

o. Enter the proper code name for the test to be run from report MF01. Use parent codes if applicable for quicker entry. (Press the down arrow key, return.)

p. To enter address for the report, press **Next Screen** (see Figure 2B).

q. Type: Report (return).

r. If an address has already been entered and matches the chain-of-custody press **Commit** and return.

s. If no address appears, press return until cursor is in the City field. Type in the city and press return.

t. Type in state (press **Commit** and return). After city and state has been entered for the first sample, it will automatically be copied for the rest of the sample numbers in a single log-in.

9. To duplicate the entry for another sample on the same chain-of-custody:

   a. If next sample to be entered has all the same information as the previous sample entered, press **Duplicate Record** key (F7).

   b. Enter sample identification and press **Commit** key, return.

10. To duplicate only the information in the upper block of the log-in screen (sample date, matrix etc.):

   a. Press **Create Record** key (Insert Here) and enter sample identification.

b. Press **Previous Screen** (F12) if sample date needs to be changed.

c. Press **Next Screen**, return. Then press **Next Screen** again to enter new product codes for the analyses requested.

d. Press **Previous Screen** to enter new sample or to quit.

e. Press **Clear Form** (F17) to enter a new chain-of-custody.

f. Press **Exit/Cancel** (PF4) to exit from the log-in screen.

1. Press **Exit/Cancel** to exit from the LIMS system.
2. At the VMS $ prompt, enter LO (return) to log off the VAX computer.
3. Enter N (return).

13. If in Step 8a. the computer says project number is invalid:

a. Press **Exit/Cancel** (PF4).

b. Enter "Y" (return).

c. On the LIMS Main menu, use the up arrow key to position the cursor on "Seedpak Management". Press **Commit** key.

d. Use the down arrow key to move to project entry. Press **Commit** key.

e. Enter project number (return).

f. Enter account number (9999) and return.

g. Enter description for chain of custody (project name).

h. Enter manager from chain-of-custody (i.e. Finner, K).

i. Press **Exit/Cancel** (PF4), twice.

j. At the LIMS main menu, select log-in and press **Commit**.

14. To print daily log-in reports:

a. Print out the appropriate FS02 reports for only the samples logged in that day. The computer printout must be checked against the custody record or field sheets by the inorganic/organic supervisors so that any errors can be corrected. Print a report as follows:

b. Select "Seedpak Reporting" from the main menu, and press **Commit**.

c. Select FS02 and press **Commit**.

d. Enter today's date, press **Commit** and return.

e. The inorganic/organic supervisors are responsible for updating and printing lab schedules as they require them.

f. Any "rush" work is to be brought to the inorganic/organic supervisors' attention immediately.

15. Sample Storage:

a. Proper handling and storage is necessary in order to maintain the integrity of the samples. Refer to Figure 3 for storage requirements.

b. Refrigerated Storage:

1. Store routine custody samples in the walk-in refrigerator. Separate samples by preservative type. Samples with organic analyte requests are stored on a separate shelf in the walk-in.

c. Non-Refrigerated Storage:

1. Metal samples are stored in the log-in area in the appropriate cabinets.

2. Other samples not requiring refrigeration are stored on the appropriate shelves next to the walk-in refrigerator.

d. Any samples removed for analysis must be put back in the original location when the analysis is complete.

16. Sample Handling:

a. Samples are handled in and out of the walk-in coolers and black cabinets by the sample custodian.

b. Analysts are responsible for filling out sample request forms (see Figure 4) to request needed samples.

c. Forms are given to the sample custodian.

d. Sample custodian gathers requested samples and leaves yellow copy of form with samples, and keeps the white copy.

e. Analysts return samples to the walk-in accompanied by the yellow copy of the request form.

f. Next morning, sample custodian puts samples back in their proper place on the shelves.

g. Sample custodian signs off on white sheet that samples were returned. Any remarks are also noted on the white sheet.

17. Storage/Disposal:

Due to limited sample storage facilities available, the following guidelines have been created for sample storage/disposal. Sample storage time may be extended upon client's request.

a. One week after the report issue date, inorganic samples which are in cold storage are to be transferred to warm storage.

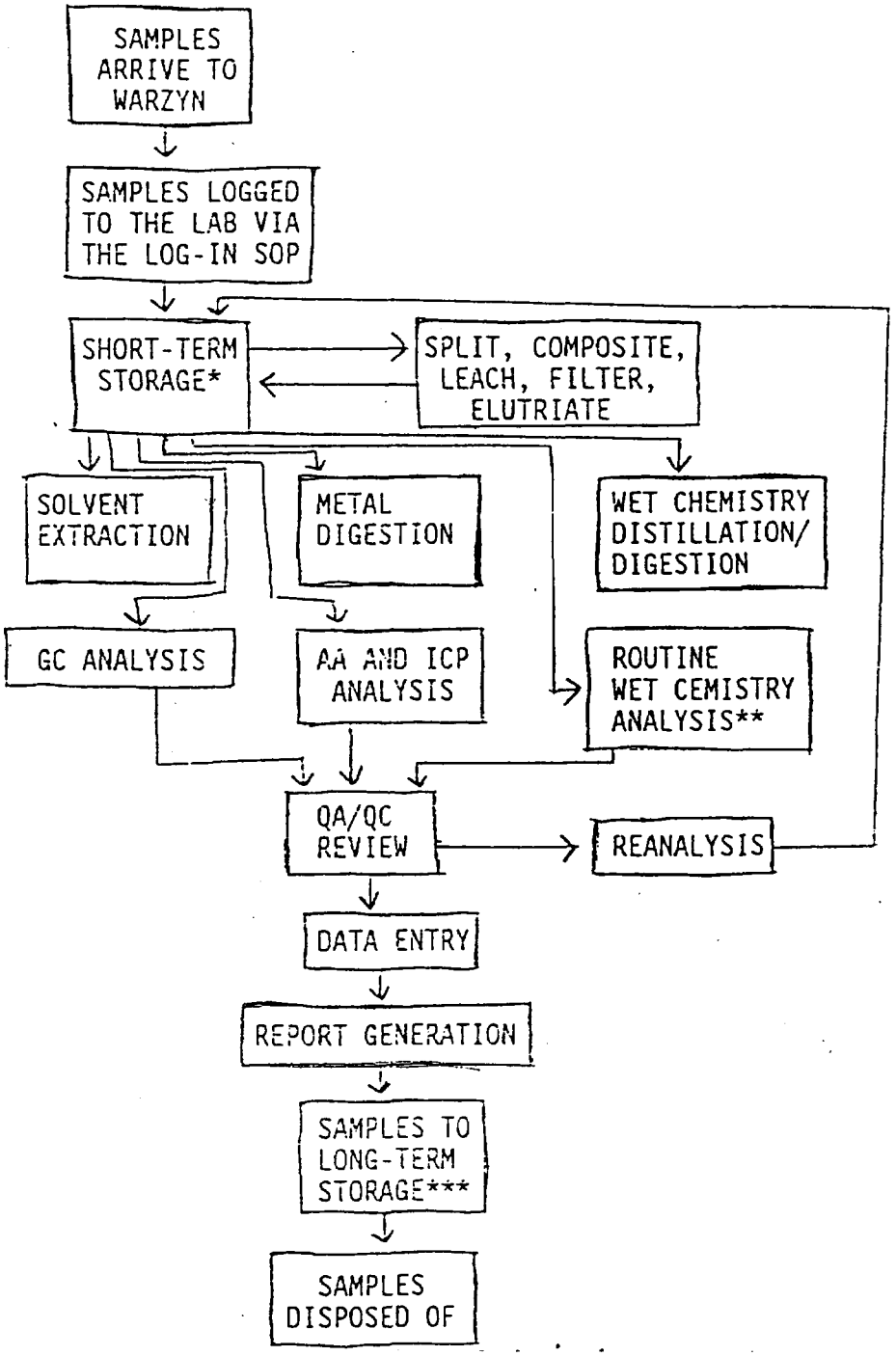
b. One month after the report issue date, organic samples which are in cold storage are to be transferred to warm storage.

c. Samples in warm storage should be disposed of as follows:

- Aqueous Samples: Dispose 1 month after report issue date.
- Non-Aqueous Samples: Dispose 6 months after report issue date.

	Revision Date
Michael Linskens Michael J. Linskens Director, Technical Services	08/18/87 08/15/88
Kim Finner Kim D. Finner Laboratory Manager	3-18-90

WARZYN ENGINEERING INC.
ANALYTICAL SERVICES
SAMPLE FLOW DIAGRAM



* SHORT TERM STORAGE: WALK-IN REFRIGERATOR FOR SAMPLES REQUIRING REFRIGERATION, BLACK CABINETS IN SAMPLE LOG-IN AREA FOR SAMPLES NOT REQUIRING REFRIGERATION, WALK--INS FOR SAMPLES REQUIRING "SUPERFUND LEVEL CHAIN-OF-CUSTODY".

** ROUTINE WET CHEMISTRY ANALYSES INCLUDE BOTH AUTOMATED AND MANUAL TESTS.

*** LONG TERM STORAGE: NON-REFRIGERATED STORAGE IS LOCATED IN THE FIELD STORAGE ROOM.

WARZYN

FIGURE 1

CHAIN OF CUSTODY RECORD

Warzyn Engineering Inc.
One Science Court
University Research Park
P.O. Box 5385
Madison, Wisconsin 53705
(608) 273-0440

PROJ. NO.	PROJECT NAME	
#1	LOCATION:	#2 also

SAMPLERS: *(Signature)*
#3

#16 LAB NO.	DATE	#4 TIME	#5. COMP.	#6 GRAB	#7 STATION LOCATION	NO. OF CONTAINERS #8							REMARKS #9

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
#10	#11				
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)* #13		Date / Time #14	

Remarks
#15

PROJECT MANAGER: #12

FIGURE 2

```
+------------------------------ Login/Order Entry ------------------------------+
|Project #: STANDARD___________ Acct:_________ ____________________________|
+-------------------------------------------------------------------------------+
| Sample #: __________ Sample Date: _________ Sample ID: ____________________|
|   Matrix: ___________ Desc: ____________________________________________|
| Received: 14-FEB-90  Days to Process:  14__  Due Date: _________ _________|
| Comments: ____________________________________ Manager: ____________________|
|    Order: __________ on 14-FEB-90          Price/Cost: 0_________/0_________|
|    Login: __________                 % Markup/Discount: 0_____                |
+-------------------------------------------------------------------------------+
| Matrix        Product            Type Price      Cost       Hold date PList |
| __ _________ ____________________ _ _________ _________ _________ _        |
| __ _________ ____________________ _ _________ _________ _________ _        |
| __ _________ ____________________ _ _________ _________ _________ _        |
| __ _________ ____________________ _ _________ _________ _________ _        |
| __ _________ ____________________ _ _________ _________ _________ _        |
| __ _________ ____________________ _ _________ _________ _________ _        |
| __ _________ ____________________ _ _________ _________ _________ _        |
| __ _________ ____________________ _ _________ _________ _________ _        |
| __ _________ ____________________ _ _________ _________ _________ _        |
| __ _________ ____________________ _ _________ _________ _________ _        |
+-------------------------------------------------------------------------------+
```

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FIGURE 2A

Login/Order Entry

Project #: 60818.00 Acct:9999 GPC-576

Sample #: 496-003 Sample Date: 28-JAN-90 Sample ID: MW-3
Matrix: 1 GroundH2O Desc:
Received: 31-JAN-90 Days to Process: 14 Due Date: 14-FEB-90 12-FEB-90
Comments: Manager: CLARK, J
Order: 619 on 31-JAN-90 Price/Cost: 312 /0
Login: 496 % Markup/Discount: 0

Matrix	Product	Type	Price	Cost	Hold date	PList
1 GroundH2O	AG	S	12	0	12-FEB-90	S
1 GroundH2O	AS	S	25	0	12-FEB-90	S
1 GroundH2O	BA	S	12	0		S
1 GroundH2O	CD	S	12	0	12-FEB-90	S
1 GroundH2O	CR	S	12	0	12-FEB-90	S
1 GroundH2O	DIGASSE	S	0	0		S
1 GroundH2O	HG	S	32	0	25-FEB-90	S
1 GroundH2O	PB	S	22	0	12-FEB-90	S
1 GroundH2O	SDF	S	10	0		S
1 GroundH2O	SE	S	25	0	12-FEB-90	S

v Char Mode: Replace Page 1 Count: 1

FIGURE 2B

Sample Addresses for 496-003___

```
Type        #    +-------------------------------------------------------+
REPORT____  1_   |        Name: ____________ _ ____________________      |
                 |      Addr 1: ____________________________________      |
                 |      Addr 2: ____________ ____________________________ |
                 |        City: ____________________________________      |
                 |   State/ZIP:   __ ____________                        |
                 |       Phone: ___-___-____                             |
Type             |                                                       |
_________ __     |        Name: ____________ _ ____________________      |
                 |      Addr 1: ____________________________________      |
                 |      Addr 2: ____________________________________      |
                 |        City: ____________________________________      |
                 |   State/ZIP: ____ ____________                        |
                 |       Phone: ___ ___ ____                             |
Type             |                                                       |
                 |        Name:                                          |
                 |      Addr 1:                                          |
                 |      Addr 2:                                          |
                 |        City:                                          |
                 |   State/ZIP:                                          |
                 |       Phone:                                          |
```

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FIGURE 3

(Page 1 of 3)

SAMPLE COLLECTION AND PRESERVATIVE REQUIREMENTS

Parameter	Container. (size in mLs)		Preservation	Maximum Holding Time
Wet Chemistry				
Acidity	P,G	(A)	Cool, 4°C	14 days
Alkalinity	P,G	(A)	Cool, 4°C	14 days
BOD_5	P,G	(1000)	Cool, 4°C	48 hours
Boron	P	(A)	Cool, 4°C	
Bromide	P,G	(A)	None Required	28 days
Carbon, Total Organic	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chemical Oxygen Demand	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chloride	P,G	(A)	None Required	28 days
Chlorine, Residual	P,G	(A)	None Required	Analyze Immediately
Cyanide, Amenable	P,G	(1000)	Cool, 4°C, NaOH to pH>12 (I)	14 days (I)
Cyanide, Total	P,G	(1000)	Cool, 4°C, NaOH to pH>12 (I)	14 days (I)
Density/Specific Weight	P,G		Cool, 4°C	
Fluoride	P,G	(A)	None Required	28 days
Hardness	P,G	(C)	HNO_3 to pH<2	6 months
Nitrogen, Ammonia	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrogen, Nitrate	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrogen, Nitrite	P,G	(A)	Cool, 4°C	48 hours
Nitrogen, Total Kjeldahl	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrogen, Total Organic	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Oil and Grease	G	(1000)	Cool, 4°C, H_2SO_4 to pH<2	28 days
pH	P,G	(A)	None Required	Analyze Immediately
Phenolics, Total	G	(500)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Phosphorous, Ortho	P,G	(A)	Filter Immediately, Cool 4°C	48 hours
Phosphorous, Total	P,G	(B)	Cool, 4°C, H_2SO_4 to pH<2	28 days
Silica, Dissolved	P	(A)	Cool, 4°C	28 days
Solids, Dissolved	P,G	(D)	Cool, 4°C	7 days
Solids, Settleable	P,G	(1000)	Cool, 4°C	48 hours
Solids, Suspended	P,G	(D)	Cool, 4°C	7 days
Solids, Total	P,G	(D)	Cool, 4°C	7 days
Solids, Volatile	P,G	(D)	Cool, 4°C	7 days
Specific Conductance	P,G	(A)	Cool, 4°C	28 days
Sulfate	P,G	(A)	Cool, 4°C	28 days
Sulfide	P,G	(500)	Cool, 4°C, add 2 mL 2N Zinc acetate/1000 mL plus NaOH to pH>9	7 days
Surfactant (MBAS)	P,G	(1000)	Cool, 4°C	48 hours
Thiocyanate	P,G	(500)	Cool, 4°C	
Total Organic Halides (TOX)	Amber Glass	(E)	Cool, 4°C	14 days
Turbidity	P,G	(A)	Cool, 4°C	48 hours
Metals				
Hexavalent Chromium	P,G	(A)	Cool, 4°C	24 hours
Mercury	P,G	(C)	HNO_3 to pH<2	14 days in plastic, 28 days in glass
Metals (all, except above)	P,G	(C)	HNO_3 to pH<2	6 months
Organics				
Gas Chromatography				
Volatile Organics	VOC vial	(2 ea, 40)	Cool, 4°C (J)	14 days (J)
Phenols	Amber glass	(H)	Cool, 4°C (K)	7 days (O)
Pesticides and PCBs	Amber glass	(H)	Cool, 4°C (L)	7 days (O)
Pesticides	Amber glass	(H)	Cool, 4°C (L)	7 days (O)
PCBs	Amber glass	(H)	Cool, 4°C	7 days (O)
Polynuclear Aromatics	Amber glass	(H)	Cool, 4°C (K), Store in Dark	7 days (O)
Organophosphorus Pesticides	Amber glass	(H)	Cool, 4°C, Store in Dark	7 days (O)
Chlorinated Herbicides	Amber glass	(H)	Cool, 4°C	7 days (O)
Total Hydrocarbon Screen	Amber glass	(H)	Cool, 4°C	7 days (O)
GC/MS				
Priority Pollutants/HSL				
Volatiles	VOC vial	(2 ea, 40)	Cool, 4°C	
Semi-volatiles	Amber glass	(H)	Cool, 4°C	
Pesticides/PCBs	Amber glass	(H)	Cool, 4°C	
CLP Protocol				
Volatiles	VOC vial	(2 ea, 40)	Cool, 4°C	
Semi-volatiles	Amber glass	(H)	Cool, 4°C	
Pesticides/PCBs	Amber glass	(H)	Cool, 4°C	
Radiological Tests				
Alpha, Beta	P,G	(250)	HNO_3 to pH<2	6 months
Radium	P,G	(1 gal)	HNO_3 to pH<2	6 months
Bacterial Tests				
Coliform, Fecal and Total	P,G	(F)	Cool, 4°C (F)	6 hours
Fecal Streptococci	P,G	(F)	Cool, 4°C (F)	6 hours
Solids				
RCRA Classification	P,G	(1000) (G)	Cool, 4°C	--
Volatile Organics	VOC vial	(140) (H)	Cool, 4°C	--
Semi-volatile Organics	G	(8 oz)	Cool, 4°C	--
Metals	P,G	(8 oz)	Cool, 4°C	--
Inorganics	P,G	(8 oz)	Cool, 4°C	--

Notes

(A) 1-3 Analyses; 250 mL required
4-7 Analyses; 500 mL required
>8 Analyses; 1000 mL required

(B) 1-3 Analyses; 250 mL required
4-7 Analyses; 500 mL required

(C) The metals selenium, arsenic, silver, mercury and hardness require additional volume. Use the following bottle scenario;
- One 250 mL bottle for all metals excluding above
- One 250 mL bottle for all metals plus two of the above analytes (except mercury). If mercury is required, an additional 250 mL is required. Label bottle for mercury analyses.
- One 500 mL bottle for all metals, including all five of the above analytes. Two 250 mL bottles may be used instead.

(D) Dissolved solids require 100 mL of sample. This volume can be included in the (A) unpreserved bottle.
The volume required for suspended, total and volatile solids depends on the nature of the sample. Dirty samples require 100-250 mL, clean require 1000 mL. Sample accordingly.

(E) Collect sample in either a 250 mL amber bottle with inverted cap or 250 or 500 mL amber bottle with teflon septa. Collect with no headspace.

(F) Collect 250 mL using either sterilized Whirl-Pak bag or nalgene bottle. Collect in Whirl-Pak bags with sodium thiosulfate ($Na_2S_2O_3$) additive if the sample is chlorinated.

(G) Collect the sample in a 1-liter wide-mouth jar. Collect at least 500 grams for analysis.

(H) Volatile organics may also be collected using two 40-mL VOC vials.

(I) Add 0.6 g ascorbic acid if residual chlorine is present.
Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, remove by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is then filtered and NaOH is added to pH >12.

(J) Preserve chlorinated samples with 1 crystal of sodium thiosulfate ($Na_2S_2O_3$) per vial.
Samples can be either preserved with 1:1 HCl or left unpreserved.
Samples receiving no pH adjustment must be analyzed within 7 days of sampling.
Samples that contain hydrocarbons (samples from gasoline or fuel oil contaminated sources) must be preserved with 1 drop of 1:1 hydrochloric acid (HCl).
If either acrolein and acrylonitrile are to be analyzed, the pH should be adjusted to 4-5 with 1:1 hydrochloric acid (HCl). Samples receiving no pH adjustment must be analyzed within 3 days of sampling.

(K) Add 0.008% sodium thiosulfate ($Na_2S_2O_3$) if residual chlorine is present.

(L) Adjust pH to 5-9 with sulfuric acid (H_2SO_4). The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of Aldrin, add 0.008% sodium thiosulfate ($Na_2S_2O_3$) to the sample.

(M) Collect two (2) 1000 mL amber glass bottles with teflon-lined caps per analysis.

(N) Collect two (2) 80 oz amber glass bottles with teflon-lined caps for one or both analyses listed. Alternatively, four (4) 1-liter amber bottles with teflon-lined caps may be used.

(O) The holding time is 7 days until extraction, 40 days after extraction.

BJH/cac
[cac-70C-93A]

FIGURE 4

WARZYN SAMPLE REQUEST SHEET

Requested By: _____

Date/Time Submitted: _____

Need Samples By:

Date: _____

Time: _____

I am requesting:

Project No.	Sample No(s)	Parameter	Matrix	Remarks

I am returning: Date/Time:

Project No.	Sample No(s)	Remarks

APPENDIX D-4

ENSECO - AIR TOXICS LABORATORY

Enseco
A Corning Company

STANDARD
OPERATING
PROCEDURE

Subject or Title: Sample Identification for Enseco - Air Toxics		Page 1 of 4
SOP No.: LP-ATL-0002	Revision No.: Original	Effective Date: November 22, 1991
Supersedes: None		

ENSECO PROPRIETARY INFORMATION STATEMENT

These documents have been prepared by and remain the sole property of Enseco, Inc. They are submitted to a client/government entity solely for its use in evaluating Enseco's qualifications in connection with the particular project, certification or approval for which they were prepared, and are to be held proprietary to Enseco.

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Prepared By: Valerie Zugates-Wildman	Date: November 19, 1991
Management Approval:	Date: 11/21/91
QA Officer Approval: Walter Berger	Date: 11-20-91

Enseco
A Corning Company

STANDARD
OPERATING
PROCEDURE

Subject or Title:
Sample Identification for Enseco - Air Toxics

SOP No.:	Revision No.:	Effective Date:
LP-ATL-0002	Original	November 22, 1991

1. SCOPE AND APPLICATION

1.1 To describe the procedures used in the assignment of laboratory identification numbers to samples.

2. POLICIES

2.1 All Enseco Safety policies will be strictly adhered to while performing the following duties.

2.2 Ensure client confidentiality at all times.

2.3 Enseco security policy will be adhered to for all non-Enseco visitors entering the sample control receiving door.

3. SAFETY

3.1 All Enseco safety policies will be adhered to at all times.

3.2 All samples are considered hazardous. Always handle samples in the most safe condition as possible.

3.3 Always ask when there is a question about safety.

4. PROCEDURE

4.1 Each sample is assigned a unique identification number. The numbers are assigned when the project is logged into the laboratory.

4.2 Samples are logged into a logbook that is used to keep track of the next available project number. The following information is recorded in the logbook:

Date Received
Time Received
Sample Description
Analysis Required
Container Type
Date/Time Sampled
Client/Project
Initials of Person Logging in Project

Enseco
A Corning Company

STANDARD
OPERATING
PROCEDURE

Subject or Title:
Sample Identification for Enseco - Air Toxics

SOP No.:	Revision No.:	Effective Date:
LP-ATL-0002	Original	November 22, 1991

4.3 The laboratory sample ID numbers consist of five parts:

4.3.1 "A-"

Every sample ID number begins with "A-" to identify that the project is from the Air Toxics laboratory of Enseco-CRL.

4.3.2 The year.

The last two digits of the current year.

4.3.3 The day.

The number of the day of the year that the samples are logged in. January 1 is "001" and December 31 is "365" ("366" in leap years).

4.3.4 The project.

Starting with "01", each project logged in is given a unique number.

4.3.5 Sample numbers.

Starting with "-001", each sample in the project is given a unique number.

Examples: Sample # A-9100503-006 would be sample #6 of the third project logged in on January 5, 1991. Sample # A-9036115-013 would be sample #13 of the 15th project logged in on December 27, 1990.

4.4 Each sample is labeled with its sample ID number by either attaching a sample ID tag or affixing a label.

Enseco
A Corning Company

STANDARD
OPERATING
PROCEDURE

Subject or Title: Sample Identification for Enseco - Air Toxics		Page 4 of 4
SOP No.: LP-ATL-0002	Revision No.: Original	Effective Date: November 22, 1991

5. Responsibilities

5.1 Sample Control Technician

5.1.1 The Sample Control Technician is responsible for following the procedures and policies set forth in this SOP in a safe and timely manner.

5.2 Laboratory Director

5.2.1 The Laboratory Director is responsible for ensuring the procedures and policies set forth in this SOP are followed in a safe and timely manner.

7. SAMPLE CUSTODY

Upon receipt by Enseco, samples proceed through an orderly processing sequence specifically designed to ensure continuous integrity of both the sample and its documentation.

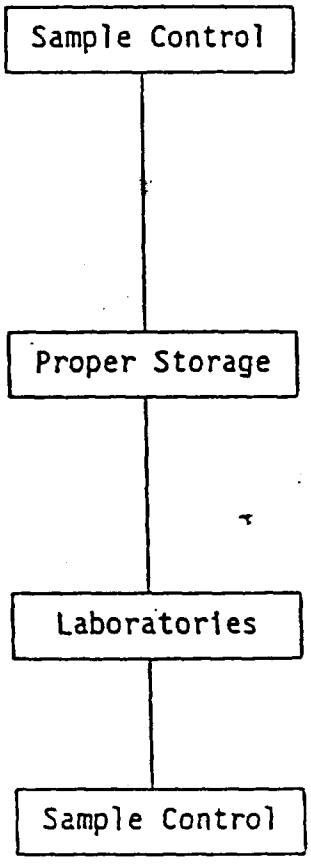
All samples are received by Enseco's Sample Control Group and are carefully checked for label identification, and completed, accurate chain-of-custody records. Photographs document the condition of samples and each sample is then assigned a unique laboratory identification number through a computerized Laboratory Information Management System (LIMS) that stores all identifications and essential information. The LIMS system tracks the sample from storage through the laboratory system until the analytical process is completed and the sample is returned to the custody of the Sample Control Group for disposal. This process is summarized in Figure 7-1. Access to all Enseco laboratories is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

An example of the Enseco Chain-Of-Custody Record used to transmit samples from the client to the laboratory is given in Figure 7-2. The Chain-Of-Custody Record (Interlaboratory Analysis Form) used to transmit samples between laboratories within Enseco is given in Figure 7-3.

Sample bottles provided to the client by Enseco are transmitted under custody using the Enseco "Sample Safe™".

Figure 7-1

ENSECO SAMPLE PROCESSING FLOW CHART



* Check and document physical condition of sample
* Verify documentation and parameter assignment
* Log into LIMS
* Send acknowledgement letter to client

* Store sample according to preservation guidelines
* Transfer sample to lab with proper documentation

* Document analytical work
* Return unused samples to Sample Control

* Return sample to client or arrange for sample disposal

Enseco

CHAIN OF CUSTODY

No. 5068

Attn: _____

Enseco Client _____

Project _____

Sampling Co. _____

Sampling Site _____

Team Leader _____

SAMPLE SAFE™ CONDITIONS

1. Packed by: _____ Seal # _____
2. Seal Intact Upon Receipt by Sampling Co.: Yes No
3. Condition of Contents: _____
4. Sealed for Shipping by: _____
5. Initial Contents Temp.: _____ °C Seal # _____
6. Sampling Status: Done Continuing Until _____
7. Seal Intact Upon Receipt by Laboratory: Yes No
8. Contents Temperature Upon Receipt by Lab: _____ °C
9. Condition of Contents: _____

Date	Time	Sample ID/Description	Sample Type	No. Containers	Analysis Parameters	Remarks

CUSTODY TRANSFERS PRIOR TO SHIPPING

	Relinquished by: (signed)	Received by: (signed)	Date	Time
1				
2				
3				

SHIPPING DETAILS

Delivered to Shipper by: _____

Method of Shipment: _____ Airbill # _____

Received for Lab: _____ Signed: _____ Date/Time _____

Enseco Project No. _____

SAMPLE

FIGURE 7-2

FIGURE 7-3

Page _____ of _____
File Index _____

Enseco

INTERLABORATORY ANALYSIS

SHIP TO: *(circle one)*

CAL ERCO CLE GAS MAR HOU

Attention:

SEND RESULTS TO:
Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada, CO 80002
(303) 421-6611 FAC: (303) 431-7171
Attention:

CLIENT NAME | PROJECT NO.

Relinquished by: *(Signature)* Received by: *(Signature)* Date Time

Relinquished by: *(Signature)* Received by: *(Signature)* Date Time

Import Lab ID	Enseco ID	Client ID	Matrix (a, s, w)	Date Sampled	Date Rec'd	Date Auth.	Analysis Requested/ P.L. Item #	Sample Condition Upon Receipt

SAMPLE

a. Written results required by (date): _____ Verbal results required by (date): _____

b. QC: ☐ Standard Enseco ☐ CLP Protocol ☐ Project-Specific _____

c. Sample Disposal: ☐ Enseco ☐ Return to Client ☐ Phone RMAL

d. Raw Data Copies Needed: ☐ Yes ☐ No

e. Detection Limits: ☐ Standard Product ☐ Other*

f. Holding Times: ☐ Enseco ☐ EPA-CLP ☐ Other*

g. *Special Instructions: _____

**h. Intercompany Rebate: *(circle one)* 0% 5% 10% i. P.O. Number _____

APPENDIX E

DATA VALIDATION PROCEDURE FOR EVALUATING INORGANIC DATA

DATA VALIDATION PROCEDURE FOR EVALUATING INORGANIC DATA

Scope and Application:

This standard operating procedure (SOP) describes Warzyn's procedure for evaluating inorganic data according to procedures specified in a Quality Assurance Project Plan (QAPP). Each analytical procedure is specific to a project and is found or referenced in the QAPP. The reviewer must evaluate whether the procedure was adhered to and that the required Quality Control (QC) requirements were met as described in the QAPP and evaluate the usability of the data.

References:

1. Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses, Hazardous Site Evaluation Division, U.S. EPA, July, 1988 (or most current).

2. Statement of Work for Inorganics Analysis, U.S. EPA Contract Laboratory Program, SOW No. 788, July, 1988 (or most current).

3. Quality Assurance Project Plan (QAPP) containing the analytical procedures required for evaluation. Each QAPP is specific to a project.

Requirements:

1. It is recommended that the reviewer be familiar with the RAS Inorganics validation process (refer to the Validation Guidelines and Statement of Work above) prior to evaluating this data. The evaluation and action criteria described in this SOP are very similar to the RAS Inorganics validation process.

2. All completed validated data packages must include the following:

   a. Data Validation Narrative
   b. Validated Results; with data qualifiers added and pages stamped "VALIDATED".

NOTE: It is not necessary to include the raw data with the completed validated data package, since a copy of the raw data package already exists in the project file. Only include raw data if there are major concerns or deficiencies with the data set.

Procedure:

1. Screen the data package to see that the samples and analytes undergoing evaluation concur with what was requested on the "Request for Data Validation" form (refer to Figure 1). If discrepancies occur, contact the data management coordinator for clarification prior to continuing.

2. Review the QAPP and the specific analytical procedure for the analyte undergoing evaluation. They will contain:

   a. A summary of the method required and method reference,
   b. The applicable matrices and recommended hold times,
   c. An approximate number of samples and matrix,
   d. Special technical instructions,
   e. Required deliverables,
   f. Data Requirements (Detection Limits, Precision, etc.), and
   g. Quality Control Requirements.

3. The reviewer must evaluate the data according to the quality control criteria specified in the QAPP. The evaluation of each analyte, in most cases, will include a review of the following criteria:

   a. Methodology,
   b. Raw Data/Quality Control Deliverables,
   c. Detection Limits,
   d. Holding Times,
   e. Calibration (Initial and Continuing),
   f. Blanks (Calibration and Preparation),
   g. Laboratory Control Samples (EPA Reference Samples),
   h. Laboratory Duplicates,
   i. Matrix Spikes,
   j. Sample Result Verification,
   k. Field Duplicates,
   l. Other (as specified in the procedure),
   m. Overall Assessment for a Case.

4. Evaluate the data for the criteria above as follows:

A. METHODOLOGY

<u>Evaluation</u>

1. Examine the data to determine if the acceptable analytical methodology was used as indicated in the QAPP.

2. Determine if the analytical procedure was followed.

<u>Action</u>

1. If an acceptable analytical method was not used, contact the Project Manager. It is the Project Manager's responsibility to inform the reviewer to continue the validation process of the data as received, or to reject the data. In some cases reanalysis or

resampling may be required, however the decision is left up to the Project Manager. The Project Manager may require that the reviewer determine the "usability" of the data package as submitted prior to deciding what formal action should be taken.

2. If the analytical procedure was not followed as stated, use professional judgement in determining whether the data should be qualified as estimated or unusable.

B. RAW DATA/QUALITY CONTROL DELIVERABLES

Evaluation

1. The QAPP will specify what information should be submitted as part of the data package. Determine whether the data package submitted is complete as described in the QAPP. Usually the following will be required:
   - Method used,
   - Calibration/standardization information,
   - Bench records tabulating the order of analysis of samples, standards, blanks, duplicates and spikes with the resulting instrument readouts and final concentrations. Records submitted should be complete enough such that all results could be recalculated from this raw data.
   - Photocopy of all instrument readouts (strip charts, printer tapes, etc.)
   - Reference (QC) samples to be identified by source, lot number and sample number, with corresponding true values and 95% confidence limits provided.
   - Custody records

Action

1. If the data package is not complete, contact the data management coordinator. The data management coordinator will contact the appropriate laboratory or project manager and obtain the information necessary to complete the evaluation process.

C. DETECTION LIMITS

Evaluation

1. Evaluate whether the detection limits required in the QAPP were achieved by the method used.

Action

1. If detection limits were not reported at least to what was required in the QAPP, review the raw data to determine if the detection limits were achievable. If they were, recalculate the data and report results to the required detection limits.

2. If detection limits were not achievable by the laboratory, determine why (laboratory sensitivity problem or detection limits unobtainable for the method used. Inform the project manager of

the situation. The project manager will determine whether the evaluation should be completed.

D. HOLDING TIMES

Evaluation

1. Determine whether the established holding times were met. The holding time is established by comparing the DATE SAMPLED with the DATE OF ANALYSIS found on the raw data. Required hold times should be noted in the QAPP.

2. Examine the digestion/distillation logs to determine if samples were preserved at the proper pH.

Action

1. If hold times were not noted in the QAPP, use the EPA-recommended hold times and apply these hold times to both water and soil samples.

2. If holding times and preservation criteria are not met, qualify all results $>$ Instrument Detection Limit (IDL) and estimated (J) and results $<$ IDL as estimated (UJ).

3. Use professional judgement in cases where the holding time is grossly exceeded. The expected bias would be low and the reviewer may determine that results $<$ IDL are unusable (R).

E. CALIBRATION

Evaluation

1. Verify that the instrument was calibrated daily and each time the instrument was set up using the correct number of standards and blank. In cases where a curve set-up is not applicable (i.e. a titrimetric procedure), verify that the titrants were standardized as required in the procedure.

2. Verify that the correlation coefficient is $\geq$ 0.995 (unless otherwise specified).

3. Verify that the calibration verification standards (ICV/CCV) were run at the appropriate frequency and that results were within acceptable limits as stated in the QAPP.

4. Recalculate approximately 10% of the ICV/CCV using the following equation:

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$

Due to possible rounding discrepancies, allow results to fall within 1% of the contract windows.

Action

1. If the minimum number of standards as required in the QAPP were not used for initial calibration, or if the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R).

2. If the correlation coefficient is < 0.995 (or as specified in the QAPP), qualify results > IDL as estimated (J) and results > IDL as estimated (UJ).

3. If standardization of titrants was required and not performed, use professional judgement in qualifying the data. The reviewer should review ICV/CCV, EPA reference sample results and other criteria prior to determining if the data should be qualified as estimated (J if > IDL; UJ if < IDL) or unusable (R).

4. If the ICV/CCV %R falls outside the acceptable limits, use professional judgement to qualify associated samples. If possible, indicate the bias in the review. The following guidelines may assist the reviewer in qualifying the data:

   - If the ICV/CCV %R is significantly greater than the upper control limit (UCL), qualify associated results > IDL as unusable (R); results < IDL are acceptable.

   - If the ICV/CCV %R is significantly lower than the lower control limit (LCL), qualify all associated data as unuseable (R)

   - If the ICV/CCV %R are outside the acceptable limits, yet relatively close to the limits, qualify associated results as estimated (J if > IDL; UJ if < IDL and the %R is below the LCL).

F. BLANKS

Evaluation

1. Review the raw data for all blanks and verify the results were accurately reported.

2. No contaminants should be found in the blanks (i.e. all blank results should be < IDL).

3. Positive blank results must not be corrected by subtracting off the blank value from the sample results.

Action

1. Sample results ≥ IDL but less than 5 times the maximum concentration found in any blank should be qualified as not detected (U).

2. Any blank with a negative result whose absolute value is > IDL must be carefully evaluated to determine if the sample data is biased in any way.

G. LABORATORY CONTROL SAMPLES (EPA REFERENCE SAMPLES)

Evaluation

1. The laboratory control sample (LCS) serves as a monitor of the overall performance of the analysis including all preparation steps. Review the LCS and verify that the results fall within the control limits required. (If no limits noted, use 80-120% for the control limits.)

2. Check the raw data to verify the reported recoveries. Recalculate one or more recoveries (%R) using the following equation:

$$\text{LCS } \%R = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

Action

1. If results are < IDL and the LCS recovery is above the UCL, the data are acceptable.

2. If the LCS recovery for any analyte falls within the range of 50-LCL, or > UCL, qualify results > IDL as estimated (J).

3. If results are < IDL and the LCS recovery falls within the range of 50-LCL, qualify the affected results as estimated (UJ).

4. If the LCS recovery results are < 50%, qualify the data for the affected analytes as unusable (R).

5. If a LCS was required and not run, use professional judgement to determine whether the data is estimated (J) or unusable (R).

H. LABORATORY DUPLICATES

Evaluation

1. Review the duplicate data and verify that the results fall within the criteria required. If no criteria exist, use the limits of 20% for the Relative Percent Difference (RPD) or ± IDL if results are < 5x IDL (35 RPD or ± 2x IDL if > 5x IDL for soils).

2. Review the data and verify that the duplicate analysis was <u>not</u> performed on a field blank.

3. Check the raw data and recalculate one or more RPD using the following:

$$RPD = \frac{|S-D|}{(S+D)/2} \times 100$$

where, S = Original sample value
D = Duplicate sample value.

Action

1. If duplicate analysis results for a particular analyte fall outside the appropriate control windows, qualify the results for that analyte in all associated samples of the same sample matrix as estimated (J).

2. If the field blank was used for duplicate analysis, carefully evaluate all other quality control data and use professional judgement in evaluating the data.

I. MATRIX SPIKES

Evaluation

1. Review the matrix spike data and verify that the results fall within the limits specified. If no criteria exists, use the limits of 75-125% for both water and soil matrices.

2. Check the raw data and recalculate the % Recovery of at least 10% of the data. Use the following equation to verify that the results were calculated correctly:

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

Where, SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

3. Review the data and verify that the field blank was not used for the spike analysis.

Action

1. If the spike recovery is > 125% (or > UCL stated in the procedure) and the sample results are < IDL, the data is acceptable for use.

2. If the spike recovery is > 125% or < 75% (> UCL or < LCL stated in the procedure) and the sample results are > IDL, qualify the data for these samples as estimated (J).

3. If the spike recovery falls within the range of 30-74% (30-LCL) and the sample results are < IDL, qualify the data for these samples as estimated (UJ).

4. If any spike recovery results are < 30% and the sample results are < IDL, qualify the data for these samples as unusable (R).

5. In cases where more than one spike analysis was performed and one or more %R results were outside acceptable ranges, use professional judgement when qualifying the data. Matrix spikes should be performed on each sample matrix. Qualify data of similar matrix as the spiked sample if different matrices in the data package.

6. If the field blank was used for the spike analysis, carefully evaluate all other quality control criteria and use professional judgement in evaluating the data.

J. SAMPLE RESULT VERIFICATION

Evaluation

1. Examine the raw data to verify the analyte quantitation was calculated as stated in the procedure. Re-calculate a minimum of 10% of the sample results to verify results were calculated correctly.

2. Examine the raw data for anomalies such as baseline drift, negative absorbances (indicative of interfences), omissions, etc.

3. Verify there are no transcription or reduction errors (dilutions, percent solids, sample weights) on at least 10% of the samples.

4. Verify that all results fall within the calibrated range.

Action

1. If any discrepancies are found and can be corrected, make the corrections and note. Increase the level of quality control if necessary. If the discrepancy cannot be resolved by the reviewer, contact the data management coordinator. The laboratory or appropriate agency will be contacted and the information necessary to complete the evaluation will be requested. The project manager should be informed of the delay in the validation process. If any discrepancy remains unresolved, use professional judgement in qualifying the data.

K. FIELD DUPLICATES

Evaluation

1. Field duplicate samples measure field and lab precision and therefore the results may have more variability than lab duplicates. Review the field duplicate data and calculate the RPD. No criteria exists for evaluating the data.

Action

1. If the RPD is $>$ 50%, note in the narrative. Use professional judgement in qualifying any data. Review the raw data to verify that no reduction errors exist. This is to verify that the discrepancy is due to sampling techniques (sampling, preservation,

filtering, etc.) rather than laboratory error. Make notation of the discrepancy in the narrative.

L. OTHER (as specified in the procedure)

Evaluation

1. Review the procedure for any other quality control criteria not covered under the above sections. Examples of additional information required may be: ICP interference check samples, special AA quality control checks, distilled/digested blanks and standards, standardization, specific methodology requests for different concentration levels, etc..

Action

1. If specific quality control criteria were required and not followed, the project manager should be informed.

2. If the data was outside the acceptable ranges, use professional judgement in qualifying the data. Clearly identify your action and justification of the action in the narrative.

M. OVERALL ASSESSMENT FOR A CASE

1. It is appropriate for the reviewer to make professional judgements and express concerns and comments on the validity of the overall data package. This is particularly appropriate when several QC criteria are outside specifications. It is the reviewers responsibility to thoroughly document and explain all data validation qualifiers added to the data.

2. The following is a summary of data validation qualifier definitions which can be used in evaluating the data:

   U The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.

   J The associated value is an estimated quantity due to quality control criteria not being met.

   R The data are unusable. The analyte may or may not be present.

   UJ The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.

5. Once the data has been evaluated against all of the points described in part 4, the evaluator can prepare the data validation narrative.

The data validation narrative should be a summary of the data quality indicating any problems or deficiencies found with the data set. If

possible, on data qualified as estimated, note if the data is biased high or low. It is easiest to discuss each analyte separately. Refer to Figure 2 for the form to be used when writing the data validation narrative.

6. Record any data validation qualifiers (DVQ) to the left of the results on the analytical report. The laboratory qualifiers (LQ) will usually recorded to the right of the result by the laboratory who performed the analytical work. A copy of the lab qualifier definitions should be supplied with the data. Data entry people need to be able to distinguish between DVQ and LQ, so note location of qualifiers if located other than above.

7. Record the sample ID and date sampled as noted on the Chain of Custody on the analytical report.

8. Hilight POSITIVE-HIT data with a hilighter.

9. Stamp all of the analytical reports "VALIDATED". A "VALIDATED" stamp can be obtained from the data management coordinator.

10. Sign and date the Data Validation Request Form.

11. A quality control review needs to be performed on the validated data package. The entire validated data package (including raw data, evaluator's notes and narrative) should be submitted to the QC reviewer for a final review.

Quality Control Review:

1. The purpose of this quality control review is to verify that the data were validated according to the guidelines above, that all data validation qualifiers were added and also that the narrative is complete.

2. Review the validated data package for completeness. The data package should contain the following:

   - Data Validation Request Form
   - Inorganic Data Validation Narrative
   - Validated Analytical Results

3. Verify that results exist for all samples and all analytes requested in the data package.

4. Review the data validation narrative with the validated results for consistency, i.e., if the narrative stated that a particular analyte was qualified, verify that the qualifier has been recorded with the associated data. Check approximately 10% of the qualifiers. If discrepancies are found, increase the level of QC checks.

5. Review the narrative; check that the data has been evaluated according to the quality control criteria specified in the QAPP.

6. Verify that all positive-hit data have been hilighted and that the sample descriptions and sampling dates have been recorded on every result page.

7. Verify that the reviewer has signed and dated the validated data package.

8. If no discrepancies exist, sign and date the Data Validation Request Form. If the data package requires clarification, review the raw data and/or obtain clarification from the reviewer.

9. Deliver the final validated data package to the Data Management Coordinator when the quality control review is completed.

10. Recycle any raw data. Raw data is not submitted with the final validated data package.

INORGANICS
DATA VALIDATION NARRATIVE

Site Name _____ Project # _____

SMO Case # (SAS #) _____ #Samples/Matrix _____

Laboratory _____ Hours for Review _____

SOW # _____ EPA Validation Guidelines # _____

Sample Numbers _____

Validated By _____ Date _____

Reviewed By _____ Date _____

SUMMARY OF REVIEW:

CAW/dlk/KJD
[dlk-601-22]
60721-MD