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Soil and Groundwater Background Study Work Plan Former Laredo Air Force Base Laredo, Webb County, Texas

Contract No. DACA56-01-D-2001
Task Order No. 0004

October 2001

Prepared for:

United States Army Corps of Engineers, Tulsa District
Tulsa, OK

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Soil and Groundwater Background Study
Work Plan
Former Laredo Air Force Base
Laredo, Webb County, Texas

USACE Contract No. DACA56-01-D-2001
USACE Task Order No. 0004
E & E Project No. 001289.UX06

October 2001

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List of Acronyms

CFR	Code of Federal Regulations
DQOs	data quality objectives
DRP	data reduction plan
E & E	Ecology and Environment, Inc.
EM	Engineer Manual
EPA	United States Environmental Protection Agency
FSP	field sampling plan
GFP	government-furnished property
IWTP	industrial waste treatment plant
LAFB	Laredo Air Force Base
QAPP	quality assurance project plan
SAP	sampling and analysis plan
SGR	shotgun range
SLF	sanitary landfill
TNRCC	Texas Natural Resource Conservation Commission
USACE	United States Army Corps of Engineers

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1 Introduction and Objectives

Previous site investigations have documented contamination in soil and groundwater at the former Laredo Air Force Base (LAFB), which is classified as a Formerly Used Defense Site (United States Army Corps of Engineers [USACE] 2000a, USACE 2000b, USACE 2000c). In these site investigations, limited background samples were collected for natural- and anthropogenic-occurring analytes.

In the previous investigations, the concentrations of specific metals, pesticides, and herbicides exceeded the Texas Natural Resource Conservation Commission (TNRCC) Risk Reduction Standard No. 2 media-specific limits at the SLF, SGR, and IWTP. Data from the previous site investigations had not been compared to the TNRCC Texas Risk Reduction Program (TRRP) protective concentration levels (PCL), which became effective on May 1, 2000.

Ecology and Environment, Inc., (E & E) was tasked by the USACE, Tulsa District, under Contract No. DACA56-01-D-2001, Task Order No. 0004, to conduct a soil and groundwater background study at the former LAFB located in Laredo, Webb County, Texas.

This document, the soil and groundwater background study work plan, includes primary objectives, a project management plan, a site safety and health plan (safety plan), a data reduction plan (DRP), a sampling and analysis plan (SAP), a property management plan, and a project schedule.

The primary objectives of the background study include the following:

- Determine soil and groundwater background concentrations of metals, pesticides,and herbicides relevant to the former LAFB;
- Statistically determine the threshold concentrations of metals, pesticides, and herbicides necessary to define the extent of contamination; and
- If required, conduct supplemental statistical and risk analyses if a localized "hot spot" is encountered during the background study.

2 Project Management Plan

A coordinated team of the USACE, Tulsa District; E & E personnel; and subcontractors will implement the former LAFB soil and groundwater background study for the former LAFB. The team members, the team organization, and the lines of communication for the team are shown in Figure 2-1.

Carol Wies is the USACE, Tulsa District, government technical manager. Lisa Lawson USACE, Tulsa District and Brian Condike, USACE Ft. Worth District, are the project managers responsible for overall project execution, including project quality, cost, and schedule. John Weatherly, Jr., is the USACE, Tulsa District, contracting officer and is responsible for all contract actions with E & E.

Frances Verhalen is the E & E contract manager and will coordinate contracting activities with Mr. Weatherly. Ms. Verhalen is responsible for ensuring that the background study is performed in accordance with E & E's contract with the USACE, Tulsa District, negotiated task order. She also will provide engineering guidance and support, and will be responsible for engineering quality.

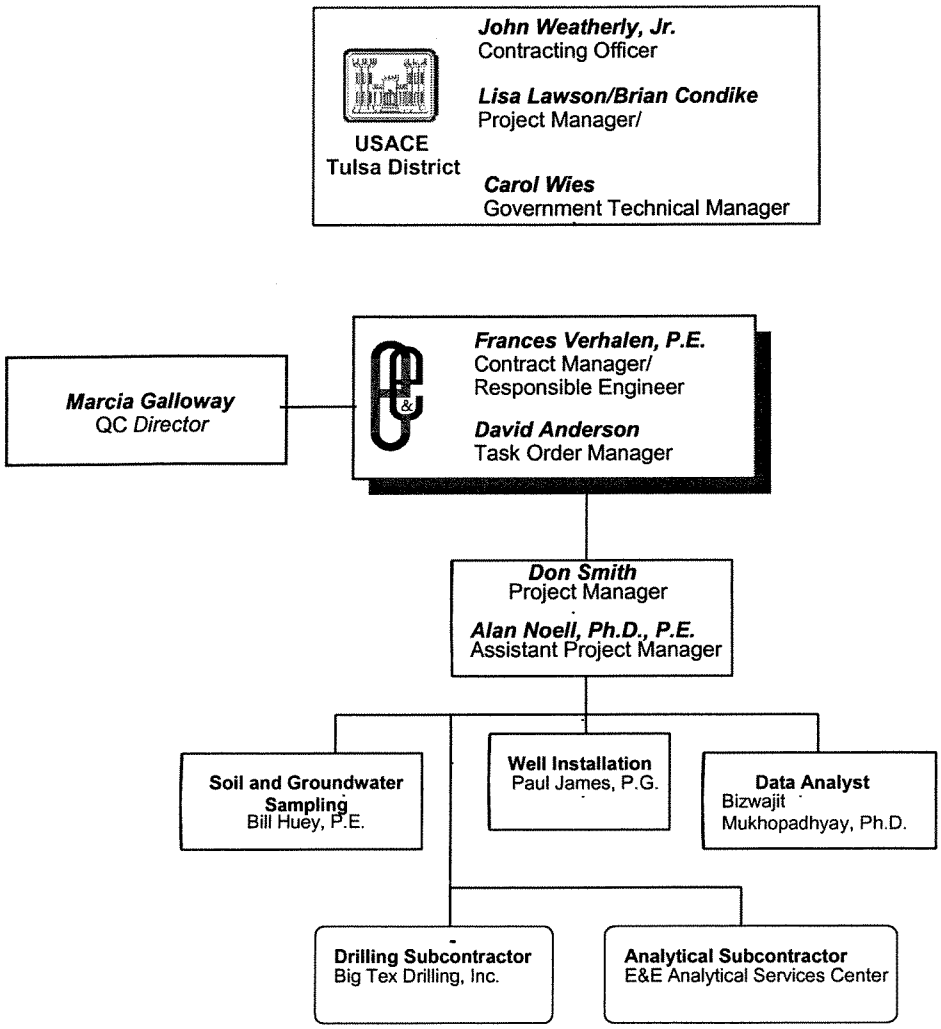
David Anderson is the E & E task order manager. Mr. Anderson is responsible for personnel, implementation of technical work, logistics, and project scheduling. He will work with Ms. Wies to resolve potential problems and initiate any modifications required to complete the project successfully.

Don Smith is the E & E project manager and will coordinate the background study activities with Ms. Wies. Mr. Smith will be responsible for coordinating specific tasks within the background study and producing final documents. He will communicate with Mr. Anderson to ensure the successful completion of project activities.

Alan Noell is the E & E assistant project manager. Dr. Noell will assist Mr. Smith in the procurement of subcontractors, coordination of project activities, and the preparation of reporting documents.

Additional project personnel and subcontractors are delineated in Figure 2-1.

Figure 2-1. Organization Chart
FORMER LAREDO AIR FORCE BASE
Laredo, Texas



3 Planning Documents

The project planning documents include a work plan, Site Health and Safety Plan, DRP, SAP, project management plan, and project schedule. The SAP includes field sampling plan (FSP) and quality assurance project plan (QAPP). The DRP, and project management plan are subsections in the work plan. The Site Health and Safety Plan, FSP, QAPP, and project schedule are provided in Appendices A, B, C, and D, respectively.

3.1 Site Health and Safety Plan

A **Site Health and Safety Plan** was prepared for the background study. The site-specific health and safety plan includes appropriate levels of personal protective equipment, exposure monitoring procedures, physical and chemical hazard task evaluations, decontamination procedures, personnel responsibilities, emergency information, contingency procedures, and other health and safety protocols. The **Site Health and Safety Plan** complies with USACE Engineer Manual (EM) 385-1-1, 29 Code of Federal Regulations (CFR) 1910.120 regarding hazardous waste operations and emergency response, and 29 CFR 1926 regarding safety and health regulations for construction.

3.2 Data Reduction Plan

E & E will utilize the guidance in *Guidance for Data Quality Assessment, Practical Methods for Data Analysis* (United States Environmental Protection Agency [EPA] QA/G-9, January 1998 [EPA 1998]), *Data Quality Evaluation Statistical Toolbox (DataQUEST)* (EPA QA/G-9D, December 1997 [EPA 1997]), and the *Handbook for Statistical Analysis of Environmental Background Data* (U.S. Navy 1999).

E & E's approach for statistically determining background concentrations is based on the assumption that the samples are representative of a single population. Preliminary estimates of the mean background concentrations of the analytes were determined using the existing data sets to estimate the relative standard deviation of the data and to specify minimum numbers of samples for the background study (USACE 2000a, USACE 2000b, USACE 2000c).

Analytical results for each soil stratum and the groundwater will be assessed separately. Before calculating the descriptive summary statistics for the data set, the distribution of the data set and the presence of outliers in the data set will be determined using the guidance from Sections 4.2 and 4.4 of EPA QA/G-9. The percentage of the samples with concentrations less than the analyte detection limit in the data set will be evaluated to select the appropriated descriptive statistics, as described in the Navy guidance. For data sets in which less than 15% of the samples have concentrations less than the analyte detection limit, E & E will determine means, medians, standard deviations and minimum and maximum values. For data sets with larger percentages of samples with concentrations less than the analyte detection limit, E & E will determine medians, trimmed means, Winsorized means and Winsorized standard deviations. E & E will use this information to verify that a sufficient number of samples were collected to meet project objectives of a 95% confidence level at a confidence interval of 2 times the quantitation limit. If all sample results for an analyte are less than the detection limit, the background will be considered as the mean sample quantitation limit. E & E will work with the analytical laboratories to insure that the reporting and detection/quantitation limits are the lowest technically feasible and are below TRRP PCLs.

The specific statistical test to compare the site and background has not been determined. Statistical tests that may be used for the evaluation include Slippage test, Quantile test or the two sample test for proportion as these tests are better suited to identify analytes that have elevated concentrations in only small areas of the site. Before beginning the evaluation, E & E will coordinate with USACE to identify critical values to use in determining whether background concentrations are exceeded.

3.3 Sampling and Analysis Plan

Specific requirements for the SAP are indicated in EM 200-1-3, *Requirements for Preparation of Sampling and Analysis Plans* (USACE n.d.a.). The SAP includes the FSP and the QAPP. The FSP includes project background, project organization and responsibilities, project scope and objectives, non-measurement data acquisition, field activities, field operations documentation, sample packaging and shipping requirements, management of investigation derived wastes, and standard operating procedures. The QAPP includes DQOs and the quality assurance/quality control requirements. DQOs are established in accordance with USACE (n.d.b.) EM 200-1-2, *Guide for Technical Project Planning Process.* The FSP and QAPP are provided in Appendices B and C, respectively.

3.4 Property Management Plan

Although E & E anticipates using rented equipment from outside vendors to complete the field tasks, the use and management of government-owned property for this site will be the responsibility of E & E. Government-owned property may be acquired through the contract or furnished by the Government.

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E & E manages government-furnished property (GFP) on multiple contracts for the federal Government in compliance with Federal Acquisition Regulations Part 45, as demonstrated by multiple audits by EPA and the Defense Contract Management Command. E & E has successfully managed GFP on various Government projects since the early 1980s.

E & E will maintain records of GFP and will record its utilization, necessary lost/damage/destruction incidents, calibration and maintenance, and condition of the equipment. A property listing of items used at the former LAFB will be prepared and submitted to the USACE, Tulsa District, monthly. For this site, E & E anticipates providing this list for a period not to exceed two months, which covers the projected field efforts.

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4 References

United States Army Corps of Engineers, n.d.a, *Requirements for Preparation of Sampling and Analysis Plans*, Engineering Manual (EM) 200-1-3.

n.d.b., *Guide for Technical Project Planning Process*, EM 200-1-2.

n.d.c., *Safety and Health Requirements Manual*, EM 385-1-1.

United States Army Corps of Engineers, Tulsa District, September 2000a, *Final Site Investigation Report, Industrial Waste Treatment Plant Site, Former Laredo Air Force Base.*

United States Army Corps of Engineers, Tulsa District, May 2000c, *Revised Site Investigation Report, Sanitary Landfill Site, Former Laredo Air Force Base.*

United States Army Corps of Engineers, Tulsa District, July2000b, *Site Investigation Report, Shotgun Range Site, Former Laredo Air Force Base.*

United States Environmental Protection Agency (EPA), January 1998, *Guidance for Data Quality Assessment, Practical Methods for Data Analysis*, EPA QA/G-9.

December 1997, *Data Quality Evaluation Statistical Toolbox (DataQUEST)*, EPA QA/G-9D.

U.S. Navy, July 1999, *Handbook for Statistical Analysis of Environmental Background Data.*

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6 Field Operations Documentation

6.1 Daily Contractor Quality Control Reports

During the field investigation, contractor quality control (CQC) reports will be prepared daily; dated; signed by the project CQC representative; and sent to the USACE, Tulsa District, government technical manager at the frequency requested by USACE, Tulsa District. The daily CQC reports will summarize the tasks performed, weather conditions at the time of sampling, field instrumentation measurements, identification of all field and control samples, departures from the approved FSP, deviations from applicable SOPs, problems encountered, and instructions from government personnel. Any deviations that may affect DQOs will be conveyed immediately to the USACE, Tulsa District, government technical manager.

If requested by the USACE, Tulsa District, government technical manager, the following forms will be attached to the daily CQC reports:

- EPA Forms II Lite generated chain-of-custody forms;
- Field-generated analytical results; and
- Any other project forms generated in the field, including well logs, geologic logs, and well development records.

An example of the daily CQC report is provided in Attachment I.

6.2 Field Activity Logbooks and/or Sample Field Sheets

Field activity logbooks will be utilized to record all pertinent information relating to field activities. The logbooks will be prepared in accordance with *Standard Operating Procedure for Field Activity Logbooks*, provided in Attachment J. Well logs, geologic logs, and well development records will be recorded on field sheets in accordance with *Standard Operating Procedure for Geotechnical Logbook/Trip Report Preparation*, provided in Attachment D.

6.3 Photographic Records

Photodocumentation will record field activities relating to drilling, monitoring well installation, groundwater sampling, soil sampling, and management of IDWs. Photo-documentation will be recorded in the field activity logbooks in accordance with *Standard Operating Procedure for Field Activity Logbooks*, provided in Attachment J.

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6.4 Sample Documentation

6.4.1 Sample Numbering System

Soil samples will be designated as *SB##-MMDDYY-##-##*. The two digits following *SB* will identify the sequential soil boring number. *MMDDYY* will designate the month, day, and year in which the sample was collected. The last two numbers will designate the depth interval, in feet, from which the sample was collected. The letters *QC* will follow the sample number to indicate replicate samples; e.g., *SB01-100101-0.5-2-QC*. All samples submitted to the laboratory will be cross-referenced with sequential numbers; i.e., *L###*. The cross-references will be written in the field activity logbooks. All samples submitted to the laboratories will be identified with a unique, sequential number which will be cross-referenced in the logbook to the sample numbers described above. The laboratories will not see the descriptive sample number.

Groundwater samples will be designated as *BGMW-#-MMDDYY*. The digit following *BGMW* will identify the background monitoring well number. *MMDDYY* will designate the month, day, and year in which the sample was collected. The letters *QC* will follow the sample number to indicate replicate samples; e.g., *BGMW-1-100101-QC*. All samples submitted to the laboratory will be cross-referenced with sequential numbers; i.e., *L###*. The cross-references will be written in the field activity logbooks. All samples submitted to the laboratories will be identified with a unique, sequential number which will be cross-referenced in the logbook to the sample numbers described above. The laboratories will not see the descriptive sample number.

6.4.2 Sample Labels and/or Tags

Samples will be labeled and sealed in accordance with the SOPs for soil sampling and groundwater well sampling, provided in Appendices H and E, respectively.

6.4.3 Chain-of-Custody Records

As discussed in the SOPs for soil sampling and groundwater well sampling, chain-of-custody forms will be completed to document the types and number of samples collected and logged. The chain-of-custody forms will be included in the sample shipments in accordance with *Standard Operating Procedure for Sample Packaging*, provided in Attachment F.

6.5 Field Analytical Records

Field analytical records will be generated for the development of the monitoring wells, as discussed in Section 5.1.2.3.10. As discussed in Section 5.1.2.4.2, the monitoring well development records will be included in the appendices of the background study report.

Field analytical procedures also will be performed to preliminarily classify IDWs. Records of these results will be recorded in the field activity logbooks. The management of IDWs is discussed in Section 8.

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6.6 Documentation Procedures and Data Management and Retention

Analytical data generated from the analysis of the soil and groundwater samples will be validated as directed by the QAPP. The analytical results and the corresponding data qualifier flags will be summarized in the background study report.

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7 Sample Packaging and Shipping Requirements

Soil and groundwater samples will be collected and prepared in accordance with the SOPs for soil sampling and groundwater well sampling, provided in Appendices H and E, respectively. Samples will be sent to the ASC in accordance with *Standard Operating Procedure for Sample Packaging*, provided in Attachment F.

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8 Investigation-Derived Wastes

8.1 Soil and Drill Cuttings

Soil cuttings from the hollow-stem-auger drilling of the four new monitoring wells (see Figure 5-1 for locations) and excess sample soil from the sampling devices will be drummed separately, one or more drums per well. All applicable information will be labeled on each drum. Because the proposed locations are intended to be in clean background areas, the drilling-related wastes initially will be considered nonhazardous. If visual and photoionization detector (PID) field screening data regarding the formations penetrated by the well indicate that the soil may be contaminated, the soils from the drum will be sampled for the contaminants of concern to determine disposal options. Soils will be disposed of in the most cost-effective legal method possible, including scattering the cuttings around the drilling site if acceptable to site representatives.

Copies of all disposal paperwork will be included as an appendix to the background study report.

8.2 Liquid Investigation-Derived Waste

The subcontract drilling rig and hollow-stem augers will be decontaminated before initial use and between boring locations. Drill rig and auger decontamination will be accomplished by washing with a heated water spray (steam cleaner). Split-spoon and core barrel samplers will be decontaminated before first use and between subsequent uses by washing in a trisodium phosphate and water solution and rinsing in water. All decontamination-derived water will be drummed and labeled properly.

If visual and PID field screening data regarding the formations penetrated by the well indicate that the soil may be contaminated, the decontamination water from the appropriate drum(s) will be sampled for the contaminants of concern to determine disposal options. Liquid IDW will be disposed of in the most cost-effective legal method possible. The driller will construct a plastic-lined pit for the collection of liquid decontamination waste. The decontamination water will be pumped into drums . The disposal of the decontamination waste water will be the responsibility of the driller. Should the results of the analyses indicate that the soil and therefore the decontamination water from the site is contaminated, the waste water will be disposed of legally with the IDW soil cuttings. If clean, water may be discharged to the surface or to storm drains at the facility.

Copies of all disposal paperwork will be included as an appendix to the background study report.

8.3 Miscellaneous Investigation-Derived Wastes

Soiled personal protective equipment, including Tyvek, sampling gloves, and wipes, will be drummed and labeled. Because all sampling locations are expected to be uncontaminated, the miscellaneous IDWs are expected to be nonhazardous. If IDWs are suspected to be hazardous, the miscellaneous IDWs will be sampled and analyzed for the chemicals of concern.

Miscellaneous IDWs will be disposed of in the most cost-effective legal method possible. Copies of all disposal paperwork will be included as an appendix to the background study report.

9 Field Assessment and Three-Phase Inspection Procedures

E & E will ensure that quality is maintained during the fieldwork by means of a three-phase control process complying with Engineer Regulation-1180-1-6 and Corps of Engineers Guide Specifications 01450.

9.1 Contractor Quality Control

CQC preparatory, initial and follow-up phases will be performed on site by E & E's assigned QC officer. A daily CQC report will summarize the activities of each CQC phase for each definable feature of work. Definable features of work will include monitoring well boring, monitoring well installation, monitoring well development, monitoring well purging and sampling, soil sampling, management and sampling of IDWs, and sample shipment. An example of the daily CQC report is provided in Attachment I.

9.1.1 Contractor Quality Control for Definable Phases

9.1.1.1 Monitoring Well Boring

During monitoring well boring, the QC officer will document all pertinent information to show that the proper tasks were performed and the borings were completed to the proper depths. A summary of this information will be included in the daily CQC report. The boring of the monitoring wells will be photodocumented, and photodocumentation will be presented as an appendix to the background study report.

9.1.1.2 Monitoring Well Installation

During each monitoring well installation, the QC officer will document all pertinent information to show that the proper tasks were performed, the monitoring well was completed to the proper depth, the screen interval was installed properly, the filter pack was installed properly, the bentonite seal thickness and quality are acceptable, and the surface completion was installed properly. A summary of this information will be included in the daily CQC report. The installation of the monitoring wells also will be photodocumented and photodocumentation will be presented as an appendix to the background study report.

9.1.1.3 Monitoring Well Development

During the development of the monitoring wells, the QC officer will document all pertinent information to show that the proper tasks were performed and that the monitoring wells were developed properly. A summary of groundwater quality measurements will be presented in the daily CQC report. The development of the monitoring wells will be photodocumented, and photodocumentation will be presented as an appendix to the background study report.

9.1.1.4 Monitoring Well Purging and Sampling

During purging and sampling of the monitoring wells, the QC officer will document all pertinent information to show that the proper tasks were performed and the wells were purged and sampled properly. A summary of groundwater quality measurements will be presented in the daily CQC report. The purging and sampling of the monitoring wells will be photodocumented, and photodocumentation will be presented as an appendix to the background study report.

9.1.1.5 Soil Sampling

During soil sampling, the QC officer will document all pertinent information to show that the proper tasks were performed and that the borings were completed to the proper depths. A summary of this information will be included in the daily CQC report. Soil sampling will be photodocumented, and photodocumentation will be presented as an appendix to the background study report.

9.1.1.6 Management and Sampling of Investigation-Derived Wastes

The QC officer will document that proper procedures regarding management of IDWs were performed. A summary of this information will be included in the daily CQC report. The management and sampling of IDW will be photodocumented, and photodocumentation will be presented as an appendix to the background study report.

9.1.1.7 Sample Shipment

The QC officer will document that proper procedures regarding sample shipment were performed. A summary of this information will be included in the daily CQC report.

9.2 Sampling Apparatus and Field Instrumentation Checklist

The QC officer will document that all proper equipment was available for each definable task. A summary of this information will be included in the daily CQC report.

10 NonConformance and Corrective Actions

This section details the actions to be taken in the event that a nonconformance is discovered. Nonconformances may be discovered by the QC officer while personnel are still in the field, by the laboratory upon receipt of samples, or during any phase of auditing.

10.1 NonConformance Discovered in Field

The field nonconformances discoverable by the QC officer may include, but are not limited to, improper sampling procedures, improper instrument calibration, incomplete or improper sample preservation, and problems with the chain of custody.

10.1.1 Improper Sampling Procedures

The QC officer will have copies of all SOPs and will be responsible for insuring compliance with these procedures.

10.1.2 Improper Instrument Calibration

The QC officer will review all instrument calibration records as soon as possible after calibration. If any instrument is not calibrated properly, he or she will ensure that proper calibration is achieved before instrument use. If the instrument will not calibrate properly, he or she will secure one that will calibrate properly.

10.1.3 Improper Sample Preservation

The QC officer will ensure that the field sampling team will carry out all proper sample preservation steps.

10.1.4 Chain-of-Custody Problems

The QC officer will review the chain-of-custody forms before sample shipment. Any problems with project identification, billing number, sample identification, or analytical procedure requests will be noted and corrected by the sampling team.

10.2 NonConformance Discovered by Laboratory

Nonconformances discovered by the laboratory may include, but not be limited to, improper sample preservation, missing custody seals, and problems with the chain of custody.

10.2.1 Improper Custody Seals and Sample Preservation

The laboratory will verify that all samples have proper custody seals and have been preserved properly. If the laboratory personnel notice any nonconformances, they will contact the E & E project manager. The E & E project manager will contact the government technical manager with information regarding the nonconformance. If the government technical manager requests, the E & E project manager will direct proper resampling.

10.2.2 Chain-of-Custody NonConformance

If the laboratory personnel notices any nonconformances regarding the chain of custody, they will contact the E & E project manager. The E & E project manager will correct any chain-of-custody nonconformance.

10.3 NonConformance Discovered by Audit

Any nonconformances discovered during an audit should be reported to the E & E project manager. The E & E project manager will ensure that the nonconformance is corrected.

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11 References

Klemt, W.B., G.L. Duffin, and G.R. Elder, 1976, *Ground-Water Resources of the Carrizo Aquifer in the Winter Garden Area of Texas*, Texas Water Development Board Report 210, two volumes.

McCoy, T.W., 1991, *Evaluation of the Ground-water Resources of the Western Portion of the Winter Garden Area*, Texas, Texas Water Development Board Report 334, 64 pages.

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Field Sampling Plan
Attachment A
Standard Operating Procedure for Overburden Monitoring Well/Piezometer Installation

TITLE:	OVERBURDEN MONITORING WELL/ PIEZOMETER INSTALLATION
CATEGORY:	ENV 3.4
REVISED:	March 1998

STANDARD OPERATING PROCEDURE

OVERBURDEN MONITORING WELL/ PIEZOMETER INSTALLATION

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TITLE:	OVERBURDEN MONITORING WELL/ PIEZOMETER INSTALLATION
CATEGORY:	ENV 3.4
REVISED:	March 1998

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TITLE:	OVERBURDEN MONITORING WELL/ PIEZOMETER INSTALLATION		
CATEGORY:	ENV 3.4	REVISED:	March 1998

1. Introduction

1.1 Scope

This document outlines the procedures to be followed by the subcontract drilling company for the drilling and installation of overburden monitoring wells and piezometers. It has been developed to supplement standard operating procedures currently used by Ecology and Environment, Inc., (E & E).

1.2 Objectives

Overburden monitoring wells and piezometers are installed to allow for the collection of groundwater samples for chemical and physical analysis, to aid in the definition and tracking of contaminants in the groundwater, and to provide access to groundwater for elevation measurement aquifer testing.

2. Selection Criteria for Wells and Piezometers

2.1 Data Objectives

The design and construction of an overburden monitoring well, or piezometer, must take into account the client's data objectives. Construction techniques and materials must not alter the chemical or physical properties of the groundwater being sampled, and must provide samples that are truly representative of the aquifers sampled. An understanding of the site geology and chemical properties of suspected contaminants is a prerequisite for the proper selection of construction techniques and materials.

2.2 Material Specifications

The type of material used in overburden monitoring wells and piezometers can have a detrimental effect on the quality of the sample to be collected. The materials of choice should retain their structural integrity for the duration of the monitoring program under actual subsurface conditions. The materials should neither adsorb nor leach chemical constituents that would bias the collection of representative samples.

The most commonly used materials for screen and riser sections include: thermoplastics such as polyvinyl chloride (PVC) and acrylonitrile butadiene styrene (ABS); fluoropolymers such

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as polytetrafluoroethylene/tetrafluoroethylene (PTFE/TFE or Teflon) and fluorinated ethylene propylene (FEP or Neflon); metallic such as carbon steel, galvanized steel and stainless steel; and fiberglass-reinforced such as fiberglass-reinforced epoxy (FRE) and fiberglass-reinforced plastic (FRP). Each material has advantages and disadvantages relating to factors such as cost, long-term structural integrity, chemical inertness under site conditions (including exposure to acids, caustic substances, and various organic solvents), and sorptive properties. The majority of overburden monitoring wells and piezometers are constructed from 2-inch inside diameter (I.D.) pipe; however, 4- and 6-inch I.D. wells are also common. The sections of pipe should be joined by flush threaded couplings to form watertight unions that retain 100% of the strength of the pipe. The bottom of the screen should be fitted with a threaded plug. No solvents or cements should be used in the construction of the wells.

2.2.1 Thermoplastics

Thermoplastics are lightweight, relatively inexpensive, and generally have good-to-excellent chemical resistance (fluoropolymers have the best chemical resistance, except for fluorinated solvents; PVC has poor resistance to high concentrations of aromatic hydrocarbons, esters, and ketones). Thermoplastics are generally weaker, less rigid, and more temperature-sensitive than metallic materials (ABS has low strength and less heat resistance compared to PVC). PVC may also absorb some constituents from groundwater, and might react with and leach some constituents into groundwater.

2.2.2 Fluoropolymers

Fluoropolymers are expensive when compared to PVC and may not be readily available. PTFE/TFE has a lower tensile strength than PVC and the screen slot opening might decrease in size over time. The anti-stick properties of fluoropolymer materials make it difficult to achieve an annular seal with cement grout, creating the potential for the alteration of groundwater chemistry by percolating surface water, or groundwater movement between water-producing zones.

2.2.3 Metallic

The advantages of metallic well construction materials are their high strength and availability. Stainless steel has the least adsorption of halogenated and aromatic hydrocarbons, and resists degradation by corrosion and oxidation. Stainless steel is heavier and more expensive than plastics, although the potential exists for stainless steel to corrode and leach some chromium into highly acidic water, and in addition, might act as a catalyst in some organic reactions. Carbon steel rusts easily under saturated conditions, providing highly sorptive surfaces for many metals, and deteriorates in corrosive environments.

2.2.4 Fiberglass-Reinforced

Fiberglass-reinforced plastics approach the strength of stainless steel, are lightweight and relatively inert in most monitoring well environments. Unfortunately, fiberglass-reinforced plastics are not readily available, and little data is available on its performance in the field.

2.2.5 Center Guides

If casing or screen assemblies exceeding 40 feet in length are installed in holes having an annulus 2 inches or larger than the outside diameter of the casing, center guides (centralizers) can be used to ensure that the screen and riser are centered in the borehole. If used, the guides should be placed at the bottom of the string and at 20-foot intervals.

2.2.6 Well Screen Characteristics

All well screens should allow groundwater to enter the monitoring well or piezometer for sampling. Well screens of appropriate length and slot size are attached to solid riser sections and placed at the desired sampling depth in the aquifer. The slot size is selected to maximize open area for water to flow through, and minimize entry of fines into the well during pumping. The major types of well intake screens are factory-slotted, continuous-slot, bridge-slot, and shutter-type. Other types include field-slotted pipe, in which slots are manually cut, and wire-wound perforated pipe. The slot size most commonly used by E & E in overburden monitoring wells and piezometers measures 0.010 inches in width, but can be as large as 0.080 inches in width for recovery wells. It is recommended that factory-slotted screens be specified to insure the integrity of the well screen.

2.2.7 Filter Pack

The filter pack increases hydraulic conductivity around the well screen and inhibits fine particles from entering the well screen during groundwater sampling. The filter pack must be clean (to minimize loss of material during development), have well-rounded grains (to increase hydraulic conductivity, porosity, yield, and effectiveness of well development), have 90 to 95% quartz grains (to minimize changes to groundwater chemistry and to eliminate loss of volume by dissolution of minerals), and have a uniform coefficient of 2.5 or less (to minimize separation during installation and lower head loss). E & E has found that a sandpack consisting of a No. 3 (00N or 3Q) quartz sand generally meets the formation requirements when used in conjunction with 0.010-inch well screens.

2.2.8 Well Seal

To prevent the vertical movement of surface water into the overburden monitoring well or piezometer, and to prevent cement grout from entering the filter pack, a bentonite seal is placed on top of the filter pack. The bentonite seal is normally two linear feet thick and can be used either as unhydrated pellets or chips with water added later, or pumped down through tremie

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pipe as a slurry. The bentonite should be tested for inorganic contaminants or should be certified by the manufacturer as meeting the ASTM D5092-90 specifications.

The annulus above the bentonite seal should be filled with a cement bentonite grout. The typical cement grout consists of Portland Type I or II cement and 3 to 6% bentonite powder.

3. Overburden Well Installation

3.1 Inspection and Cleaning of Drilling Equipment

Proper cleaning of the drill rig and down hole equipment upon arriving at the site and between drilling locations is necessary to minimize the potential introduction of contaminants into the groundwater. Care should be taken to steam clean the drill rig and all other equipment and well components before they are used. The drill rig should be checked repeatedly for oil and hydraulic fluid leaks. These precautions are essential to ensure that trace contaminants from the drilling process are not introduced to the monitoring well.

3.2 Installation Procedures

The monitoring well or piezometer will be installed in a borehole drilled and sampled as outlined in Standard Operating Procedures for Borehole Installation (GEO 4.7). Unless otherwise indicated, the monitoring well will be installed in such a manner as to allow a minimum of 2 feet of screen to extend above the static groundwater level. The screen and riser sections will be decontaminated as outlined in the work plan. (At a minimum, the screen and riser sections will be washed with a high-pressure washer/steam and allowed to air dry.) The plug will be installed in the bottom of the first screen section placed inside the hollow stem auger. The first section of screen will be lowered until 2 feet of screen extends above the top of the augers, with a well clamp used to hold the screen in place. Additional screen and riser sections will be added until the well has reached its designed depth. The top of the casing will extend 2 to 3 feet above ground elevation, except when the well is flush-mounted.

The augers will be raised in 2-foot increments and the filter pack installed in the annulus. This process will continue until the filter pack extends at least 2 linear feet above the screened interval. The sand may bridge in the annulus if the sand is poured too quickly, requiring a water flush to clear the bridge.

After the filter pack has been installed and measurements have been taken to ensure the proper location of the filter pack, at least 2 linear feet of bentonite pellets or bentonite slurry will be placed on top of the filter pack. If the bentonite seal is above the water table, then water must be added during the placement of the seal to hydrate the bentonite at a rate of approximately one gallon of water for every two pounds of bentonite.

After the bentonite seal is in place, has been allowed to set, and measurements have been taken to ensure its proper location, a cement and bentonite grout will be tremied in place from the top of the seal to the land surface. This will be accomplished in such a manner that a tight,

continuous grout seal is present through the entire interval. Cement and bentonite mixtures will consist of potable water, bentonite powder, and Type I portland cement with 94 pounds of cement and 5 pounds of bentonite per 6.5 gallons of water. The grout will be allowed to set at least 24 hours before the well is developed.

3.3 Well Security

If the well is to be completed using aboveground casing, the riser will extend 2 to 3 feet above land surface. A vented cap will be installed in the top of the riser. The riser will be covered by a lockable steel casing that is centered around the riser. The steel casing should be fitted with a locking cap and be of sufficient diameter to allow easy access to the well riser. The steel casing will be seated in a 2-foot by 2-foot by 4-inch concrete surface pad. In order to minimize the effects of frost action, the concrete pads will be constructed with galvanized steel-reinforcing fibers which will impart a greater durability to the well pad. The pad will be sloped away from the steel casing. Drain hole(s) will be drilled in the steel casing to facilitate drainage.

If the above-grade well is in a location accessible to vehicular traffic, three 4-inch diameter by 5-foot long cement filled steel guard posts will be installed radially around the well. The guard posts will be recessed approximately two feet into the ground and set in concrete. The post will be placed around, but not in, the concrete pad placed at the well base.

4. Overburden Well Development

4.1 Rationale

During the drilling process, fine-grained materials are forced into the open borehole, forming a mudcake that reduces the hydraulic conductivity of the materials opposite the screened portion of the well. Proper development of the well or piezometer removes fines from the filter pack, which improves the hydraulic performance and eliminates or reduces the amount of sediment in water samples. Proper development also rectifies damage done during drilling to the borehole walls and adjacent formation.

4.2 Methods

Following well completion and set-up of grout, the well will be developed to remove the residuals from the annular wall spaces and to re-establish the normal hydraulic properties of the water-bearing zone. Each overburden well and piezometer will be developed by bottom-filling bailer, surging, or pumping until the minimum three well volumes of water as recommended by the American Society for Testing and Materials (ASTM) has been removed. Routine field tests will be conducted on the development water to determine turbidity, specific conductivity, pH, and temperature. Development will be considered completed when these field test parameters

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have stabilized and no further improvement in the turbidity of the development water is observed.

5. Disposal of Drill Cuttings and Fluids

5.1 Containerization of Drill Cuttings and Fluids

Drill cuttings, drill fluids, and development water must be handled as outlined in the work plan for the site. In most instances, the drill cuttings are classified as hazardous waste under the Resource Conservation and Recovery Act (RCRA) and must be placed in U.S. Department of Transportation (DOT) approved 55-gallon steel drums pending analysis. Drums containing drill cuttings must be properly labeled and marked with the source of the drill cuttings (i.e., "MW-2") prior to being staged. Drill fluids and development water must also be placed in DOT-approved 55-gallon steel drums pending analysis. The drums of drill fluids and development water must be properly labeled and marked with the source of the fluids and the date that the material was generated prior to being staged.

5.2 Disposal of Drill Cuttings and Fluids

Upon receipt of the analytical results, the drill cuttings, drill fluids, and development water can be properly classified. It is the responsibility of the property owner and/or client to arrange for the disposal of the drill cuttings and fluids at an approved facility.

6. Well Completion Reports

6.1 Well Construction Summary Sheets

At the completion of the overburden monitoring well or piezometer, a well construction summary sheet will be completed by the driller. An example of the well construction summary sheet used by E & E is attached as Figure 1. The summary sheet summarizes details of the well construction (i.e., screened interval, water level, filter pack height, bentonite placement, etc.).

6.2 Well Completion Cost Sheet

At the completion of the fieldwork, a cost sheet outlining the costs associated with the installation of the overburden monitoring well or piezometer will be completed. An example of a cost sheet is attached as Figure 2.

WELL INSTRUMENTATION LOG

Project Name: _____ Project Number: _____

Client: _____ Location: _____

Start Date: _____ Completion Date: _____

Drilling Method: _____ Driller: _____

Development Method: _____ Development Duration: _____

Water Level in Completed Borehole (ft/m): _____

Split Spoon Size:

Depth _____

_____

_____

Shelby Tube Size: _____

Depths: _____

_____

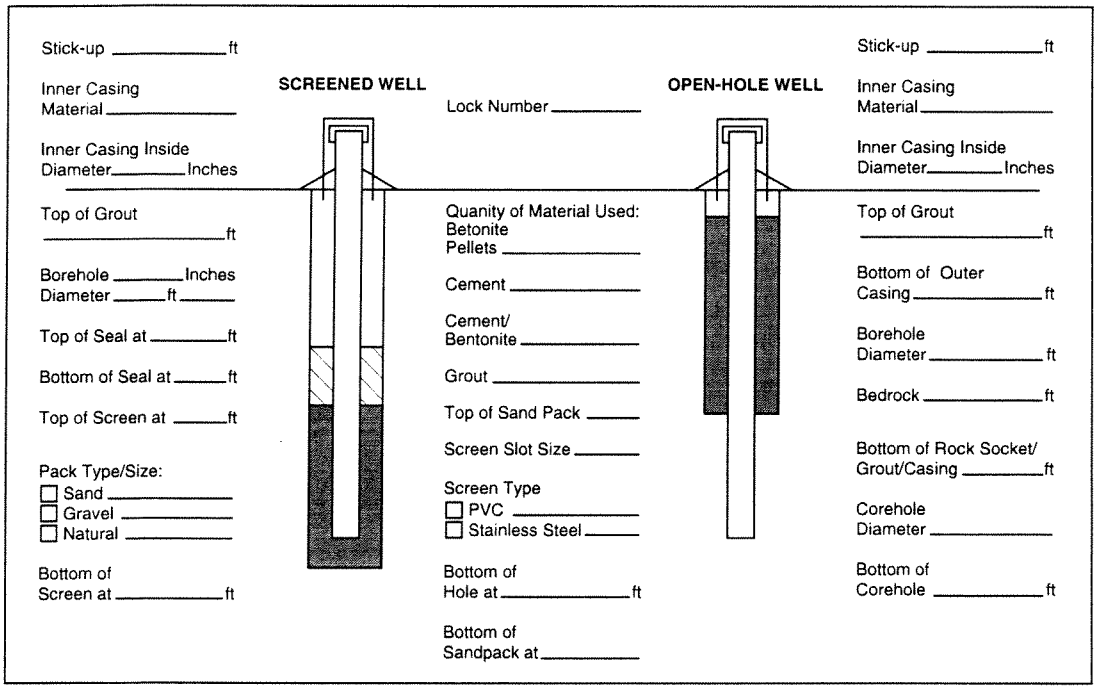

Figure 1 Well Construction Summary Sheet

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COST SHEET FOR DRILLING, SAMPLING, AND INSTALLATION
OF TWO-INCH I.D. MONITORING WELLS

Client: _____

Job Number: _____ Prepared by: _____ Date: _____

Bid Item	Description	Unit Price	Estimated Quantities	Totals
1	Mobilization and Demobilization	$4.00/Mile or $400.00	_____ Miles	_____
2	Soil Boring (4.25" HSA)	$12.00/LF	_____ LF	_____
2a	Soil Boring (6.25" HSA)	$15.00/LF	_____ LF	_____
3	Well Installation	$13.00/LF	_____ LF	_____
4	Split Spoon Samples Every 5 Feet	$20.00/ Sample	_____ Samples	_____
5	Split Spoon Samples Continuous	$10.00/Foot	_____ Feet	_____
6	Rock Coring/Concrete Coring	$30.00/LF	_____ LF	_____
7	Core Boxes	$40.00 ea	_____	_____
8	2-Inch I.D. PVC Riser	$3.75/LF	_____ LF	_____
9	2-Inch I.D. PVC Screen (0.010")	$6.75/LF	_____ LF	_____
10	8-Inch I.D. Carbon Steel Casing	$24.00/LF	_____ LF	_____
10a	4-Inch I.D. Carbon Steel Casing	$19.00/LF	_____ LF	_____
11	Plugs and Caps, PVC	$4.44 ea	_____	_____
11a	Plugs and Caps, SS	$30.00 ea	_____	_____

Figure 2 Well Completion Cost Estimate Sheet

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Bid Item	Description	Unit Price	Estimated Quantities	Totals
12	Sand	$6.00/LF	_____ LF	_____
13	Bentonite (Pellets)	$15.00/LF	_____ LF	_____
14	Cement (Boreholes)	$5.00/LF	_____ LF	_____
15	Cement/Bentonite (Wells) (10%) Grout	$6.00/LF	_____ LF	_____
16	Shelby Tube, 3" by 30"	$100.00 ea	_____	_____
17	Well Development (1.5 Hours/Well)	$150.00	_____ Wells	_____
18	Decontamination Time	$100.00/HR	_____ HR	_____
19	Furnish and Installed Flush Well Protector	$500.00/Well	_____ Wells	_____
20	Reaming to 5⅞ Inches	$26.00/LF	_____ LF	_____
21	Bentonite Seal Between Auger and Bedrock	$36.00/LF	_____ LF	_____
22	Decon Pad Construction	$100.00/Pad	_____ Pads	_____
23	Standby Time	$85.00/HR	_____ HR	_____
24	Well Abandonment (2.5 Hours/Well)	$250.00 ea	_____ Wells	_____

This cost sheet represents a summary of the estimated drilling costs associated with the installation of monitoring wells and/or soil borings at the location noted above. Any questions should be brought to the attention of E & E as soon as possible.

Subtotal _____

Discount _____

Total _____

Figure 2 Well Completion Cost Estimate Sheet

7. References

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Field Sampling Plan
Attachment B
Standard Operating Procedure for Geologic Logging

TITLE:	GEOLOGIC LOGGING
CATEGORY:	GEO 4.8
REVISED:	March 1998

STANDARD OPERATING PROCEDURE

GEOLOGIC LOGGING

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TITLE:	GEOLOGIC LOGGING		
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TITLE:	GEOLOGIC LOGGING		
CATEGORY:	GEO 4.8	REVISED:	March 1998

1. Introduction

Geologic logging involves keeping detailed records during the drilling of boreholes, the installation of monitoring wells, and the excavation of test pits, and entering the geologic descriptions of the soil and rock samples recovered on a standardized form. E & E has adapted a standardized geotechnical logbook (see DOC 2.4 in E & E's Standard Operating Procedures [SOPs]) that contains items deemed important to record when installing monitoring wells, piezometers, and/or soil borings. This document discusses general procedures for completing geologic logs.

2. Drilling Logs

2.1 Basic Documentation

When drilling boreholes, the project geologist should maintain a log that describes each borehole. The E & E Geotechnical Logbook contains records for boreholes. The following basic information should be entered on the heading of each drilling log sheet (see Figure 1):

- Borehole/well number;
- Project name;
- Site location;
- Dates and times that drilling was started and completed;
- Drilling company;
- E & E geologist's name;
- Drill rig type used to drill the borehole;
- Drilling method(s) used to drill the borehole;

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DRILLING LOG FOR _____

Project Name _____

Site Location _____

Date Started/Finished _____

Drilling Company _____

Driller's Name _____

Geologist's Name _____

Geologist's Signature _____

Rig Type(s) _____

Drilling Method(s) _____

Bit Size(s) _____ Auger Size(s) _____

Auger/Split Spoon Refusal _____

Total Depth of Borehole is _____

Total Depth of Corehole is _____

Water Level (TOIC)		
Date	Time	Level (Feet)

Well Location Sketch

Depth (Feet)	Sample Number	Blows on Sampler	Soil Components CL SI S GR	Rock Pile	Penetration Times	Run Number	Core Recovery	RQD	Fracture Sketch	HNu/OVA (ppm)	Comments
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											

Figure 1 Drilling Log

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- Bit and auger size(s);
- Depth of auger/split barrel sampler refusal;
- Total depth of borehole;
- Total depth of corehole (if applicable);
- Water level at time of completion measured from top of inside casing (TOIC); and
- A well location sketch.

2.2 Technical Information

During the drilling of a borehole, specific technical information about the unconsolidated material and rock encountered should be recorded on the drilling log sheet. The following minimum technical information should be recorded:

- Depth that sample was collected or encountered;
- Sample number assigned (if applicable);
- The number of blow counts required to drive the split barrel sampler 2 feet at 6-inch intervals (see Table 1);
- Description of soil components (see Figure 2);
- Description of rock profile (see Figure 3);
- Rock qualitative designation (RQD) (see Figure 4);
- Rock penetration time;
- Core run number (if applicable) and percent recovery; and
- Organic vapor readings in the sample.

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Table 1 STANDARD PENETRATION TEST FOR SOIL DENSITY	
N-Blows/Feet	**Relative Density**
Cohesionless Soils	
0 - 4	Very loose
4 - 10	Loose
10 - 30	Medium
30 - 50	Dense
50	Very dense
Cohesive Soils	
2	Very soft
2 - 4	Soft
4 - 8	Medium
8 - 15	Stiff
15 - 30	Very stiff
30	Hard

3. Soil Classification

Soils should be described using the Unified Soil Classification System (USCS) in the narrative lithologic description section of Figure 5. Figure 6 is a summary of the American Society for Testing and Materials (ASTM) criteria for describing soils. Soil descriptions should be concise, stressing major constituents and characteristics, and should be given in a consistent order and format. The following order is recommended by the ASTM:

1. Soil name. The basic name of the predominant constituent and a single-word modifier indicating the major subordinate constituent.

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SOIL AND SEDIMENT CLASSIFICATION

GRAIN-SIZE SCALE (Modified Wentworth Scale)

phi	mm	inches	U.S. Standard Sieve Series	Grade Name		
-12	4096	161.30				
-11	2048	80.60		very large		
-10	1024	40.30		large	Boulders	
-9	512	20.20		medium		
-8	256	10.10		small		
-7	128	5.00		large	Cobbles	GRAVEL
-6	64	2.52	63.0 mm	small		
-5	32	1.26	31.5 mm	very coarse		
-4	16	0.63	16.0 mm	coarse		
-3	8	0.32	8.0 mm	medium	Pebbles	
-2	4	0.16	No. 5	fine		
-1	2	0.08	No. 10	very fine		
0	1	0.04	No. 18	very coarse		
+1	0.500		No. 35	coarse	Sand	SAND
+2	0.250		No. 60	medium		
+3	0.125		No. 120	fine		
+4	0.062		No. 230	very fine		
+5	0.031			coarse	Silt	
+6	0.016			medium		
+7	0.008			fine		MUD
+8	0.004			very fine		
+9	0.002			coarse		
+10	0.001			medium		
+11	0.0005			fine		
+12	0.00025			very fine		CLAY SIZE

PROPORTIONS USED IN DESCRIBING SOILS

Trace - Particles are present but estimated to be less than 5%
Few - 5 to 10%
Little - 15 to 25%
Some - 30 to 45%
Mostly - 50 to 100%

i.e.: Sand with a trace of silt = 95% Sand, < 5% Silt

(When sampling gravelly soils with a standard split spoon, the true percentage of gravel is often not recovered due to relatively small sample diameter.)

Soil Density from Standard Penetration Test (ASTM D1586)

Granular Soils	Cohesive Soils
0 - 10: Loose	0 - 4: Soft
10 - 30: Medium Dense	4 - 8: Medium Stiff
30 - 50: Dense	8 - 15: Stiff
Over 50: Very Dense	15 - 30: Very Stiff

CLASSIFICATION CHART(United Soil Classification System)

MAJOR DIVISIONS		Symbols	TYPICAL NAMES
Coarse Grained Soils (More than 1/2 of soil > no 200 sieve size)	GRAVELS (More than 1/2 of coarse fraction > No. 4 sieve size)	GW	Well-graded gravels or gravel-sand mixtures, little or no fines
		GP	Poorly graded gravels or gravel-sand mixtures little or no fines
		GM	Silty gravels, gravel-sand-silt mixture
		CC	Clayey gravels, gravel-sand-clay mixtures
	SANDS (More than 1/2 of coarse fraction < No. 4 sieve size)	SW	Well-graded sands or gravelly sands, little or no fines
		SP	Poorly graded sands or gravelly sands, little or no fines
		SM	Silty sands, sand-silt mixtures
		SC	Clayey sands, sand clay mixtures
Fine Grained Soils (More than 1/2 of soil < no 200 sieve size)	SILTS AND CLAYS LL< 50	ML	Inorganic silts, and very fine sands, rock flour, silty or clayey fine sands or clayey silts with slight plasticity
		CL	Inorganic clays, of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays
		OL	Organic silts and organic clays of low plasticity
	SILTS AND CLAYS LL> 50	MH	Inorganic silts, micaceous or diatomaceous, fine sandy or silty soils, elastic silts
		CH	Inorganic clays of high plasticity, fat clays
		OH	Organic clays of medium to high plasticity, organic silty clays, organic silts
HIGHLY ORGANIC SOILS		Pt	Peat and other highly organic soils

Figure 2 USCS Soil Classification Chart

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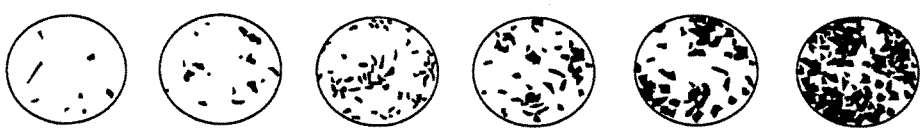
ROCK DESCRIPTIVE TERMS

Term		Defining Characteristics	
Hardness	Soft	Scratched by fingernail	
	Moderately Hard	Scratched easily by penknife	
	Hard	Difficult to scratch with a penknife	
	Very Hard	Cannot be scratched by penknife	
Weathering	Unweathered	Rock is unstained. May be fractured, but discontinuities are not stained.	
	Slighty	Rock is unstained. Discontinuities show some staining on the surfaces of rocks, but discoloration does not penetrate rock mass.	
	Moderate	Discontinuity surfaces are stained. Discoloration may extend into rock along discontinuity surfaces.	
	High	Individual rock fragments are thoroughly stained and may be crumbly.	
	Severe	Rock appears to consist of gravel-sized fragments in a "soil" matrix. Individual fragments are thoroughly discolored and can be broken with fingers.	
Bedding Planes	Laminated	< .04 in.	< 1 mm
	Parting	.04 in. - .24 in.	1mm - 6mm
	Banded	.24 in. - 1in.	6 mm - 3 cm
	Thin	1 in. - 4 in.	3 cm - 9.1 cm
	Medium	4 in. - 12 in.	9.1 cm - 30.5 cm
	Thick	12 in.- 36 in.	30.5 cm - 1m
	Massive	> 36 in.	> 1 m
Joints and Fracture Spacing	Very tight	< 2 in.	< 5.1 cm
	Tight	2 in. - 1ft.	5.1 - 30.5 cm
	Moderately tight	1ft. - 3 ft.	30.5 cm - 91.4 cm
	Wide	3 ft. - 10 ft.	91.4 cm - 3 M
	Very wide	> 10 ft.	> 3 M
Voids	Porous	Smaller than a pinhead. Their presence is indicated by the degree of absorbency.	
	Pitted	Pinhead size to a 1/4 inch. If only thin walls separate the individual pits, the core may be described as honeycombed.	
	Vug	1/4 inch to the diameter of the core. The upper limit will vary with core size.	
	Cavity	Larger than the diameter of the core.	

Rock Particle Percent Composition Estimation



1% 5% 10% 15% 25% 50%

Figure 3 Rock Descriptive Terms

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TITLE:	GEOLOGIC LOGGING		
CATEGORY:	GEO 4.8	REVISED:	March 1998

ROCK QUALITY DESIGNATION (RQD) AND FRACTURE FREQUENCY

Core borings are a useful means of obtaining information about the quality of rock mass. The recoverable core indicates the character of the intact rock and the number and character of the natural discontinuities.

Another quantitative index that has proved useful in logging NX core is a rock quality designation (RQD) developed by Deere (1963). The RQD is a modified core recovery percentage in which all the pieces of sound NX core over 4 inches long are counted as recovery. The length of the core run is the distance to the nearest tenth of a foot from the corrected depth of the hole at the end of the previous run to the corrected depth of the hole at the end of subject run. The smaller pieces are considered to be due to close shearing, jointing, faulting, or weathering in the rock mass and are not counted. The RQD is a more general measure of the core quality than the fracture frequency. Core loss, weathered and soft zones, as well as fractures, are accounted for in this determination. The RQD provides a preliminary estimate of the variation of the in situ rock mass properties from the properties of the "sound" portion of the rock core. Thus, a general estimate of the behavior of the rock mass can be made. An RQD approaching 100 percent denotes an excellent quality rock mass with properties similar to that of an intact specimen. RQD values ranging from 0 to 50 percent are indicative of a poor quality rock mass having a small fraction of the strength and stiffness measured for an intact specimen.

RQD (Rock Quality Designation)

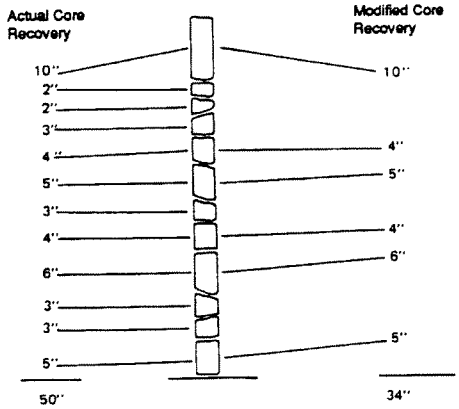
0 - 25	Very Poor
25 - 50	Poor
50 - 75	Fair
75 - 90	Good
90 - 100	Excellent

An example of determining the RQD from a core run of 60 inches measured from corrected depth to corrected depth is given in Diagram 1 . For this particular case, the core recovery was 50 inches and the modified core recovery was 34 inches. This yields an RQD of 57 percent, classifying the rock mass in the fair category.

Problems arise in the use of RQD for determining the in situ rock mass quality. The RQD evaluates fractures in the core caused by the drilling process, as well as in natural fractures previously existing in the rock mass. For example, when the core hole penetrates a fault zone or a joint, additional breaks may form that, although not natural fractures, are caused by natural planes of weakness existing in the rock mass. These fresh breaks occur during drilling and handling of the core and are not related to the quality of the rock mass. The skill of the driller will affect the amount of breakage and the core loss that occurs. Poor drilling techniques will "penalize" the rock by lowering its apparent quality. It is difficult to distinguish between drilling breaks and those natural and incipient fractures that reflect the quality of the rock mass. In certain instances, it may be advisable to include all fractures when estimating RQD. Obviously, some judgement is involved in core logging.

Another problem with the use of the RQD index is that the determinations are not sensative to the tightness of the individual joints, whereas in some instances, the in situ deformation modulus may be strongly affected by the average joint opening.

RQD OF A SINGLE CORE RUN *



Core Run 60" (Corrected depth to corrected depth)

$\frac{34"}{60"}$ x 100% = 57% = Fair

* Typical calculation of RQD of a single core run. Note that the run is calculated from corrected depth to corrected depth.

Figure 4 Rock Qualitative Designation (RQD)

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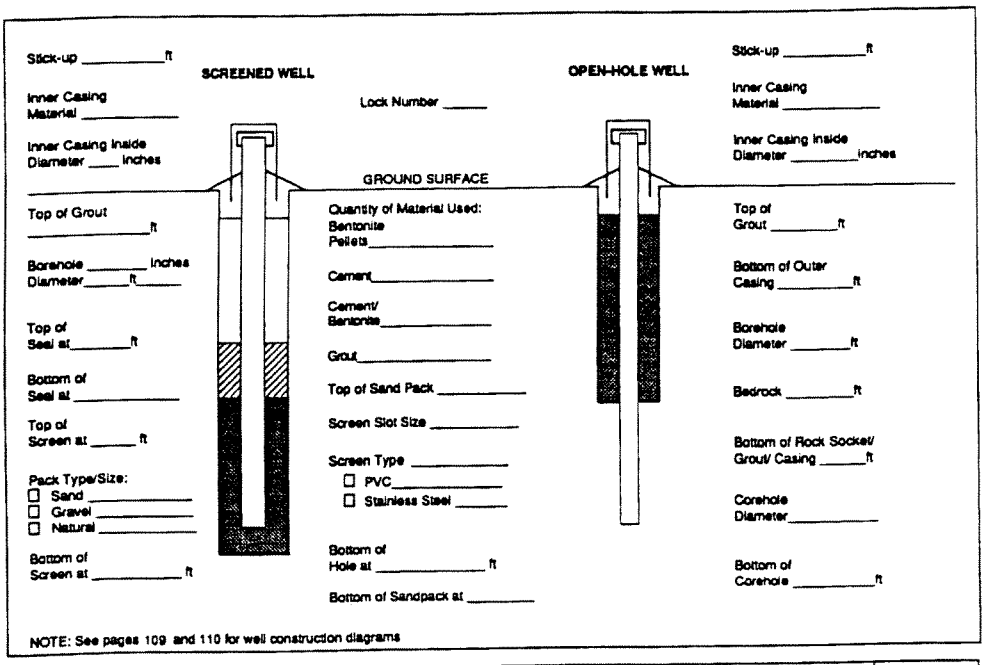

Depth-ft.	NARRATIVE LITHOLOGIC DESCRIPTION	Moisture Content: Dry	Moist	Wet
1		○	○	○
2		○	○	○
3		○	○	○
4		○	○	○
5		○	○	○
6		○	○	○
7		○	○	○
8		○	○	○
9		○	○	○
10		○	○	○
11		○	○	○
12		○	○	○
13		○	○	○
14		○	○	○

Figure 5 Narrative Lithologic Description

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ASTM CRITERIA FOR DESCRIBING SOIL

Criteria for Describing Angularity of Coarse-Grained Particles

Description	Criteria
Angular	Particles have sharp edges and relatively plane side with unpolished surfaces
Subangular	Particles are similar to angular description but have rounded edges
Subrounded	Particles have nearly plane sides but have well-rounded corners and edges
Rounded	Particles have smoothly curved side and no edges

Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in the specimen.
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.

Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.
Medium	Medium pressure is required to roll the thread to near plastic limit. The thread and the lump have medium stiffness.
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness.

Criteria for Describing Dry Strength

Description	Criteria
None	The dry specimen crumbles into powder with mere pressure of handling
Low	The dry specimen crumbles into powder with some finger pressure
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure
High	The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface.
Very High	The dry specimen cannot be broken between the thumb and shard surface

Criteria for Describing Structure

Description	Criteria
Stratified	Alternating layers of varing material or color with layers at least 6 mm thick; note thickness.
Laminated	Alternating layers of varying materials or color with the layers less than 6 mm thick; note thickness.
Fissured	Breaks along definite planes of fracture with little resistance to fracturing.
Slickensided	Fracture planes appear polished or glossy, sometimes striated.
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown.
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness.
Homogeneous	Same color and appearance throughout.

Figure 6 ASTM Criteria for Describing Soil

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CRITERIA FOR DESCRIBING SOIL (Cont.)

Criteria for Describing the Reaction with HCl

Description	Criteria
None	No visible reaction
Weak	Some reaction, with bubbles forming slowly
Strong	Violent reaction, with bubbles forming immediately

Criteria for Describing Consistency

Description	Criteria
Very Soft	Thumb will penetrate soil more than 1 inch (25 mm)
Soft	Thumb will penetrate soil about 1 inch (25 mm)
Firm	Thumb will indent soil about 1/4 inch (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very Hard	Thumbnail will not indent soil

Criteria for Describing Cementation

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Moderate	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure

Criteria for Describing Particle Shape

The particle shape shall be described as follows where length, width, and thickness refer to greatest, intermediate, and least dimensions of a particle, respectively (see page 104).

Flat	Particles with width/thickness ratio > 3
Elongated	Particles with length/width ratio > 3
Flat and Elongated	Particles meet criteria for both flat and elongated

Criteria for Describing Plasticity

Description	Criteria
Nonplastic	A 1/8 inch (3 mm) thread cannot be rolled at any water content.
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit.
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit.
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit.

Identification of Inorganic Fine-Grained Soils from Manual Tests

Soil Symbol	Dry Strength	Dilatancy	Toughness
ML	None to low	Slow to rapid	Low or thread cannot be formed
CL	Medium to high	None to slow	Medium
MH	Low to medium	None to slow	Low to medium
CH	High to very high	None	High

Criteria for Describing Moisture Condition

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp but no visible water
Wet	Visible free water, usually soil is below water table

Figure 6 ASTM Criteria for Describing Soil (cont.)

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2. Gradation or Plasticity. Granular soils (i.e., sands or gravels) should be described as well-graded, poorly-graded, uniform, or gap-graded, depending on the gradation of the minus 3-inch fraction. Cohesive soils (i.e., silts and clays) should be described as nonplastic, slightly plastic, moderately plastic, or highly plastic, depending on results of the manual evaluation for plasticity.

3. Particle size distribution. An estimate of the percentage and grain-size range of each subordinate constituent of the soil. This description may also include a description of angularity (see Figure 7).

4. Color. The basic color of the soil.

5. Moisture content. The amount of soil moisture (dry, moist, or wet).

6. Relative density or consistency. An estimate of density of a granular soil or consistency of a cohesive soil, usually based on the standard penetration test results (see Table 1).

7. Soil Structure or Mineralogy. Description of discontinuities, inclusions, and structures. Includes joints, fissures, and slickensides.

4. Core Logging

4.1 Handling of Core

After the core has been recovered from the corehole and the core barrel has been opened, the core should be placed in a core box. The top of the core should be placed at the back left corner of the core box, and the remaining core placed to the right of the preceding section (see Figure 8). The core box should be filled in this manner, moving to the front sections of the core box. The beginning of each run should be marked on the core and also noted with a marked wooden block.

4.2 Rock Description

Each stratigraphic unit in the core shall be logged. A line marking the depth of the top and the bottom of the unit shall be drawn horizontally. In classifying the rock, the geologist should avoid being too technical, as the information presented must be used by numerous people with widely divergent backgrounds.

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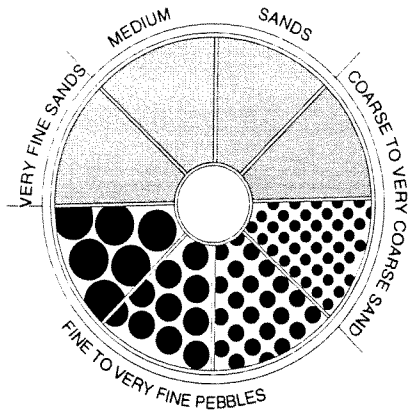
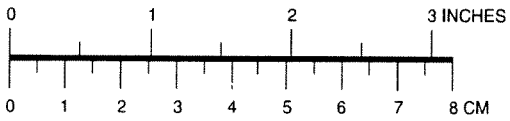
SEDIMENT PARTICLE SIZE AND SHAPE ESTIMATES

GRAPH FOR DETERMINING SIZE OF SEDIMENTARY PARTICLES





COBBLES RANGE FROM 6.4 TO 25.6 cm (~2.5 TO 10.1 INCHES)
BOULDERS ARE LARGER THAN 25.6 cm (>10.1 INCHES)

SEDIMENT PARTICLE SHAPES

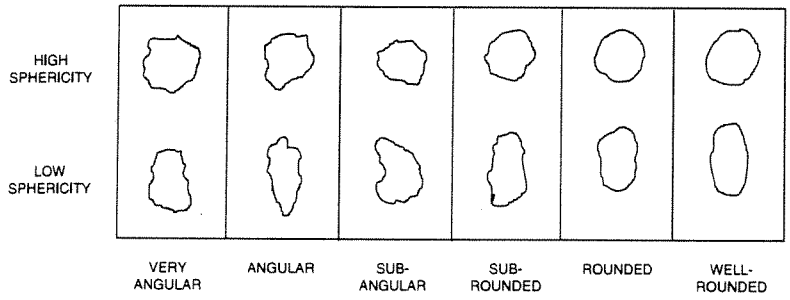


Figure 7 Sediment Particle Size and Shape Estimates

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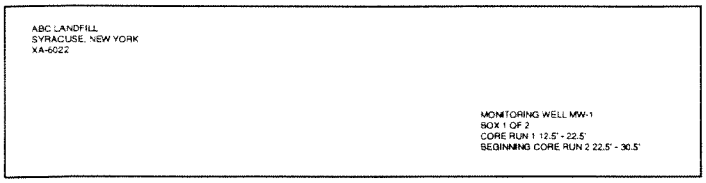

EXAMPLE: OUTSIDE CORE BOX COVER

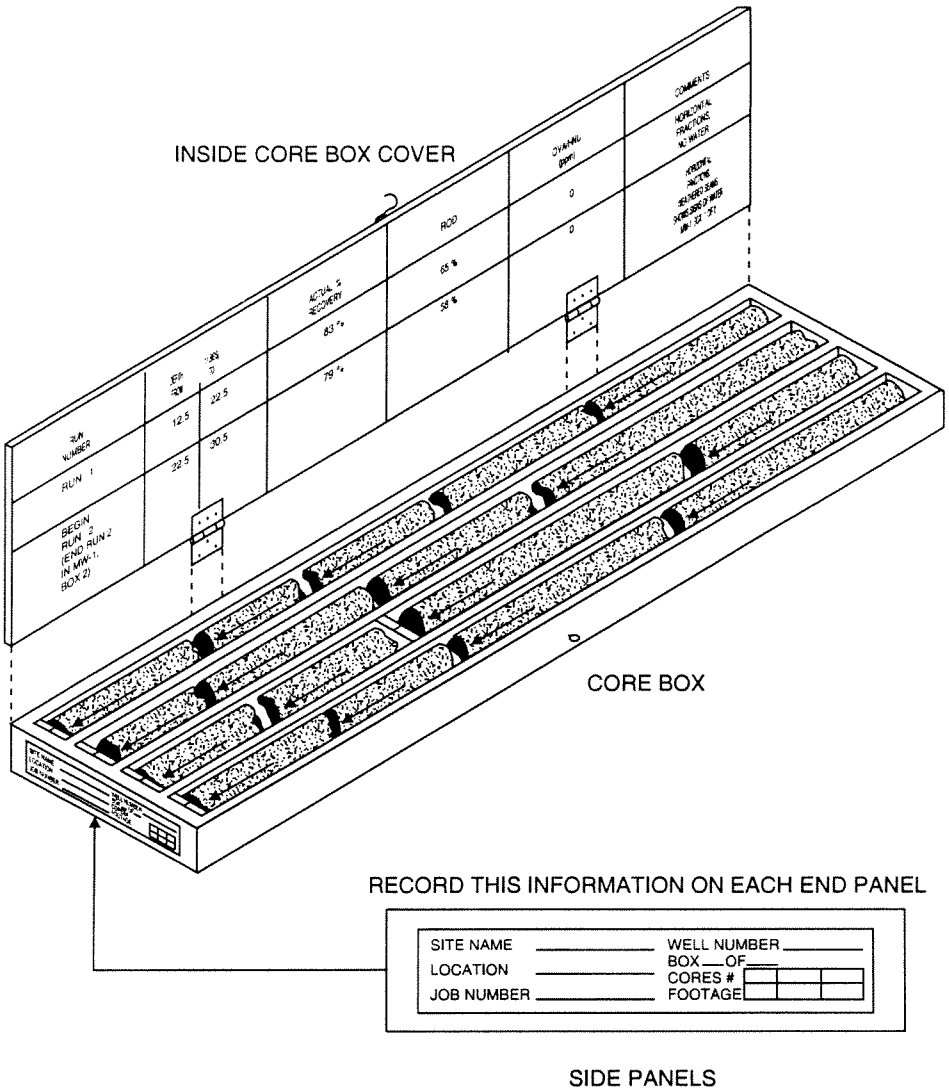

Figure 8 Core Box

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The classification and description of each unit should be given in the following order, as applicable:

1. Unit designation (Miami oolite, Clayton Formation, Chattanooga shale);
2. Rock type;
3. Hardness;
4. Degree of weathering;
5. Texture;
6. Structure;
7. Color;
8. Solution and void conditions;
9. Swelling properties;
10. Slaking properties; and
11. Additional description, such as mineralization, size, and spacing shale seams, etc.

Variations from the general description of the unit and features not included in the general description shall be indicated by brackets and lines to show the depth and the interval in the core where the feature exists. These variations and features shall be identified by terms that will adequately describe the feature or variation so as to delineate it from the unit. These may be zones or seams of different color, texture, etc., from that of the unit as a whole, such as staining; variations in texture; shale seams, gypsum seams, chert nodules, calcite masses, etc.; mineralized zones; vuggy zones, joints, fractures; open and/or stained bedding planes; faults, shear zones, gouge; cavities' thickness, open or filled, nature of filling, etc.; or any core left in the bottom of the hole after the final pull.

Rock Type and Lithology

1. Rock will be classified according to the following 24 types:
   - Sandstone

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- Conglomerate
- Coal
- Compaction Shale
- Cemented Shale
- Indurated Clay
- Limestone
- Chalk
- Gneiss
- Schist
- Graywacke
- Quartzite
- Dolomite
- Marble
- Soapstone and Serpentine
- Slate
- Granite
- Diorite
- Gabbro
- Rhyolite
- Andesite
- Basalt

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- Tuff or Tuff Breccia
- Agglomerate or Flow Breccia

2. Lithologic characteristics should be included to differentiate rocks of the same classification. These adjectives should be simple and easily understood, such as shaly, sandy, dolomitic, etc. Inclusions, nodules, and concretions should also be noted here.

3. It is important to maintain a simple but accurate rock classification. The rock type and lithologic characteristics are essentially used to differentiate the rock units encountered.

Hardness

The terms for hardness, as outlined below, were modified to include the use of a rock hammer.

1. **Very soft** or plastic - can be deformed by hand (has a rock-like character but can be broken easily by hand).

2. **Soft** - can be scratched with a fingernail (cannot be crumbled between fingers but can be easily pitted with light blows of a geology hammer).

3. **Moderately hard** - can be scratched easily with a knife; cannot be scratched with a fingernail (can be pitted with moderate blows of a geology hammer).

4. **Hard** - difficult to scratch with a knife (cannot be pitted with a geology hammer but can be chipped with moderate blows of the hammer).

5. **Very hard** - cannot be scratched with a knife (chips can be broken off only with heavy blows of the geology hammer).

Weathering

The degree and depth of weathering is very important and should be accurately detailed in the general description and clearly indicated on the drill log.

1. **Unweathered** - no evidence of any mechanical or chemical alteration.

2. **Slightly weathered** - superficial discoloration, alteration, and/or discoloration along discontinuities; less than 10% of the rock volume is altered; strength is essentially unaffected.

3. **Moderately weathered** - discoloration is evident; surface is pitted and altered, with alterations penetrating well below rock surfaces; 10% to 50% of the rock is altered; strength is noticeably less than unweathered rock.

4. **Highly weathered** - entire section is discolored; alteration is greater than 50%; some areas of slightly weathered rock are present; some minerals are leached away; retains only a fraction of its original strength (wet strength is usually lower than dry strength).

5. **Decomposed** - saprolite; rock is essentially reduced to a soil with a relic rock texture; can be molded or crumbled by hand.

Texture

Texture is used to denote the size of the grains or crystals comprising the rock, as opposed to the arrangement of the grains or crystals, which is considered a structure.

1. **Aphanitic** - grain diameter less than 0.004 inch (0.1 mm); individual grains or crystals are too small to be seen with the naked eye.

2. **Fine-grained, finely crystalline** - grain diameter between 0.004 inch (0.1 mm) and 0.003 (1 mm); grains or crystals can be seen with the naked eye.

3. **Medium-grained, crystalline** - grain diameters between 0.003 foot (1 mm) and 0.0175 foot (5 mm).

4. **Coarse-grained, coarsely crystalline** - grain diameter greater than 0.0175 foot (5 mm).

Structure

The structural character of the rock shall be described in terms of grain or crystal alignment, bedding, and discontinuities, as applicable. The terms may be used singularly or paired.

1. **Foliation and/or lineation** - give approximate dip uniformity, degree of distinctiveness, banding, etc.

2. **Joints:**
   a. Type - bedding, cleavage, foliation, extension, etc.
   b. Degree of openness - tight or open.
   c. Surface or joint plane characteristics - smooth, rough, undulating.
   d. Weathering - degree, staining.
   e. Frequency - see (4).

3. **Fractures, shears, gouge:**
   a. Nature - single plane or zone. (Note thickness.)
   b. Character of materials in plane or zone.
   c. Slickensides.

4. **Frequency:**
   a. Intact - spacing greater than 6 feet (2 m).
   b. Slightly jointed (fractured) - spacing 3 feet (1 m) to 6 feet (2 m).
   c. Moderately jointed (fractured) - spacing 1 foot (0.3 m) to 3 feet (1 m).
   d. Highly jointed (fractured) - spacing 0.3 foot (9.1 cm) to 1 foot (0.3 m).
   e. Intensely jointed (fractured) - spacing less than 0.3 foot (9.1 cm).

5. **Bedding** is used to describe the average thickness of the individual beds within recognized unit, and the terms thick, medium, or thin should not be applied to the individual beds. "Parting" and "band" are used to describe single stratum as outlined below:
   a. Massive - over 3 feet thick (1 m).
   b. Thick - 1 foot (30.5 cm) to 3 feet (1 m) thick.
   c. Medium - 0.3 foot (9.1 cm) to 1 foot (30.5 cm) thick.
   d. Thin - 0.1 foot (3.0 cm) to 0.3 foot (9.1 cm) thick.
   e. Band - 0.02 foot (6 mm) to 0.1 foot (3.0 cm) thick, described to the nearest 0.01 foot.
   f. Parting - less than 0.02 foot (6 mm).
   g. Paper-thin parting.

The terms and descriptions for the structure of the rock are to be used to describe the character of the rock units recognized and are not to be used as a substitute for describing individual discontinuities. Except for areas where the rock is intensely fractured or jointed, each discontinuity should be described on the log as to position, dip, staining, weathering, breccia, gouge, etc.

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Color is often valuable in correlating or differentiating samples, but can be misleading or uninformative. The color of a sample should represent the sample in terms of basic hues (i.e., red, blue, gray, black), supplemented with modifying hues as required (i.e., bluish gray, mottled brown). The core should be surface wet when describing the color; if it is dry, the log should indicate "dry color." Subjective colors, such as buff or maroon, should not be used. Specific color charts, such as the Mumsel Color Chart or the Color Index in the Colorado School of Mines, Quarterly, Volume 50, No. 1, are useful in describing color of samples. When such a chart or index is used, it should be noted on the log in the remarks column.

Solution and Void Conditions shall be described in detail, as these features can affect the strength of the rock and can indicate potential seepage paths through the rock. When cavities are detected by drill action, the depth to top and bottom of the cavity should be determined by measuring the stick-up of the drill tools when the cavity is first encountered and again at the bottom, as it is very difficult to reconstruct cavities from the core alone. Filling material, when present and recovered, should be described in detail opposite the cavity. When no material is recovered from the area of the cavity, the inspector should note the probable conditions of the cavity as determined from observing the drilling action and the color of the drill fluid. If the drill action indicated material was present (i.e., slow rod drop, no loss of drill water, noticeable change in color of water return), it should be noted on the log that the cavity was probably filled and the materials should be described as best as possible from the cuttings or traces left on the core. If drill action indicates the cavity was open (i.e., no resistance to the drill tools, loss of drill fluid), this should be noted on the drill log. Partially filled cavities should also be noted. All of these observations require close observation of the drill action and water return by both the inspector and the driller; accurate measurement of stick-ups; and detailed inspection of the core. When possible, filling material should be wrapped in foil if left in the core box. If the material is to be tested or examined in the lab, it should be sealed in a jar with proper labels and a spacer, with a note showing the disposition of the material should be placed in the core box at the point from which the material was taken. Terms to describe voids encountered shall be as follows:

1. **Porous** - voids less than 0.003 foot (1 mm) in diameter.

2. **Pitted** - voids 0.03 foot (1 mm) to 0.02 foot (6 mm) in diameter.

3. **Vug** - voids 0.02 foot (6 mm) to the diameter of the core.

4. **Cavity** - voids greater than diameter of the core.

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4.3 Core Labeling

The top of the core should be shown on each piece of core with an arrow written in a black, waterproof marker. The arrow will indicate which end of the core is nearer the ground surface. Other core markings may include locations of mechanical breaks and drilling footages.

4.4 Core Box Labeling

Each core box should be labeled as follows:

- On the top left corner of the outer core box, the project name, site location (city and state), and project number should be written.
- On the lower right corner of the outer core box, the corehole number (e.g., MW1, BH2), core box number (e.g., 1 of 2, 2 of 2), and the interval of the core run contained in the core box should be written.
- The side panels should be marked as indicated in Figure 8.
- The inside of the core box cover should be marked as indicated in Figure 8.

4.5 Core Storage

It is important to use proper-sized (HQ or NQ) wooden core boxes for rock core storage. After labeling the box and before closing the box for final storage or shipment, wooden spacers should be inserted into each compartment that contains rock core. This will prevent lateral movement of the cores, which could damage the rock material during handling.

After properly logging, labelling, and packing the cores, the core boxes should be stored in a dry location, preferably off of the floor on a pallet. The boxes can be stacked to a reasonable height so as not to be unstable, with end labelling facing out.

5. References

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Field Sampling Plan
Attachment C
Standard Operating Procedure for Measuring the Water Level and Well Depth

TITLE:	MEASURING WATER LEVEL AND WELL DEPTH
CATEGORY:	GEO 4.15
REVISED:	March 1998

STANDARD OPERATING PROCEDURE

MEASURING WATER LEVEL AND WELL DEPTH

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TITLE:	MEASURING WATER LEVEL AND WELL DEPTH		
CATEGORY:	GEO 4.15	REVISED:	March 1998

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TITLE:	MEASURING WATER LEVEL AND WELL DEPTH		
CATEGORY:	GEO 4.15	REVISED:	March 1998

1. Scope and Application

This document describes E & E's standard operating procedure (SOP) for measuring water level and well depth in monitoring wells and piezometers.

2. Equipment

The following is a list of equipment and items typically used for measuring water level and well depth:

- Electronic water level indicator with graduated cable measured at increments of 0.1 and 0.01 foot;
- Plastic sheeting; and
- Folding ruler or pocket steel tape.

3. Procedure

3.1 Preliminary Steps

1. Locate the well or piezometer and verify its position on the site map. Record whether positive identification was obtained, including the well number and any identifying marks or codes contained on the well casing or protective casing. Gain access to the top of the well casing and note the date and time the well was opened. If specified in the work plan or site health and safety plan, use monitoring equipment to measure or take readings of the well headspace. Record all measurements and observations (e.g., odor).

2. Locate and record the specified benchmark or survey point for the well or piezometer, which may be a mark at the top of the casing or a surveyor's pin embedded in the protective structure. Determine the elevation of this point from the records and record in the notebook. Measure and record the vertical distance from the benchmark to the top of the well casing to the nearest 0.01 foot. Measure and record the metal casing stickup (i.e., the distance between the top of the casing and nominal ground level).

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3. Record any observations and remarks regarding the completion characteristics and well condition, including evidence of cracked casing or surface seals, security of the well (locked cap), and evidence of tampering.

4. Keep all equipment and supplies protected from contamination with clean plastic sheeting. Keep the water level indicator probe in its protective case when not in use.

3.2 Operation

1. Remove the water level indicator probe from the case, turn on the sounder, and test-check the battery and sensitivity scale by pushing the red button. Adjust the sensitivity scale until you can hear the buzzer and see the red indicator light.

2. Slowly lower the probe and cable into the well, allowing the cable reel to unwind. Continue lowering until the meter buzzes. Raise and lower the probe very slowly until the meter begins to buzz continuously. Mark the spot by grasping the cable with the thumb and forefingers at the top of the casing, withdraw the cable, and record the depth.

3. To measure the total well or piezometer depth, lower the probe until slack is felt in the cable. Very slowly raise and lower the cable until the exact bottom of the well is detected. As before, grasp the cable with the thumb and forefinger at the top of the casing and note the depth. If a water level probe, such as the Solinst, is used to measure total depth, the weight of the probe will likely extend approximately 6 centimeters beyond the calibrated "zero" point of the measuring cable. If this is the case, use the cable to accurately measure the distance from the end of the weight to the point of the needle (in the "window" of the probe) and add this length to the depth noted above. Record the sum of these two lengths as the total depth of the well.

4. Withdraw the cable and probe, and decontaminate according to the SOP for Equipment Decontamination (ENV 3.15).

3.3 Data Recording and Manipulation

Record the following computations:

- Casing elevation = bench mark elevation + casing stickup
- Water level elevation = casing elevation - depth of water
- Well bottom elevation = casing elevation - depth to bottom
- Total well depth = cable-measured depth + length of the weight extension

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4. Calibration

No calibration is needed for the electronic water level indicator.

5. Precautions

Because some casings have rough or sharp edges, use caution when lowering and retrieving the water level cable from within the well casing. These edges can cut and scrape the cable, obscuring the calibrated markings on the cable, and can eventually lead to failure (shorting out) of the electronic cable.

Always use caution when opening capped wells, because escaping (venting) headspace gases may be hazardous.

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Field Sampling Plan
Attachment D
Standard Operating Procedure for
Geotechnical Logbook/Trip Report Preparation

TITLE:	GEOTECHNICAL LOGBOOK/ TRIP REPORT PREPARATION
CATEGORY:	DOC 2.4
REVISED:	April 1998

STANDARD OPERATING PROCEDURE

GEOTECHNICAL LOGBOOK/ TRIP REPORT PREPARATION

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ecology and environment, inc.
368 Pleasant View Drive / Lancaster, New York 14086 / (716) 684-8060

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TITLE:	GEOTECHNICAL LOGBOOK/ TRIP REPORT PREPARATION
CATEGORY:	DOC 2.4
REVISED:	April 1998

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TITLE:	GEOTECHNICAL LOGBOOK/ TRIP REPORT PREPARATION		
CATEGORY:	DOC 2.4	REVISED:	April 1998

1. Introduction

This Standard Operating Procedure (SOP) outlines the procedures followed by Ecology and Environment, Inc. (E & E), for the entry of information into the geotechnical logbook, thereby ensuring that field activities are properly documented.

It is the responsibility of the site geologist to ensure that the proper information is collected in the field in order to fulfill the obligations of the contract.

2. Purpose

The purpose of this document is to establish the minimum content requirements of the geotechnical logbook entries for drilling projects. The document provides guidance to ensure that the documentation for drilling projects is correct, complete, and adequate for use in any potential legal proceeding. It is important to remember that field activity documentation can become evidence in civil and criminal law- enforcement proceedings, as well as in administrative hearings. Accordingly, such documentation is subject to judicial or administrative review; even more importantly, it is subject to the review of an opposing attorney attempting to discredit its value as evidence. Complete and accurate entries in the geotechnical logbook are important for two reasons: to maintain quality control, and to support any legal proceedings associated with the project.

3. Geotechnical Logbook Procedures

3.1 General Information

Information concerning the project can be found in the geotechnical logbook. The majority of the information should be obtained prior to arriving on site. Figure 1 is an example of a completed general information sheet.

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TITLE:	GEOTECHNICAL LOGBOOK/ TRIP REPORT PREPARATION		
CATEGORY:	DOC 2.4	REVISED:	April 1998

CLIENT: Chemical Corporation of America
JOB NUMBER: ZZ-1020

SITE NAME: ABC Landfill
DRILLER: Paul Barth E+E Drilling and Testing Company
LOCATION: CITY/TOWN: Midway
STATE: Tennessee

PROJECT MANAGER: Buff Walter

FIELD TEAM LEADERS: Marilyn Frappa /
SITE SAFETY OFFICER(S): Greg Jones /
TEAM MEMBERS: Paul Barth (EEDT)
Kevin Williamson (EEDT)

JOB START/FINISH DATE: 1/4/94 / 1/18/94

BOOK i OF 3

E&E CORPORATE: (716) 684-8060 FAX (716) 684-0844
E&E EMERGENCY RESPONSE CENTER: (716) 684-8940
E&E ANALYTIC SERVICE CENTER: (716) 631-0360 FAX (716) 631-0378
E&E EQUIPMENT SERVICE CENTER: (716) 681-9797 FAX (716) 681-4356
FEDERAL EXPRESS TOLL FREE: (800) 238-5355

PROJECT/CLIENT CONTACT(S)	AFFILIATION	PHONE
Buff Walter	E+E	703/522-6065

For inquires regarding the distribution, scope, and/or organization of the Geotechnical Logbook please contact:

Figure 1 General Information

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3.2 Daily Logs

3.2.1 General Guidelines

The following includes general guidelines for preparing the geotechnical logbook:

- A separate logbook must be maintained for each project.
- No pages can be removed for any reason, even if they are partially mutilated or illegible.
- All field activities (e.g., meetings, sampling, surveying) must be recorded in the geotechnical logbook.
- All information is **printed** legibly into the logbook in waterproof ink, preferably black. If weather conditions do not permit this (i.e., if it is too cold or too wet to write with ink), another medium such as a pencil may be used, but it should be specifically noted in the logbook why waterproof ink was not used.
- The language used in the geotechnical logbook should be objective, factual, and free of personal feelings or terminology that might prove inappropriate.
- Chronological entries are preferred. If entries cannot be made at the time observed, record them as soon as possible, noting both the time of the entry and the time of the observation.
- Each successive day's first entry is made on a new, blank page.
- Each page should be dated, and all entries should have a time notation based on the 24-hour clock (e.g., 0900 for 9:00 a.m., 2100 for 9:00 p.m.).
- At the completion of the drilling activity, the geotechnical logbook must be placed in the permanent project file.

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3.2.2 Format

The information listed below is not meant to be all-inclusive. Each project manager is responsible for determining the information requirements for each geotechnical logbook; such information requirements will vary depending on the nature and scope of the project.

First Page: On the first page of every daily entry, the date and project number should be entered on the top line. The proposed work for the day, weather on site, and personnel on site follow below the date. Log entries include the time that personnel arrive on site, the time personnel depart the site, the time that site safety meetings are held (with signatures of the personnel attending the briefing), the levels of personal protection used by the team, specific activities undertaken (e.g., drilling operations, air monitoring, sample collection), and equipment calibration data. At the bottom of the page, the signature of the person keeping the log and the date should be entered.

Successive Pages: The date and project number should appear at the top of each successive page, and the person keeping the log should sign and date the page.

Last Page: On the last page of the daily log, the work completed that day and future plans and recommendations should be entered.

Samples: Each sample must be properly accounted for in the geotechnical logbook. Information entered in the log should include the location where the sample was collected; the time that the sample was collected; the type of sample (e.g., subsurface composite soil sample or groundwater sample); on-site measurement data (e.g., pH, temperature, conductivity); a preliminary description of the sample; preservatives used (if any); air monitoring instrument readings; and the Federal Express (or other carrier's) air bill number.

3.2.3 Photographs

If photographs are taken of soil samples, drill cuttings, or core samples, the following information should be entered in the geotechnical logbook:

- Time, location, direction, and weather conditions.
- Complete description or identification of the subject in the photograph and reason for taking the picture.
- Sequential number of photograph and film number.

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- Camera type and serial number (e.g., Olympus 35-mm #1164916), and lens size and serial number (if appropriate).

- Name of photographer.

Upon return to the office, the above information will be used to prepare photograph logs.

3.2.4 Signature

Each page of the geotechnical logbook must be initialed by the person recording the information. When two individuals make entries on the same page, they must initial their own entries. The individual making the last entry on the page must sign and date the bottom of the page.

3.2.5 Corrections

If corrections are necessary, they may be made by drawing a single line through the entry and writing the corrected entry next to it. The correction must be initialed and dated. Do not render the incorrect notation illegible; make the correction in such a manner that the original entry can still be read.

3.3 Drilling Information

Information concerning the installation of the borehole is entered in the drilling log. The geotechnical logbook is divided in such a manner that the information for four boreholes can be entered in one logbook. In addition to information about the location, start and finish dates, drill rig, and driller, a sketch indicating the location where the borehole or well was installed should be drawn.

Sample information should also be included in this section. The number of blows it took to drive the split-barrel sampler 2 feet below the bottom of the hollow-stem augers in 6-inch intervals should be recorded. The amount of soil recovered in the split-barrel sampler, organic vapor readings noted in the sample, and soil components must also be recorded in this section. An example of a completed drilling log can be found in Figure 2.

3.4 Lithologic Description

A description of the materials used in the construction of the monitoring well and the type of well (e.g., screened or open-hole well) must also be recorded. Figure 3 is an example of a completed well installation diagram. In addition to the well installation diagram, a narrative description of the lithology of the soil and bedrock encountered is also recorded in this section.

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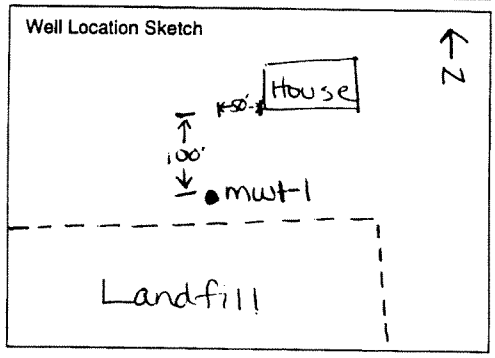
DRILLING LOG FOR MW1-1

Project Name ABC Landfill RI
Site Location Midway, TN
Date Started/Finished 1/6/94 - 1/6/94
Drilling Company E&E Drilling & Testing
Driller's Name Paul Barth
Geologist's Name Marylyn Frappa
Geologist's Signature MarylynFrappa
Rig Type (s) DIEDRICH D50
Drilling Method (s) 4.25" HSA
Bit Size (s) _____ Auger Size (s) 8" O.D.
Auger/Split Spoon Refusal 24 ft
Total Depth of Borehole is 24 ft.
Total Depth of Corehole is N/A

Water Level (TOIC)		
Date	Time	Level (Feet)
1/6/94	1640	16.25
1/8/94	0920	13.75
1/14/94	0830	13.86
1/14/94	1020	14.26

Well Location Sketch



Depth (Feet)	Sample Number	Blows on Sampler		Soil Components CL SL S GR	Rock Profile Recovery	(PPM) Head Space	Run Number	Core Recovery	RQD	Fracture Sketch	OVA (ppm)	Comments
0'-.5' Asphalt												
1	1	10	9	SL/S	16"	Ø ppm	1	N/A		NONE	Ø PPM	
2		16	19									
3	2	7	8	SL/S	6"	Ø PPM	2	N/A		NONE	Ø PPM	
4		10	9									
5	3	6	8	SL/S	10"	Ø ppm	3	N/A		NONE	Ø PPM	
6		5	9									
7	4	4	11	SL/S	6"	Ø PPM	4	N/A		NONE	Ø PPM	
8		23	10									
9	5	7	8	SL/S	4"	Ø PPM	5	N/A		NONE	Ø PPM	
10		10	9									
11	6	6	9	S/GR	5"	28 PPM	6	N/A		NONE	1 PPM	
12		26	32									
13	7	12	24	S/GR	6"	Ø PPM	7	N/A		NONE	Ø PPM	
14		34	48									

Figure 2 Drilling Information

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Stick-up 2 ft

Inner Casing Material PVC

Inner Casing Inside Diameter 2 Inches

Top of Grout 0 ft

Borehole 8 Inches Diameter ft

Top of Seal at 10 ft

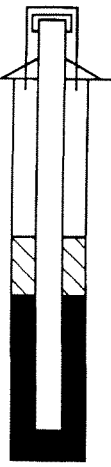
Bottom of Seal at 12 ft

Top of Screen at 14 ft

Pack Type/Size:
- [x] Sand #00N
- [ ] Gravel
- [ ] Natural

Bottom of Screen at 24 ft

SCREENED WELL



Lock Number IG031

Quanity of Material Used:
Betonite Pellets 2 Buckets
Cement 4 Bags
Cement/ Bentonite
Grout
Top of Sand Pack 12'
Screen Slot Size 0.010
Screen Type Continuous
- [x] PVC 2"
- [ ] Stainless Steel

Bottom of Hole at 24 ft

Bottom of Sandpack at 24'

OPEN-HOLE WELL



Stick-up ft

Inner Casing Material

Inner Casing Inside Diameter Inches

Top of Grout ft

Bottom of Outer Casing ft

Borehole Diameter ft

Bedrock ft

Bottom of Rock Socket/ Grout/Casing ft

Corehole Diameter

Bottom of Corehole ft

NOTE: See page 109 and 110 for well construction diagrams

Depth-ft.	NARRATIVE LITHOLOGIC DESCRIPTION	Moisture Content: Dry	Moist	Wet
1	8" Asphalt then Fill - Black + Dry (Cuttings)	●	○	○
2	↓	●	○	○
3	Fill Materials, Mainly Black Cinders, Some whitish Last 3"	●	○	○
4	↓	●	○	○
5	Brown Sandy-Silt Matrix about 50% Small Large Gravels, Subangular	●	●	○
6	↓	●	●	○
7	Same as above	○	●	○
8	↓	○	●	○
9	Same as above	○	●	○
10	↓	○	●	○
11	Same as above with large Gravel 1-2", Moist, 1PPM	○	●	○
12	↓	○	●	○
13	3" Rock + Ground Rock, Grey Sands wet	○	●	○
14	↓	○	○	●
		○	○	●

Figure 3 Well Construction/Lithologic Description

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3.5 Well Development Record

The proper development of monitoring wells will prevent the buildup of fines on the screened interval and will provide groundwater samples that are representative of the groundwater conditions. To determine the volume of water to be removed from the well, the inside diameter of the monitoring well or diameter of the borehole is determined and the appropriate line is found on the table in Figure 4.

3.6 Well Development—Parameter Measures

Physical measurements are made on samples of the groundwater during well development. At routine intervals, a sample of the development water is tested for temperature, conductivity, corrosivity (pH), and turbidity. The results of each test are recorded as shown in Figure 5. When two consecutive tests have the same readings, development of the well is considered complete.

3.7 Investigation-Derived Waste Inventory

In most instances, the drill cuttings, drilling fluids, development and purge water, and personal protective equipment must be containerized and handled as a hazardous waste as described by the Resource Conservation and Recovery Act (RCRA) until analytical results are available to reclassify the wastes. All hazardous wastes must be properly marked and labeled and must be disposed of in a specific period of time. The contents of each container, the source of the waste, the date that the waste was generated, the approximate volume of waste in the container, and the location where the container is being stored must be noted in the geotechnical logbook. Figure 6 is an example of a completed investigation-derived waste inventory sheet.

4. Trip Report

The geologist or team leader is responsible for filing a trip report upon returning to the office. The trip report must include information regarding the persons making the trip; the date of the trip; location of the trip; and, most importantly, the purpose of the trip. The trip report provides the means to convey information gathered and observed to the project manager. Trip reports should be written and submitted within two working days of return from a trip. Figure 7 shows the format for a trip report.

5. References

United States Environmental Protection Agency, 1986, RCRA Groundwater Monitoring Technical Enforcement Guidance Document, OSWER Directive 9950-1, September 1986.

_____, 1986, Region IV Engineering Support Branch, Standard Operating Procedures and Quality Assurance Manual, April 1986.

WELL DEVELOPMENT RECORD

SITE ABC Landfill DATE 1/14/94

LOCATION Midway, TN WELL NO. MW1-1

MEASUREMENT OF WATER LEVEL AND WELL VOLUME

- Prior to sampling, the static water level and total depth of the well will be measured with a calibrated weighted line. Care will be taken to decontaminate equipment between each use to avoid cross contamination of wells.
- The number of linear feet of static water (difference between static water level and total depth of well) will be calculated.
- The static volume will be calculated using the formula:

$$V = Tr^2(0.163)$$

Where:
V = Static volume of well in gallons;
T = Depth of water in the well, measured in feet;
r = Inside radius of well casing in inches;
and 0.163 = A constant conversion factor which compensates for r^2h factor for the conversion of the casing radius from inches to feet, the conversion of cubic feet to gallons, and (pi).
1 well volume (v) = _____ gallons.

Volume of Water in Casing or Hole

Diameter of Casing or Hole (in)	Gallons per Foot of Depth	Cubic Feet per Foot of Depth	Liter per Meter of Depth	Cubic Meters per Meter of Depth
1	0.041	0.0055	0.509	0.509×10^{-3}
1 1/2	0.092	0.0123	1.142	1.142×10^{-3}
2	0.163	0.0218	2.024	2.024×10^{-3}
2 1/2	0.255	0.0341	3.167	3.167×10^{-3}
3	0.367	0.0491	4.558	4.558×10^{-3}
3 1/2	0.500	0.0668	6.209	6.209×10^{-3}
4	0.653	0.0873	8.110	8.110×10^{-3}
4 1/2	0.826	0.1104	10.260	10.260×10^{-3}
5	1.020	0.1364	12.670	12.670×10^{-3}
5 1/2	1.234	0.1650	15.330	15.330×10^{-3}
6	1.469	0.1963	18.240	18.240×10^{-3}
7	2.000	0.2673	24.840	24.840×10^{-3}
8	2.611	0.3491	32.430	32.430×10^{-3}
9	3.305	0.4418	41.040	41.040×10^{-3}
10	4.080	0.5454	50.670	50.670×10^{-3}
11	4.937	0.6600	61.310	61.310×10^{-3}
12	5.875	0.7854	72.960	72.960×10^{-3}
14	8.000	1.0690	99.350	99.350×10^{-3}
16	10.440	1.3960	129.650	129.650×10^{-3}
18	13.220	1.7670	164.180	164.180×10^{-3}
20	16.320	2.1820	202.680	202.680×10^{-3}
22	19.750	2.6400	245.280	245.280×10^{-3}
24	23.500	3.1420	291.850	291.850×10^{-3}
26	27.580	3.6870	342.520	342.520×10^{-3}
28	32.000	4.2760	397.410	397.410×10^{-3}
30	36.720	4.9090	456.020	456.020×10^{-3}
32	41.780	5.5850	518.870	518.870×10^{-3}
34	47.160	6.3050	585.680	585.680×10^{-3}
36	52.880	7.0690	656.720	656.720×10^{-3}

1 Gallon = 3.785 liters
1 Meter = 3.281 feet
1 Gallon water weighs 8.33 lbs. = 37.785 kilograms
1 Liter water weighs 1 kilogram = 2.205 pounds
1 Gallon per foot of depth = 12.419 liters per foot of depth
1 Gallon per meter of depth = 12.419×10^{-3} cubic meters per meter of depth

INITIAL DEVELOPMENT WATER

WATER LEVEL (TOIC) 13.86'
WELL DEPTH (TD) 24
COLOR Brown Silty
ODOR Musty
CLARITY Opaque

FINAL DEVELOPMENT WATER

WATER LEVEL (TOIC) 14.26'
WELL DEPTH (TD) 24'
COLOR Clear
ODOR None
CLARITY Clear

DESCRIPTION OF DEVELOPMENT TECHNIQUE Stainless Steel Hand Bailer measured volume removed with bucket.

Figure 4 Well Development Record

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WELL DEVELOPMENT - PARAMETER MEASUREMENTS

TIME	TOTAL VOL. WITHDRAWN		pH	COND. (umhos/cm)	TEMP. (c)	TURB. (NTU)	COMMENTS
	GALS.	BORE VOL.					
0830	0	26	9.23	4.86	20°C	–	
0838	2	26	8.52	4.69	20.2°	–	
0845	4	26	8.49	4.67	20.3°	–	
0852	6	26	8.37	4.65	20.4°	–	
0900	8	26	8.14	4.63	20.5°	–	
0908	10	26	8.05	4.63	20.5°	–	
0913	12	26	7.96	4.58	20.4°	–	
0926	14	26	7.92	4.50	20.4°	–	
0933	16	26	7.91	4.49	20.6°	–	
0945	18	26	7.90	4.48	20.7°	–	
0953	20	26	7.89	4.42	20.8°	–	
1005	22	26	7.88	4.40	21.0°	–	
1014	24	26	7.88	4.39	21.1°	–	
1020	26	26	7.88	4.39	21.1°	–	

DEVELOPED BY: Kevin Williamson DATE 1/14/94

Figure 5 Well Development-Parameter Measurements

INVESTIGATION-DERIVED WASTE INVENTORY SHEET

Site: ABC Landfill, Midway TN (ZZ-1020) No. of Drums 6

Inventory Date: January 15, 1994

Waste Source	Drum/Container ID Number	Date Generated	Contents (Solid, Liquids, etc.)	Approximate Volume	Drum Location/ Comments
MW1-1	ZZ001	1/6/94	Drill Cuttings	Full	Site 1 Staging Area
MW1-1	ZZ002	1/6/94	Drill Fluids	Full	Site 1 Staging Area
MW1-2	ZZ003	1/7/94	Drill Cuttings	Full	Site 1 Staging Area
MW1-2	ZZ004	1/7/94	Drill Fluids	Full	Site 1 Staging Area
MW1-1	ZZ005	1/14/94	Dev./Purge Water	½ Full	Site 1 Staging Area
MW1-2	ZZ006	1/14/94	Dev./Purge Water	½ Full	Site 1 Staging Area

Figure 6 Investigation-Derived Waste Inventory

TRIP REPORT

Project No. _____

FROM: Persons Making Trip

TO: Project Manager and/or Staff Members

DATE: Date of Trip

LOCATION: Region, Municipality, or Agency with Address Visited

PURPOSE: Reason for Trip

TRIP DISCUSSION: _____

List of Accompanying Contact Reports (if appropriate)
Person Contacted and Date

Figure 7 Trip Report Format

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Field Sampling Plan
Attachment E
Standard Operating Procedure for Groundwater Well Sampling

TITLE:	GROUNDWATER WELL SAMPLING
CATEGORY	ENV 3.7
REVISED:	March 1998

STANDARD OPERATING PROCEDURE

GROUNDWATER WELL SAMPLING

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368 Pleasant View Drive / Lancaster, New York 14086 / (716) 684-8060

5207

TITLE:	GROUNDWATER WELL SAMPLING
CATEGORY:	ENV 3.7
REVISED:	March 1998

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TITLE:	GROUNDWATER WELL SAMPLING		
CATEGORY:	ENV 3.7	REVISED:	March 1998

1. Introduction

The objective of this Standard Operating Procedures (SOP) document is to provide recommended procedures for the sampling of groundwater wells, and is primarily concerned with the collection of water samples from the saturated zone of the subsurface. Every effort must be made to ensure that the sample is representative of the particular zone of water being sampled. Groundwater sampling procedures appropriate to the project objectives and site conditions will define a sampling event.

Analysis of groundwater samples may determine pollutant concentrations and its risk to public health, welfare, or the environment; extent of contaminants; and confirmation of remedial standards.

2. Scope

This document describes procedures for obtaining representative groundwater samples, quality assurance/quality control (QA/QC) measures to be followed, proper documentation of sampling activities, and recommendations for personnel safety.

3. Method Summary

Before sampling a monitoring well, the well must be purged. This may be done with a number of portable devices, including bailers, submersible pumps, bladder pumps, gas-driven pumps, gas-lift pumps, suction-lift pumps, and inertial-lift pumps. Refer to the E & E Standard Operating Procedure for *Groundwater Sampling Devices* (ENV 3.6) for information on different groundwater purging and sampling devices.

A minimum of three well volumes should be removed during well purging to ensure that a representative sample of the groundwater will be sampled. Once the purging is completed and the properly prepared sample containers have been selected, sampling may proceed. Numerous types of sampling devices may be selected for the collection of the groundwater sample, but care should be taken when selecting the sampling device, as some will affect the integrity of the sample.

Sampling should occur in a progression from the least to most contaminated well, if known. Ideally, a dedicated sampling device should be used for each well. However, dedicated sampling devices may not be practical if there are a large number of groundwater samples to be collected. In this case, sampling devices should be cleaned between sampling events using the decontamination procedures outlined in E & E Standard Operating Procedure for *Equipment Decontamination* (ENV 3.15).

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4. Sample Preservation, Containers, Handling, and Storage

The type of analysis for which a sample is being collected determines the type of bottle, preservative, holding time, and filtering requirements (see Table 1). Chemical preservation and cooling of samples to 4 degrees Celcius only retards biological and chemical degradation of contaminants in the sample. Therefore, it is prudent to have the samples delivered to the laboratory as soon as possible following collection.

Sample containers should be precleaned in accordance with U.S. Environmental Protection Agency (EPA) standards and prelabeled, and preservatives should be placed in the containers prior to sample collection. When filling containers, never overfill or prerinse with the water sample, since oil or other substances may remain in the container. For analyses that may require filtered samples (e.g., metals and TOC), the samples should be filtered in the field using one 0.45-micrometer (μm) membrane filter per sample container prior to being preserved.

When all samples have been collected, a field data sheet and a chain-of-custody (C-O-C) form should be completed, and all pertinent data entered in the field logbook. Samples will be placed in a cooler to be maintained on ice at 4 degrees Celcius. Samples must be shipped to arrive at the designated laboratory well before their holding times are reached. It is preferable that these samples be shipped or delivered daily to the laboratory as outlined in the E & E Standard Operating Procedure for *Sample Packaging and Shipping* (ENV 3.16).

5. Potential Problems

5.1 General

The primary goal is to obtain a representative analysis of the groundwater body. The analysis can be compromised by field personnel in two primary ways: by collecting an unrepresentative sample, and by incorrect handling of the sample. There are numerous ways that foreign contaminants can be introduced into the sample, and these must be avoided by following strict sampling procedures and utilization of trained personnel.

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Table 1

SW-846 SAMPLE HOLDING TIMES, PRESERVATION METHODS, AND VOLUME REQUIREMENTS FOR WATER SAMPLES

Protocol Parameter	Holding Time	Minimum Volume	Container Type	Preservation
VOA	14 days from date sampled	One 40-ml vial; no air space	Two 40-ml vials	Add HC1 until pH <2 and ice to 4° C
Semi-VOA (BNAs)	7 days to extract from date sampled	One 1-L jar	1/2-gallon amber glass bottle	Ice to 4°C
PCBs	7 days to extract from date sampled	One 1-L jar	1/2-gallon amber glass bottle	Ice to 4°C
Pesticides and PCBs	7 days to extract from date sampled	One 1-L jar	1/2-gallon amber glass bottle	Ice to 4°C
Metals	6 months from date sampled	One 300-ml bottle	1-L poly bottle	Add $HN0_3$ until pH <2 and ice to 4°C
Cyanide	14 days from date sampled	One 100-ml bottle	1-L poly bottle	Add NaOH until pH >12 and ice to 4°C
Hexavalent chromium	24 hours from time sampled	One 50-ml bottle	125-ml poly bottle	Ice to 4°C
TOC	28 days from date sampled	One 10-ml bottle	125-ml poly bottle	Add H_2SO_4 until pH <2 and ice to 4°C
TOX	7 days from date sampled	One 200-ml bottle	1-L amber glass bottle	Add H_2SO_4 until pH <2 and ice to 4°C
TRPHs	28 days from date sampled	One 1-L bottle	1-L amber glass bottle	Add H_2SO_4 until pH <2 and ice to 4°C

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5.2 Purging

In a nonpumping well, there will be little or no vertical mixing of the water, and stratification will occur. The well water in the screened interval will mix with the groundwater due to normal flow patterns, but the water above the screened interval will remain isolated and become stagnant. Sampling team members should realize that stagnant water may contain foreign material inadvertently or deliberately introduced from the surface. To safeguard against collecting nonrepresentative stagnant water in a sample, the following guidelines and techniques should be adhered to during well purging and sampling:

- As a general rule, all monitoring wells should be pumped or bailed prior to the collection of the sample. Evacuation of a minimum of one volume of water in the well casing, and preferably three to five volumes, is recommended for a representative sample. In a high-yielding groundwater formation and where there is no stagnant water in the well above the screened section, evacuation prior to sample collection is not as critical. However, in all cases where the monitoring data are to be used for enforcement actions, evacuation is recommended.

- For wells that can be pumped or bailed dry, the well should be evacuated and allowed to recover prior to sample withdrawal. If the recovery rate is fairly rapid and time allows, evacuation of more than one volume of water is preferred.

- A nonrepresentative sample can also result from excessive pumping of the monitoring well. Stratification of the leachate concentrations in the groundwater formation may occur or compounds that are heavier than water may sink to the lower portions of the aquifer. Excessive pumping can dilute or increase the contaminant concentrations from what is representative of the sampling point of interest.

Table 2

ADVANTAGES AND DISADVANTAGES OF VARIOUS GROUNDWATER SAMPLING DEVICES

Device	Advantages	Disadvantages
Bailer	• The only practical limitations are size and materials • No power source needed • Portable • Inexpensive; it can be dedicated and hung in a well, reducing the chances of cross-contamination • Minimal outgassing of volatile organics while sample is in bailer • Readily available • Removes stagnant water first • Rapid, simple method for removing small volumes of purge water	• Time consuming, especially for large wells • Transfer of sample may cause aeration
Submersible Pump	• Portable; can be used on an unlimited number of wells • Relatively high pumping rate (dependent on depth and size of pump) • Generally very reliable; does not require priming	• Potential for effects on analysis of trace organics • Heavy and cumbersome, particularly in deeper wells • Expensive • Power source needed • Susceptible to damage from silt or sediment • Impractical in low-yielding or shallow wells
Non-Gas Contact Bladder Pump	• Maintains integrity of sample • Easy to use	• Difficult to clean, although dedicated tubing and bladder may be used • Only useful at depths down to approximately 100 feet • Supply of gas for operation (bottled gas and/or compressor) is difficult to obtain and is cumbersome
Suction Pump	• Portable, inexpensive, and readily available	• Only useful at depths down to approximately 25 feet • Vacuum can cause loss of dissolved gases and volatile organics • Pump must be primed and vacuum is often difficult to maintain • May cause pH modification
Inertia Pump	• Portable, inexpensive, and readily available • Rapid method for purging relatively shallow wells	• Only useful at depths down to approximately 70 feet • May be time consuming to use • Labor-intensive • WaTerra pump is only effective in 2-inch diameter wells

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5.3 Materials

The material used to construct groundwater purging and sampling devices can have a significant impact on the analytical results. If practical, equipment that contacts the groundwater should be constructed from stainless steel, teflon, or glass. The use of plastic should be avoided when analyzing for organics. Table 2 discusses the advantages and disadvantages of groundwater sampling devices, and Table 3 provides a ranking of sample material compatibility under various aqueous environments.

Table 3

RELATIVE COMPATIBILITY OF RIGID GROUNDWATER SAMPLING MATERIALS

	PVC I	Galvanized Steel	Carbon Steel	Low-carbon Steel	Stainless Steel 304	Stainless Steel 316	Teflon
Buffered Weak Acid	100	56	51	59	97	100	100
Weak Acid	98	59	43	47	96	100	100
Mineral Acid/High Solids	100	48	57	60	80	82	100
Aqueous/Organic Mixtures	64	69	73	73	98	100	100
Percent Overall Rating	91	58	56	59	93	96	100

Preliminary Ranking of Rigid Materials:

Teflon
Stainless Steel 316
Stainless Steel 304
PVC I
Low-Carbon Steel
Galvanized Steel
Carbon Steel

6. Equipment Checklist

6.1 General

- Water level indicator (e.g., electric sounder, steel tape, transducer, reflection sounder, air line, etc.);
- Depth sounder;
- Appropriate keys for well cap locks;
- Steel brush;
- Organic vapor analyzer (OVA) or photo-ionization meter (HNu);
- Oil/water interface indicator (if necessary);
- Timepiece (preferably a stopwatch);
- Logbook;
- Calculator;
- Field data sheets;
- Bottle labels;
- Chain-of-custody forms;
- Custody seals;
- Sample containers;
- Engineer's rule;
- Sharp knife (locking blade);
- Tool box (screwdrivers, pliers, hacksaw, hammer, flashlight, adjustable wrench, bolt cutters, etc.);
- Leather work gloves;
- Appropriate personnel protection equipment;

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- 5-gallon pails;
- Plastic sheeting;
- Sealable plastic bags;
- Shipping containers;
- Packing material;
- U.S. Department of Transportation (DOT) shipping labels;
- 55-gallon 1A2 (17-H) drums (if necessary);
- Decontamination solutions;
- Tap water;
- Non-phosphate soap;
- Aluminum foil;
- Garden sprayers;
- Brushes;
- Preservatives; and
- Distilled or deionized water, as necessary.

6.2 Groundwater Sampling Devices

Bailers

- Clean decontaminated bailers of appropriate size and construction material;
- Nylon line (enough to dedicate to each well);
- Sharp knife;
- Aluminum foil (to wrap clean bailers);

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- Submersible pumps;
- Pump(s);
- Adequate power supply, generator, or battery;
- 1-inch black poly vinyl chloride (PVC) coil pipe (enough to dedicate to each well);
- Hose clamps;
- Safety cable (i.e., heavy-grade nylon line);
- Tool box supplement (pipe wrenches, wire strippers, electric tape, heat shrink, hose connectors, teflon tape);
- Winch or pulley (if desired);
- Gasoline for generator;
- Flow meter with gate valve; and
- 1-inch nipples and various pipe connectors.

Bladder Pumps

- Non-gas contact bladder pump;
- Compressor or nitrogen gas bottles;
- Batteries and charger;
- Teflon tubing (enough to dedicate to each well);
- Swagelock fitting; and
- Toolbox supplement (same as submersible pump).

Suction Pump

- Pump;
- Black coil pipe tubing (enough to dedicate to each well);

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- Gasoline (if required);
- Toolbox supplement (same as submersible pump);
- Various hose connectors and nipples; and
- Flow meter with gate valve.

7. Preparation

7.1 Office Preparation

- The preparation of a Site-Specific Safety Plan (SSSP) is required prior to any sampling. The SSSP must be approved and signed by the Corporate Health and Safety Officer or designee (i.e., the Regional Safety Coordinator [RSC]);
- Prepare a Site-Specific Work Plan (SSWP) to meet the data quality objectives of the project in accordance with contract requirements. Review available background information (e.g., topographic maps, hydrogeologic maps, geologic maps, other site reports, etc.) to determine the extent of the sampling effort, the sampling method to be employed, and the type and amounts of equipment and supplies required;
- Obtain necessary sampling and monitoring equipment (see Section 6), preclean the sampling equipment, and ensure that it is in proper working order;
- Ensure that batteries are charged, including the OVA, HNu, pump control box, and large storage batteries;
- Locate local sources for preservatives and decontamination solutions. Review this matter with the RSC or site safety coordinator;
- Contact delivery service to confirm ability to ship all equipment and samples. Determine if shipping restrictions exist; and
- Prepare schedules and coordinate with staff, clients, and regulatory agencies, if appropriate.

7.2 Field Preparation

- Identify local suppliers of expendable sampling equipment such as ice and baggies, and overnight delivery services;
- Inspect all sampling equipment and reclean, if necessary, prior to ground-water sampling (see Table 4);
- A general site survey should be performed prior to site entry in accordance with the SSSP followed by a site safety meeting; and
- Identify all well locations.

Table 4

DECONTAMINATION SOLUTIONS

Type of Hazard	Name of Solution	Remarks
Amphoteric-acids and bases	Sodium bicarbonate	5-15% aqueous solution
Inorganic acids, metal processing wastes, heavy metals	Sodium carbonate	Good water softener, 10-20% aqueous solution
Solvents and organic compounds, oily, greasy unspecified wastes	Trisodium phosphate	Good rinsing solution of detergent, 10% aqueous solution
Pesticides, fungicides, cyanides, ammonia, and other non-acidic inorganic wastes	Calcium hypochlorite	Excellent disinfectant, bleaching and oxidizing agent, 10% aqueous solution
Other Types of Decontamination Solutions		
Other Detergents and Aqueous Surfactants		
Phosphate-free laboratory detergent (Alconox, Liquinox), Pennsalt 91, Oakite, Gunk, Clorox		
Solvents		
1,1,2-Trichloroethane, H2-ethyl-hexyl acetate, pesticide-grade isopropanol/acetone/methanol/hexane, heptane (nonhydrogen bonding), alcohol, diesel fuel, naphtha, beta-propiolactone, carbon tetrachloride, 8% formalinethylene, 8% hexachloromelamine, 1,2-dichloroethane (in solution), Quadcoat		
Other Solutions		
10% nitric acid, 0.1 N/10%/20% hydrochloric acid		
Water		
Potable/tap water (demonstrated to be analyte-free), distilled water, deionized water, reagent-grade distilled and deionized water		

Source: Adapted from Devinny *et al.* 1990; Mickam *et al.* 1989.

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8. Reagents

Except for decontamination solutions and sample preservatives, there are no reagents required for these procedures. Refer to E & E Standard Operating Procedure for *Equipment Decontamination* (ENV 3.15), the SSSP, or the SSWP for proper decontamination procedures and appropriate solvents.

9. Field Sampling Procedures

9.1 Sampling Preparation

- Start at the least-contaminated well, if known;
- Remove locking well cap. Note the location of the well, time of day, and date in the field logbook or sample log;
- Remove the well cap covering the well riser;
- Test the well for volatile organic compounds (VOCs) and methane by conducting a headspace analysis with a combustible gas indicator, an OVA (for VOCs and methane), or an HNu (for VOCs). Record all readings in the field logbook;
- Lower water level measuring device into well until the surface of the water table is encountered;
- Measure the distance from the top of the water table to a reference point on the well riser or casing (e.g., top of inside casing [TOIC]) and record the distance in the field logbook;
- Lower the water level measuring device to the bottom of the well, and measure the total depth of the well using the same reference point on the well riser or casing. Record the distance in the field logbook.
- Measure the diameter of the well, and calculate the volume of water in the well by multiplying the number of feet of water by the number of gallons per foot (see Section 10);
- Determine the required volume of groundwater to be removed from the well (e.g., three well volumes or as indicated in the SSWP);

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- Place plastic sheeting on the ground around the well to minimize the likelihood of contamination of sampling equipment from soil adjacent to the well; and

- Prepare the purging and sampling equipment.

9.2 Purging

The amount of flushing that a well receives prior to sample collection depends on the intent of the monitoring program, as well as the hydrogeologic conditions. Programs in which overall quality determinations of water resources are involved may require long pumping periods to obtain a sample that is representative of the groundwater. The pumped volume can be determined prior to sampling, or the well can be pumped until selected parameters (e.g., temperature, electrical conductance, pH, turbidity, etc.) have stabilized. Care must be taken not to exceed the recommended purging rate for monitoring well screens (see Table 5).

Monitoring for defining a contaminant plume requires a representative sample of a small volume of the aquifer. These circumstances require that the well be pumped enough to remove the stagnant water, but not enough to induce flow from other areas.

During purging, water level measurements may be taken regularly at 15- and 30-second intervals. The data may be used to compute water table or aquifer transmissivity and other hydraulic characteristics.

Information on the most commonly used groundwater purging and sampling devices can be found in E & E's SOP for *Groundwater Sampling Devices* (ENV 3.6).

9.2.1 Bailers

Equipment needed will include a clean decontaminated bailer, nylon line, a sharp knife, and plastic sheeting. Place the plastic sheeting around the well to prevent contact of the bailer or line with the ground. Attach the line to the bailer, and then lower the bailer until it is completely submerged. Pull the bailer out of the well; ensure that the line either falls onto the plastic sheeting or never touches the ground. Empty the bailer into a 5-gallon pail. Repeat the procedure until the required purge volume has been removed. When the 5-gallon pail is full, pour the water into a 55-gallon drum or handle as indicated in the SSWP.

9.2.2 Submersible Pumps

- Assemble the pump, hose, and safety cable;

- Lower the pump and assembly into the monitoring well to a point a few feet below the water level;

- Attach to a power source and commence purging operations;

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Table 5

MAXIMUM RECOMMENDED PURGING RATE FOR MONITORING WELL SCREENS

Screen Type	Diameter (in)	Slot (in)	Open Area (ft^2/ft)	Open Area (%)	Recommended Pumping Rate		
					gpm/ft at 0.1 ft/s	gpm/ft at 0.07 ft/s	gpm/ft at 0.03 ft/s
PVC (machine slot)	2	0.01	0.018	3.4	0.804	0.563	0.241
	2	0.02	0.033	6.4	1.496	1.047	0.449
	2	0.025	0.042	8.0	1.870	1.309	0.561
	2	0.04	0.060	11.5	2.693	1.885	0.808
	2	0.051	0.075	14.4	3.385	2.369	1.015
	4	0.01	0.036	3.4	1.608	1.126	0.482
	4	0.02	0.067	6.4	2.992	2.094	0.898
	4	0.025	0.083	8.0	3.740	2.618	1.122
	4	0.04	0.120	11.5	5.386	3.770	1.616
	4	0.051	0.151	14.4	6.773	4.741	2.032
PVC (wound)	2	0.01	0.047	9.0	2.119	1.484	0.636
	2	0.02	0.089	17.0	3.989	2.793	1.197
	2	0.03	0.124	23.7	5.579	3.905	1.674
	2	0.04	0.156	29.7	6.981	4.887	2.094
	2	0.05	0.183	34.9	8.197	5.738	2.459
	4	0.01	0.078	7.5	3.522	2.465	1.057
	4	0.02	0.147	14.1	6.607	4.625	1.982
	4	0.03	0.208	19.9	9.350	6.545	2.805
	4	0.04	0.262	25.0	11.750	8.225	3.525
	4	0.05	0.309	29.5	13.869	9.708	4.161
Stainless Steel (wire-wound)	2	0.01	0.090	17.1	4.021	2.814	1.206
	2	0.02	0.157	30.0	7.044	4.931	2.113
	2	0.03	0.210	40.2	9.444	6.610	2.833
	2	0.04	0.253	48.4	11.376	7.963	3.413
	2	0.05	0.287	54.8	12.872	9.010	3.862
	4	0.01	0.177	16.9	7.948	5.563	2.384
	4	0.02	0.307	29.3	13.776	9.643	4.133
	4	0.03	0.410	39.1	18.388	12.872	5.517
	4	0.04	0.492	47.0	22.097	15.468	6.629
	4	0.05	0.560	53.4	25.120	17.584	7.536

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- Using a flow meter or pail and a stopwatch, determine the flow rate and calculate the time required to remove the required volume of water from the well;
- Place the purge water in 55-gallon drums or handle as indicated in the SSWP; and
- Lower the pump by stages until it is just above the screen, and continue to purge until the required volume of water has been removed from the well. In cases where the well will not yield water at a sufficient recharge rate, pump the well dry and allow it to recover.

9.2.3 Non-Gas Contact Bladder Pumps

- Assemble the teflon tubing, pump, and charged control box;
- Procedures for purging with a bladder pump are the same as for a submersible pump (Section 9.2.2); and
- Be sure to adjust the flow rate to prevent violent jolting of the hose.

9.2.4 Suction Pumps

- Assemble the pump, tubing, and power source; and
- Procedures for purging with a suction pump are the same as for a submersible pump (Section 9.2.2).

9.3 Sampling

Groundwater samples can be obtained through the use of a number of groundwater sampling devices. Each groundwater sampling device has its advantages (and disadvantages) over other devices. Ideally, groundwater sampling devices should be completely inert, economical to manufacturer, easily cleaned for reuse, able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for both well purging and sample collection. There are several other factors to consider when choosing a groundwater sampling device and care should be taken when selecting the device. Refer to the E & E Standard Operating Procedure for Groundwater Sampling Devices (ENV 3.6) for additional information.

9.3.1 Bailers

- Make sure that clean plastic sheeting has been placed around the well;

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- Attach a line to the bailer. If a bailer was used for purging, the same bailer and line may be used for sampling;
- Lower the bailer slowly and gently into the well, taking care not to shake the well casing or splash the bailer into the water. Lower the bailer to different points adjacent to the well screen to ensure that a representative water sample is collected;
- Slowly and gently retrieve the bailer from the well, avoiding contact with the well riser;
- Remove the cap from a sample container and place the cap on plastic sheeting or in a location where it will not be contaminated. Refer to Section 9.6 for special considerations for volatile organic analysis (VOA) samples;
- Slowly pour the water into the container;
- Filter and preserve samples as required by the SSWP. Mark the water level on the container with a pen;
- Prepare the necessary QA samples as outlined in the SSWP;
- Record sample information in the field logbook or on field data sheets, and complete the C-O-C form;
- Package samples in accordance with the SSWP; and
- Repeat this process until all groundwater samples have been collected.

9.3.2 Submersible Pumps

- Allow the monitoring well to recharge after purging, keeping the pump just above the screened interval;
- Attach a gate valve to the discharge hose, and reduce the flow rate to a manageable sampling rate;
- Prepare the sample containers;
- If no gate valve is available, discharge the sample into a clean jar and fill the sample containers from the jar;

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- Complete the sampling and documentation procedures as outlined in Section 9.3.1; and

- Upon completion, remove the pump and assembly and properly decontaminate the pump prior to use in the next well. Do not reuse the discharge tubing.

9.3.3 Bladder Pump

- Allow the well to recharge after purging;

- Prepare the sample containers;

- Turn the pump on. Increase the cycle time and reduce the pressure to the minimum that will allow groundwater to come to the surface;

- Complete the sampling and documentation procedures as outlined in Section 9.3.1;

- Upon completion, remove the tubing from the well and either replace the teflon tubing and bladder with new dedicated tubing and bladder, or properly decontaminate the existing material;

- Nonfiltered groundwater samples should be collected directly from the outlet tubing into the sample containers; and

- Filtered groundwater samples should be obtained by connecting the pump outlet tubing directly to the filter unit. The pump pressure should be reduced to prevent a pressure buildup on the filter, which could damage the pump bladder.

9.3.4 Suction Pumps

- Allow the well to recharge;

- Attach a gate valve to the discharge line if the suction pump discharge rate cannot be controlled, or discharge the sample into a clean glass jar and fill the sample containers from the jar;

- Sample as outlined in Section 9.3.1; and

- Upon completion, remove the tubing and properly decontaminate the pump prior to use in the next well. Do not reuse the tubing.

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9.4 Filtering

Samples being analyzed for total dissolved metals and total organic carbons (TOC) may require filtering. Two types of filters are commonly used: barrel filters and vacuum filters. A barrel filter works with a bicycle pump, which is used to build up positive pressure in the chamber containing the sample. Water is then forced through 0.45-µm filter paper into a jar. The barrel itself is filled manually.

A vacuum filter involves two chambers: the upper chamber contains the sample, and a 0.45-µm filter divides the two chambers. Using a portable vacuum pump, air is withdrawn from the lower chamber, creating a vacuum, which causes the sample to move through the filter into the lower chamber. Repeated pumping may be required to drain all of the sample into the lower chamber. If preservation of the samples is necessary, this should be done after filtering.

9.5 Post-Operation

After all samples have been collected and preserved, the sampling equipment should be properly decontaminated to prevent cross-contamination of samples.

- Decontaminate all equipment according to the SSWP;
- Replace sampling equipment in storage containers;
- Prepare groundwater samples for shipment. Check sample documentation and make sure samples are properly packed for shipment; and
- Organize field notes into a report format and transfer logging information to appropriate forms.

9.6 Special Consideration for VOA Sampling

The proper collection of a sample for dissolved VOCs requires minimal disturbance of the sample to limit volatilization and subsequent loss of volatiles from the sample.

Sample retrieval systems suitable for the valid collection of volatile organic samples include: positive-displacement bladder pumps, gear-driven submersible pumps, and syringe samplers and bailers. Field conditions and other constraints will limit the choice of appropriate systems. The principal objective is to provide a valid sample for analysis that has been subjected to the least amount of turbulence possible.

The following procedures should be followed when collecting VOA samples:

- Open the vial, set the cap in a clean place, and place the proper amount of preservatives (HCl) in the vial;
- Fill the vial to the top until a convex meniscus forms on the top of the vial. Do not overfill the vial;

- Check that the cap has not been contaminated, and carefully cap the vial. Place the cap directly over the top and screw down firmly. Do not overtighten and break the cap;

- Invert the vial and tap gently. If an air bubble appears, discard the sample and begin again. It is imperative that no entrapped air remains in the sample vial;

- Place the VOA vial in a cooler, oriented so that it is lying on its side, not straight up; and

- The holding time, under most protocol parameters, for VOAs is 14 days (see Table 1). It is recommended that samples be shipped or delivered to the laboratory daily. Ensure that the samples remain at 4 °C, but do not allow them to freeze.

10. Calculations

Table 6 presents the volume of water in different size casings and holes. To determine the volume of water in a well, the calculations are as follows:

$$V = Tr2(0.163)$$

where:

V = Static volume of well in gallons

T = Depth of water in well, measured in feet (determined by subtracting the static water level from the total depth of the well)

r = Inside radius of well casing, measured in inches

0.163 = A constant conversion factor for the conversion of the casing radius from inches to feet and cubic feet to gallons

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Table 6

VOLUME OF WATER IN CASING OR HOLE

Diameter of Casing or Hole (in)	Gallons per Foot of Depth	Cubic Feet per Foot of Depth	Liter per Meter of Depth	Cubic Meters per Meter of Depth
1	0.041	0.0055	0.509	0.509×10^{-3}
1.5	0.092	0.0123	1.142	1.142×10^{-3}
2	0.163	0.0218	2.024	2.024×10^{-3}
2.5	0.255	0.0341	3.167	3.167×10^{-3}
3	0.367	0.0491	4.558	4.558×10^{-3}
3.5	0.500	0.0668	6.209	6.209×10^{-3}
4	0.653	0.0873	8.110	8.110×10^{-3}
4.5	0.826	0.1104	10.260	10.260×10^{-3}
5	1.020	0.1364	12.670	12.670×10^{-3}
5.5	1.234	0.1650	15.330	15.330×10^{-3}
6	1.469	0.1963	18.240	18.240×10^{-3}
7	2.000	0.2673	24.840	24.840×10^{-3}
8	2.611	0.3491	32.430	32.430×10^{-3}
9	3.305	0.4418	41.040	41.040×10^{-3}
10	4.080	0.5454	50.670	50.670×10^{-3}
11	4.937	0.6600	61.310	61.310×10^{-3}
12	5.875	0.7854	72.960	72.960×10^{-3}
14	8.000	1.0690	99.350	99.350×10^{-3}
16	10.440	1.3960	129.650	129.650×10^{-3}
18	13.220	1.7670	164.180	164.180×10^{-3}
20	16.320	2.1820	202.680	202.680×10^{-3}
22	19.750	2.6400	245.280	245.280×10^{-3}
24	23.500	3.1420	291.850	291.850×10^{-3}
26	27.580	3.6870	342.520	342.520×10^{-3}
28	32.000	4.2760	397.410	397.410×10^{-3}
30	36.720	4.9090	456.020	456.020×10^{-3}
32	41.780	5.5850	518.870	518.870×10^{-3}
34	47.160	6.3050	585.680	585.680×10^{-3}
36	52.880	7.0690	656.720	656.720×10^{-3}

1 Gallon = 3.785 liters
1 Meter = 3.281 feet
1 Gallon water weighs 8.33 lbs. = 37.785 kilograms
1 Liter water weighs 1 kilogram = 2.205 pounds
1 Gallon per foot of depth = 12.419 liters per foot of depth
1 Gallon per meter of depth = 12.419×10^{-3} cubic meters per meter of depth

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11. Quality Assurance/Quality Control

The objective of QA/QC is to identify and implement methodologies that limit the introduction of error into sampling and analytical procedures. Groundwater sampling protocols appropriate to the data quality objectives and site conditions will define the specific procedures that will be followed for sampling events (see Figure 1).

There are seven primary areas of concern for QA in the collection of representative groundwater samples:

- The SSWP should be reviewed by all team personnel involved in the collection of the groundwater samples before any sampling is attempted, with attention to contaminant type and potential concentration variations;
- Log documentation should be reviewed to determine whether the required volume of purge water was removed from the well and that the temperature, electrical conductance, and pH had been stabilized to ensure that a representative water sample of the aquifer was obtained;
- The purging and sampling devices should be made of materials and utilized in a manner that will not interact with or alter the analysis;
- The results generated by these procedures are reproducible as demonstrated through the use of duplicate samples;
- The possibility of cross-contamination is reduced by collecting samples from the least contaminated well first. Rinsate blanks should be incorporated where dedicated sampling and purging equipment is not utilized and decontamination of the equipment between sampling events is required;
- Samples are properly labeled, documented (C-O-C), preserved, and shipped; and
- A record of daily field activities, such as sample collection and tracking information, is kept in a bound book.

12. Data Validation

The data generated will be reviewed according to the QA/QC considerations presented in Section 11.

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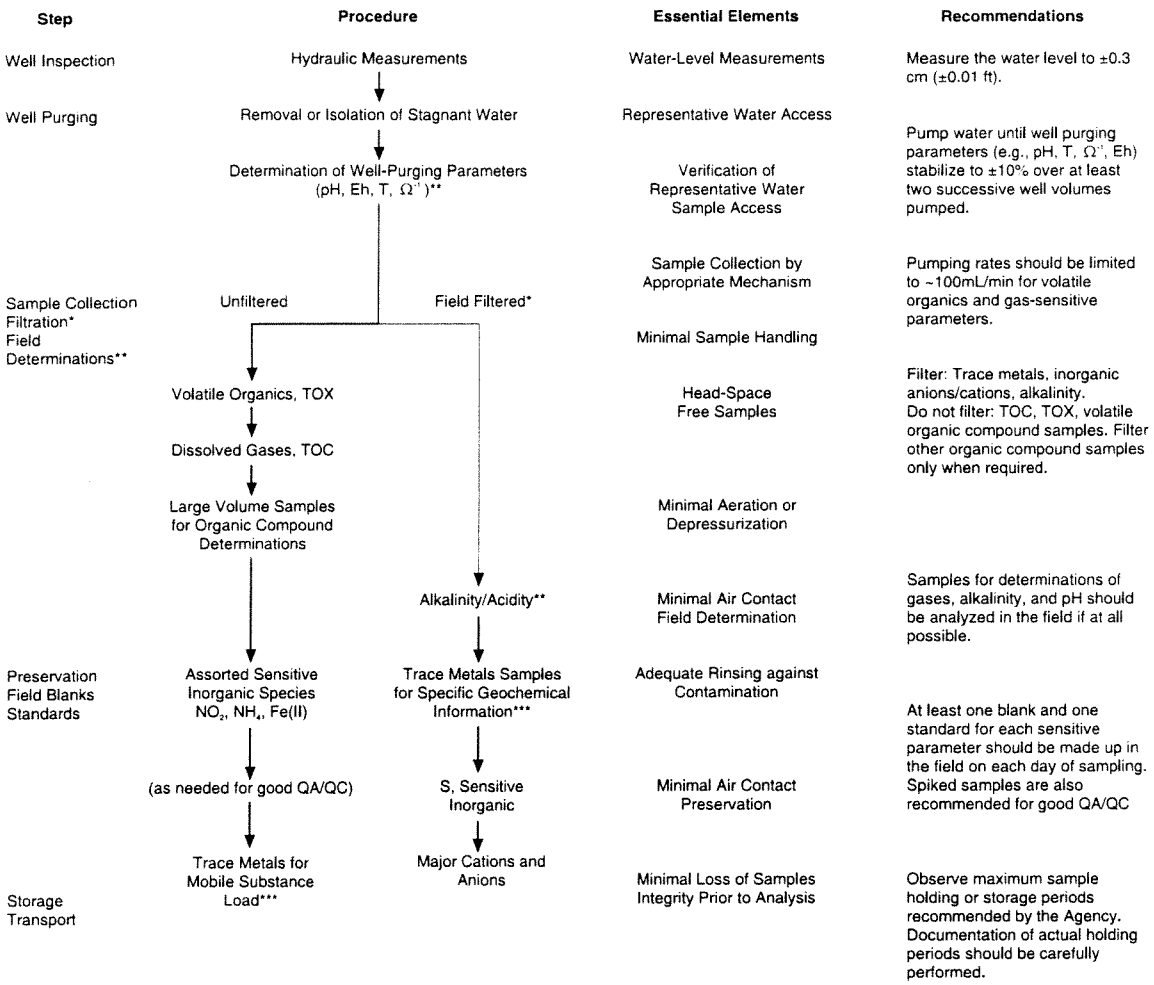
TITLE:	GROUNDWATER WELL SAMPLING		
CATEGORY:	ENV 3.7	REVISED:	March 1998



* Denotes samples that should be filtered to determine dissolved constituents. Filtration should be accomplished preferably with in-line filters and pump pressure or by N_2 pressure methods. Samples for dissolved gases or volatile organics should not be filtered. In instances where well development procedures do not allow for turbidity-free samples and may bias analytical results, split samples should be spiked with standards before filtration. Both spiked samples and regular samples should be analyzed to determine recoveries from both types of handling.

** Denotes analytical determinations that should be made in the field.

See Puls and Barcelona (1989).

Figure 1 Generalized Flow Diagram of Groundwater Sampling Protocol

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13. Health and Safety

Depending on the site-specific contaminants, the type of personnel protective equipment (PPE) used during the purging and sampling of the wells is outlined in the SSSP. The SSSP should be reviewed with specific emphasis placed on the safety procedures to be followed for the well sampling tasks. Standard safe operating practices should be followed, such as minimizing contact with potential contaminants in both the vapor phase and liquid matrix through the use of respirators and protective clothing.

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Field Sampling Plan
Attachment F
Standard Operating Procedure for Sample Packaging

TITLE:	SAMPLE PACKAGING
CATEGORY:	ENV 3.16
REVISED:	March 1998

STANDARD OPERATING PROCEDURE

SAMPLE PACKAGING

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TITLE:	SAMPLE PACKAGING
CATEGORY:	ENV 3.16
REVISED:	March 1998

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TITLE:	SAMPLE PACKAGING		
CATEGORY:	ENV 3.16	REVISED:	March 1998

1. Introduction

Liquid and solid environmental samples are routinely collected by E & E during field surveys, site investigations, and other site visits for laboratory analysis. Unless the samples have anesthetic, noxious, or other properties that could inhibit the ability of a flight crew member to perform his or her duty or are known to meet the established U.S. Department of Transportation criteria for hazardous material (i.e., explosive, corrosive, flammable, poisonous), they are not regulated as hazardous materials.

This Standard Operating Procedure (SOP) describes the packaging procedures to be used by E & E's staff to ensure the safe arrival of the samples at the laboratory for analyses. These procedures have been developed to reduce the risk of damage to the samples (i.e., breakage of the sample containers), promote the maintenance of sample temperature within the cooler, and prevent spillage of the sampled material should a container be broken.

In the event the sample material meets the established criteria of a DOT hazardous material, the reader is referred to E & E's Hazardous Materials/Dangerous Goods Shipping Guidance Manual (see H&S 5.5).

2. Scope

This SOP describes procedures for the packaging of environmental samples in:

- Coolers;
- Steel, aluminum and plastic drums; and
- 4GV fiberboard boxes.

The Hazardous Materials/Dangerous Goods Shipping Guidance Manual will complete the information needed for shipping samples by providing guidance on:

- Hazard determination for samples which meet the USDOT definition of a hazardous material;
- Shipping profiles for "standard" shipments;
- Shipping procedures for "non-standard" shipments;
- Marking of packages containing hazardous materials;

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- Labeling of packages containing hazardous materials; and
- Preparation of shipping papers for hazardous materials shipment.

3. Sample Packaging Procedures

3.1 General

It is E & E's intent to package samples so securely that there is no chance of leakage during shipment. This is to prevent the loss of samples and the expenditure of funds for emergency responses to spills and the efforts necessary to re-obtain the sample.

Over the years, E & E has developed several "standard" package configurations for the shipping of environmental samples. These standard package configurations are described below.

Liquid samples are particularly vulnerable. Because transporters (carriers) do not know the difference between a package leaking distilled water and a package leaking a hazardous chemical, they will react to a spill in an emergency fashion, potentially causing enormous expense to E & E for the cleanup of the sample material. Therefore, liquids are to be packed in multiple layers of plastic bags and absorbent/cushioning material to preclude any possibility of leaks from a package. This section defines the standard packaging configurations for environmental samples.

3.2 Liquid Environmental Sample Packaging Procedures

Liquid environmental samples should be collected and preserved as outlined in the Standard Operating Procedures (SOP) for Surface Water Sampling (ENV 3.12), and Groundwater Well Sampling (ENV 3.7). **Preserved water samples are not considered to meet the HM/DG definitions of Class 8 (Corrosive) and are therefore considered to be nonhazardous samples.** Liquid environmental samples may be shipped using an 80-quart cooler or an outer package consisting of either a steel or aluminum drum. Because the steel and aluminum drums provide little insulating capability, they should not be used for samples that require icing.

Packaging Liquid Environmental Samples Using the 80-Quart Cooler

- Label and seal all water sample bottles according to appropriate sampling SOPs;
- Secure the bottle caps using fiberglass tape; and
- Place each amber, poly, and volatile organic analysis (VOA) bottle in a sealable plastic bag. Mark the temperature blank VOA bag for identification.

If a foam block insert is used:

- Line the cooler with two plastic bags;

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- Place a foam insert (with holes cut to receive the sample bottles) inside the plastic bag;
- Place the bottles in the holes in the foam block;
- Fill void spaces with bagged ice to the top of the cooler;
- Fold over the plastic bags lining the cooler and secure shut with tape;
- Place Chain-of-Custody (C-O-C) form in a sealable bag and tape it to the inside of the cooler lid; and
- Secure the cooler with strapping tape and custody seal. Cover the custody seals with clear tape.

If vermiculite is used:

- Place 1 inch of inert absorbent material (vermiculite) in the bottom of the cooler;
- Line the cooler with two plastic bags;
- Place each sample bottle inside the inner bag;
- Fill the void spaces around the bottles with vermiculite to about half the height of the large bottles;
- Fill the remainder of the void spaces with bagged ice to within 4 inches of the top of the cooler, making sure the VOAs are in direct contact with a bag of ice;
- Fold over the plastic bags lining the cooler and secure shut with tape;
- Fill the remaining space in the cooler with vermiculite to the top of the cooler;
- Place C-O-C form in a sealable bag and tape it to the inside of the cooler lid; and
- Secure the cooler with strapping tape and custody seal. Cover the custody seals with clear tape.

Alternate Packaging Using 1A2/1B2 Drum

- Place 3 inches of inert absorbent material (vermiculite) in the bottom of the drum;
- Line the drum with two plastic bags;

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- Place each sample bottle inside the inner bag;
- Fill the void spaces around the bottles with vermiculite to the height of the larger bottles;
- Fold over the plastic bags lining the drum and secure shut with tape;
- Fill the remaining space in the drum with vermiculite to the top of the drum;
- Place C-O-C form in a sealable bag and tape it to the inside of the drum lid; and
- Secure the drum with closing ring and apply custody seals. Cover the custody seals with clear tape.

3.3 Soil/Sediment Environmental Sample Packaging Procedures

Soil/sediment environmental samples should be collected as outlined in the SOP for Soil Sampling (ENV 3.13), and SOP for Sediment Sampling (ENV 3.8). Soil/sediment environmental samples may be shipped using an 80-quart cooler, a 4GV fiberboard combination package, or an outer package consisting of either a steel or aluminum drum. Because the steel and aluminum drums provide little insulating capability, they should not be used for samples that require icing.

Packaging Soil/Sediment Environmental Samples

- Label and seal each sample container according to SOPs;
- Secure the bottle caps using fiberglass tape;
- Place each sample bottle inside a sealable plastic bag and place it in its original shipping box or in individual fiberboard boxes. Mark the temperature blank bag for identification; and
- Secure the original shipping box with strapping tape, place shipping box in a plastic bag, and secure the plastic bag with tape.

If an 80-quart cooler is used:

- Place bubble pack or similar material on the bottom and sides of an 80-quart cooler;
- Place the bagged shipping boxes in the cooler with a layer of bubble pack between each box;

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- Fill the void spaces with "blue ice" or ice in baggies to the top of the cooler;
- Place C-O-C form in a sealable baggie and tape it to the inside of the cooler lid; and
- Secure the cooler with strapping tape and custody seal. Cover the seals with clear tape.

If a 1A2/1B2 drum is used:

- Place 3 inches of inert absorbent material (vermiculite) in the bottom of the drum;
- Line the drum with two plastic garbage bags;
- Place the boxes inside the inner bag;
- Fill the space around the samples with vermiculite;
- Fold over the plastic bags lining the drum and secure shut with tape;
- Fill the remaining space around the bags with vermiculite to the top of the drum;
- Place C-O-C form in a sealable bag and tape it to the inside of the drum lid; and
- Secure the drum with the closing ring and apply custody seals. Cover the custody seals with clear tape.

Note: If a small number of samples are being shipped, it may be more practical to package them using the vermiculite or foam block configurations used for shipping liquid samples.

4. Shipping Procedures

Environmental samples are to be shipped as nonhazardous cargo. Unless the samples have anesthetic, noxious, or other properties that could inhibit the ability of a flight crew member to perform his or her duty or are known to meet the established U.S. Department of Transportation criteria for a hazardous material (i.e., explosive, corrosive, flammable, poisonous), they are not regulated as hazardous materials. When preparing the containers (i.e., cooler, drum, or box) for shipment, E & E staff **must** remove all labels from the outside container. Labels indicating that the contents may be hazardous are misleading and are not appropriate. Markings indicating ownership of the container, destination, and chain of custody labels are acceptable and can be attached as required.

When completing the paperwork for shipment, the standard nonhazardous forms must be used. Do not use the hazardous materials/dangerous goods airbills, either in total or in part; these forms are coded and their use will invite unnecessary questions. This will only serve to confuse Airborne or Federal Express' terminal personnel and will cause much frustration and the delay of sample shipment.

Environmental sample packages can be shipped overnight by both Airborne and Federal Express. When choosing between the two, cost should be considered. It is normally much cheaper to ship Airborne. For work conducted and paid for by E & E, it is E & E's policy that you must first attempt to ship by Airborne before considering Federal Express. In addition, Airborne tends to have remote locations open later in the evenings than Federal Express, which may be helpful when trying to complete a full day's sampling effort and still make the flights on time. Although both companies offer pickup of samples at the site, it is advisable to call ahead and ensure that this service is offered beforehand. In almost all cases, both companies will deliver to the laboratory of your choice on Saturdays. When planning for sampling activities, check with the companies in advance to verify pick-up and delivery schedules.

When dropping off the sample packages at Airborne or Federal Express, do not indicate that these samples may be hazardous. This will only raise questions and delay the shipment. Although you may have an idea of the kind of contamination that is contained in the samples, you must remember that you are sending these samples to a laboratory for analyses to determine the amount and type of contamination, if any. Until that is determined, the presumption is that the samples are environmental in nature and not subject to the hazardous materials restrictions.

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Field Sampling Plan
Attachment G
Standard Operating Procedure for Sampling Equipment Decontamination

TITLE:	Sampling Equipment Decontamination
CATEGORY:	ENV. 3.15
REVISED:	March 1999

STANDARD OPERATING PROCEDURE

SAMPLING EQUIPMENT DECONTAMINATION

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TITLE:	Sampling Equipment Decontamination		
CATEGORY:	ENV. 3.15	REVISED:	March 1999

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TITLE:	Sampling Equipment Decontamination		
CATEGORY:	ENV. 3.15	REVISED:	March 1999

1. Scope and Application

The purpose of this procedure is to provide a description of methods for preventing or reducing cross-contamination and general guidelines for designing and selecting decontamination procedures for use at potential hazardous waste sites. The decontamination procedures chosen will prevent introduction and cross-contamination of suspected contaminants in environmental samples, and will protect the health and safety of site personnel.

2. Method Summary

Removing or neutralizing contaminants that have accumulated on personnel and equipment ensures protection of personnel from permeating substances, reduces/eliminates transfer of contaminants to clean areas, prevents the mixing of incompatible substances, and minimizes the likelihood of sample contamination.

Cross-contamination can be removed by physical decontamination procedures. The abrasive and non-abrasive methods include the use of brushes, high pressure water, air and wet blasting, and high pressure Freon cleaning. These methods should be followed by a wash/rinse process using appropriate cleaning solutions. A general protocol for cleaning with solutions is as follows:

1. Physical removal.
2. Non-phosphate detergent plus tap water.
3. Tap water.
4. 10% nitric acid.
5. Distilled/deionized water rinse.
6. Solvent rinse.
7. Total air dry.
8. Triple rinse with distilled/deionized water.

This procedure can be expanded to include additional or alternate solvent rinses that will remove specified target compounds if required by site-specific work plans (WP) or as directed by a particular client.

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3. Interferences

The use of distilled/deionized water commonly available from commercial vendors may be acceptable for decontamination of sampling equipment provided that it has been verified by laboratory analysis to be analyte-free distilled/deionized water. Distilled water available from local grocery stores and pharmacies is generally not acceptable for final decontamination rinses. Contaminant-free deionized water is available from commercial vendors and may be shipped directly to the site or your hotel.

The use of an untreated potable water supply is not an acceptable substitute for tap water. Tap water may be used from any municipal water treatment system.

4. Equipment/Apparatus

The following are standard materials and equipment used as a part of the decontamination process:

- Appropriate protective clothing;
- Air purifying respirator (APR);
- Field log book;
- Non-phosphate detergent;
- Selected high purity, contaminant-free solvents;
- Long-handled brushes;
- Drop cloths (plastic sheeting);
- Trash containers;
- Paper towels;
- Galvanized tubs or equivalent (e.g., baby pools);
- Tap water;
- Contaminant-free distilled/deionized water;
- Metal/plastic container for storage and disposal of contaminated wash solutions;
- Pressurized sprayers, H_2O;
- Pressurized sprayers, solvents;
- Trash bags;
- Aluminum foil;
- Sample containers;
- Safety glasses or splash shield; and
- Emergency eyewash bottle.

5. Reagents

There are no reagents used in this procedure aside from decontamination solutions used for the equipment. The type of decontamination solution to be used shall depend upon the type and degree of contamination present and as specified in the project/site-specific Quality Assurance Project Plan (QAPP).

In general, the following solvents are utilized for decontamination purposes:

- 10% nitric acid wash (reagent grade nitric acid diluted with deionized/distilled water – 1 part acid to 10 parts water)[a];
- Acetone (pesticide grade)[b] ;
- Hexane (pesticide grade)[b];
- Methanol; and
- Methylene chloride[b].

[a]Only if sample is to be analyzed for trace metals.
[b]Only if sample is to be analyzed for organics requiring specific or specialized decontamination procedures. These solvents must be kept away from samples in order to avoid contamination by decon solvents.

6. Procedures

Decontamination is the process of removing or neutralizing contaminants that have accumulated on both personnel and equipment. Specific procedures in each case are designed accordingly and may be identified in either the Health and Safety Plan (HSP), WP, QAPP, or all three.

As part of the HSP, a personnel decontamination plan should be developed and set up before any personnel or equipment enters the areas of potential contamination. Decontamination procedures for equipment will be specified in the WP and the associated QAPP. These plans should include:

- Number and layout of decontamination stations;
- Decontamination equipment needed (see Section 4);
- Appropriate decontamination methods;
- Procedures to prevent contamination of clean areas;
- Methods and procedures to minimize worker contact with contaminants during removal of protective clothing;

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- Methods and procedures to prevent cross-contamination of samples and maintain sample integrity and sample custody; and
- Methods for disposal of contaminated clothing, equipment, and solutions.

Revisions to these plans may be necessary for health and safety when the types of protective clothing, site conditions, or on-site hazards are reassessed based on new information.

Prevention of Contamination

Several procedures can be established to minimize contact with waste and the potential for contamination. For example:

- Employing work practices that minimize contact with hazardous substances (e.g., avoid areas of obvious contamination, avoid touching potentially hazardous substances);
- Use of remote sampling, handling, and container-opening techniques;
- Covering monitoring and sampling equipment with plastic or other protective material;
- Use of disposable outer garments and disposable sampling equipment with proper containment of these disposable items;
- Use of disposable towels to clean the outer surfaces of sample bottles before and after sample collection; and
- Encasing the source of contaminants with plastic sheeting or overpacks.

Proper procedures for dressing prior to entrance into contaminated areas will minimize the potential for contaminants to bypass the protective clothing. Generally, all fasteners (zippers, buttons, snaps, etc.) should be used, gloves and boots tucked under or over sleeves and pant legs, and all junctures taped (see the Health and Safety Plan for these procedures).

Decontamination Methods

All personnel, samples, and equipment leaving the contaminated area of a site must be decontaminated to remove any chemicals or infectious organisms that may have adhered to them. Various decontamination methods will either physically remove, inactivate by chemical detoxification/disinfection/sterilization, or remove contaminants by both physical and chemical means.

In many cases, gross contamination can be removed by physical means. The physical decontamination techniques can be grouped into two categories: abrasive methods and non-abrasive methods.

6.1 Abrasive Cleaning Methods

Abrasive cleaning methods work by rubbing and wearing away the top layer of the surface containing the contaminant. The following reviews the available abrasive methods.

Mechanical

Mechanical methods include using brushes with metal, nylon, or natural bristles. The amount and type of contaminants removed will vary with the hardness of bristles, length of time brushing, and degree of brush contact. Material may also be removed by using appropriate tools to scrape, pry, or otherwise remove adhered materials.

Air Blasting

Air blasting equipment uses compressed air to force abrasive material through a nozzle at high velocities. The distance between nozzle and surface cleaned, air pressure, and time of air blasting dictate cleaning efficiency. The method's disadvantages are its inability to control the exact amount of material removed and its large amount of waste generated.

Wet Blasting

Wet blast cleaning involves the use of a suspended fine abrasive. The abrasive/water mixture is delivered by compressed air to the contaminated area. By using very fine abrasives, the amount of materials removed can be carefully controlled.

6.2 Non-abrasive Cleaning Methods

Non-abrasive cleaning methods work by either dissolution or by forcing the contaminant off a surface with pressure. In general, less of the equipment surface is removed using non-abrasive methods.

High-Pressure Water

This method consists of a high-pressure pump, an operator controlled directional nozzle, and high-pressure hose. Operating pressure usually ranges from 340 to 680 psi, which relates to flow rates of 20 to 140 lpm.

Steam Cleaning

This method uses water delivered at high pressure and high temperature in order to remove accumulated solids and/or oils.

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Ultra-High-Pressure Water

This system produces a water jet from 1,000 to 4,000 atm. This ultra-high-pressure spray can remove tightly-adhered surface films. The water velocity ranges from 500 m/sec. (1,000 atm) to 900 m/sec. (4,000 atm). Additives can be used to enhance the cleaning action, if approved by the QAPP for the project.

High-Pressure Freon Cleaning

Freon cleaning is a very effective method for cleaning cloth, rubber, plastic, and external/internal metal surfaces. Freon 113 (trichlorotriflorethane) is dense, chemically stable, relatively non-toxic, and leaves no residue. The vapor is easily removed from the air by activated charcoal. A high pressure (1,000 atm) jet of liquid Freon 113 is directed onto the surface to be cleaned. The Freon can be collected in a sump, filtered, and reused.

Physical removal of gross contamination should be followed by a wash/rinse process using cleaning solutions. One or more of the following methods utilize cleaning solutions.

Dissolving

Removal of surface contaminants can be accomplished by chemically dissolving them, although the solvent must be compatible with the equipment and protective clothing. Organic solvents include alcohols, ethers, ketones, aromatics, straight-chain alkanes, and common petroleum products. Halogenated solvents are generally incompatible with protective clothing and are toxic. Table 1 provides a general guide to the solubility of contaminant categories in four types of solvents.

Surfactants

Surfactants reduce adhesion forces between contaminants and the surface being cleaned and prevents reposition of the contaminants. Non-phosphate detergents dissolved in tap water is an acceptable surfactant solution.

Rinsing

Contaminants are removed and rinsing through dilution, physical attraction, and solubilization.

Disinfection/Sterilization

Disinfectants are a practical means of inactivating infectious agents. Unfortunately, standard sterilization methods are impractical for large equipment and personal protective clothing.

6.3 Field Sampling Equipment Cleaning Procedures

The following steps for equipment cleaning should be followed for general field sampling activities.

1. Physical removal (abrasive or non-abrasive methods).
2. Scrub with non-phosphate detergent plus tap water.
3. Tap water rinse.
4. 10% nitric acid (required during sampling for inorganics only).
5. Distilled/deionized water rinse.
6. Solvent rinse (required during sampling for organics only).
7. Total air dry (required during sampling for organics only).
8. Triple rinse with distilled/deionized water.

Table 1 lists solvent rinses which may be required for elimination of particular chemicals. After each solvent rinse, the equipment should be air-dried and triple-rinsed with distilled/deionized water.

Table 1

DECONTAMINATION SOLVENTS

Solvent	Soluble Contaminants
Water	Low-chain compounds Salts Some organic acids and other polar compounds
Dilute Bases For example: – detergent – soap	Acidic compounds Phenol Thiols Some nitro and sulfonic compounds
Organic Solvents: For example: – alcohols (methanol) – ethers – ketones – aromatics – straight-chain alkanes (e.g., hexane) – common petroleum products (e.g., fuel oil, kerosene)	Nonpolar compounds (e.g., some organic compounds)
WARNING: Some organic solvents can permeate and/or degrade the protective clothing.	

Solvent rinses are not necessarily required when organics are not a contaminant of concern. Similarly, an acid rinse is not necessarily required if analysis does not include inorganics.

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NOTE: Reference the appropriate analytical procedure for specific decontamination solutions required for adequate removal of the contaminants of concern.

Sampling equipment that requires the use of plastic or teflon tubing should be disassembled, cleaned, and the tubing replaced with clean tubing, if necessary, before commencement of sampling or between sampling locations.

7. Quality Assurance/Quality Control

QA/QC samples are intended to provide information concerning possible cross-contamination during collection, handling, preparation, and packing of samples from field locations for subsequent review and interpretation. A field blank (rinsate blank) provides an additional check on possible sources of contamination from ambient air and from sampling instruments used to collect and transfer samples into sample containers.

A field blank (rinsate blank) consists of a sample of analyte-free water passed through/over a precleaned/decontaminated sampling device and placed in a clean area to attempt to simulate a worst-case condition regarding ambient air contributions to sample contamination.

Field blanks should be collected at a rate of one per day per sample matrix even if samples are not shipped that day. The field blanks should return to the lab with the trip blanks originally sent to the field and be packed with their associated matrix.

The field blank places a mechanism of control on equipment decontamination, sample handling, storage, and shipment procedures. It is also indicative of ambient conditions and/or equipment conditions that may affect the quality of the samples.

Holding times for field blanks analyzed by CLP methods begin when the blank is received in the laboratory (as documented on the chain of parameters and associated analytical methods).

Holding times for samples and blanks analyzed by SW-846 or the 600 and 500 series begins at the time of sample collection.

8. Health and Safety

Decontamination can pose hazards under certain circumstances even though performed to protect health and safety. Hazardous substances may be incompatible with decontamination methods (i.e., the method may react with contaminants to produce heat, explosion, or toxic products). Decontamination methods may be incompatible with clothing or equipment (e.g., some solvents can permeate and/or degrade protective clothing). Also, a direct health hazard to workers can be posed from chemical decontamination solutions that may be hazardous if inhaled or may be flammable.

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The decontamination solutions must be determined to be compatible before use. Any method that permeates, degrades, or damages personal protective equipment should not be used. If decontamination methods do pose a direct health hazard, measures should be taken to protect personnel or modified to eliminate the hazard.

All site-specific safety procedures should be followed for the cleaning operation. At a minimum, the following precautions should be taken:

1. Safety glasses with splash shields or goggles, neoprene gloves, and laboratory apron should be worn.

2. All solvent rinsing operations should be conducted under a fume hood or in open air.

3. No eating, smoking, drinking, chewing, or any hand-to-mouth contact is permitted.

9. References

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

A Compendium of Superfund Field Operations Methods, EPA 540/p-87/001.

Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual, USEPA Region IV, April 1, 1986.

Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities, NIOSH/OSHA/USCG/EPA, October 1985.

Field Sampling Plan
Attachment H
Standard Operating Procedure for Soil Sampling

TITLE:	SOIL SAMPLING		
CATEGORY:	ENV 3.13	REVISED:	August 1997

STANDARD OPERATING PROCEDURE

SOIL SAMPLING

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368 Pleasant View Drive / Lancaster, New York 14086 / (716) 684-8060

TITLE:	**SOIL SAMPLING**
CATEGORY:	ENV 3.13
REVISED:	August 1997

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TITLE:	SOIL SAMPLING		
CATEGORY:	ENV 3.13	REVISED:	August 1997

1. Introduction

This document describes the procedures for the collection of representative soil samples. Representative sampling ensures the accurate characterization of site conditions. Analysis of soil samples may determine pollutant concentrations and the accompanying risks to public health, welfare, or the environment.

2. Scope

Included in this discussion are procedures for obtaining representative samples, quality assurance/quality control (QA/QC) measures, proper documentation of sampling activities, and recommendations for personnel safety.

3. Method Summary

Soil samples may be recovered using a variety of methods and equipment. These are dependent on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type.

Samples of near-surface soils may be easily obtained using a spade, stainless-steel spoon, trowel, or scoop. Sampling at greater depths may be performed using a hand auger, a power auger, or, if a test pit is required, a backhoe.

All sampling devices should be cleaned using pesticide-grade acetone (assuming that acetone is not a target compound) or methanol, then wrapped in clean aluminum foil, and custody sealed for identification. The sampling equipment should remain in this wrapping until it is needed. Each sampler should be used for one sample only. However, dedicated tools may be impractical if there are a large number of soil samples required. In this case, samplers should be cleaned in the field using standard decontamination procedures as outlined in E & E's SOP for Equipment Decontamination (see ENV 3.15).

4. Sample Preservation, Containers, Handling, and Storage

The chemical preservation of solids is not generally recommended. Refrigeration is usually the best approach, supplemented by a minimal holding time.

Soil samples should be handled according to the procedures outlined in E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).

5. Potential Problems

Potential problems with soil sampling include cross-contamination of samples and improper sample collection. Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment and bottles. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection is generally the result of the use of

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Appendix A
Site Safety and Health Plan

ecology and environment, inc.

SITE-SPECIFIC
HEALTH AND SAFETY PLAN

Project: Former Laredo Air Force Base

Project No.: 001289.UX06.01

TDD/PAN No.:

Project Location: Laredo, TX

Proposed Date of Field Activities: 7/16/01

Project Director: Frances Verhalen

Project Manager: Don Smith

Site Safety Officer: Don Smith

Prepared by: Melissa Fritsch Date Prepared: 6/27/01

Approved by: Don Smith Date Approved: 7/13/01

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1. INTRODUCTION

1.1 POLICY

It is E & E's policy to ensure the health and safety of its employees, the public, and the environment during the performance of work it conducts. This site-specific health and safety plan (SHASP) establishes the procedures and requirements to ensure the health and safety of E & E employees for the above-named project. E & E's overall safety and health program is described in *Corporate Health and Safety Program for Toxic and Hazardous Substances* (CHSP). After reading this plan, applicable E & E employees shall read and sign E & E's Site-Specific Health and Safety Plan Acceptance form.

This SHASP has been developed for the sole use of E & E employees and is not intended for use by firms not participating in E & E's training and health and safety programs. Subcontractors are responsible for developing and providing their own safety plans.

This SHASP has been prepared to meet the following applicable regulatory requirements and guidance:

Applicable Regulation/Guidance
29 CFR 1910.120, Hazardous Waste Operations and Emergency Response (HAZWOPER)
Other: USACE EM 385-1-1

1.2 SCOPE OF WORK

Description of Work: Will conduct sampling mission to determine future regulatory activities.

Equipment/Supplies: See equipment checklist.

The following is a description of each numbered task:

Task Number	Task Description
1	Photo documentation and logbook documentation
2	Drilling monitoring wells
3	Soil Sampling

1.3 SITE DESCRIPTION

Site Map: A site map or sketch is attached at the end of this plan.

Site History/Description (see project work plan for detailed description): Former military base location from 1942-1974. Land sold to several federal, state, county agencies, and private firms for development. Now residential and golf course development with former air field now Laredo International Airport.

Is the site currently in operation? ☐ Yes ☒ No

Locations of Contaminants/Wastes: Soil

Types and Characteristics of Contaminants/Wastes:

☐ Liquid	☒ Solid	☐ Sludge	☐ Gas/Vapor
☐ Flammable/Ignitable	☐ Volatile	☐ Corrosive	☐ Acutely Toxic
☐ Explosive	☐ Reactive	☐ Carcinogenic	☐ Radioactive
☐ Medical/Pathogenic	☒ Poison	Other:	

2. ORGANIZATION AND RESPONSIBILITIES

E & E team personnel shall have on-site responsibilities as described in E & E's standard operating procedure (SOP) for Site Inspection. The project team, including qualified alternates, is identified below.

Name	Site Role/Responsibility
Don Smith	Project/Task Manager
Don Smith	Site Safety Officer
Bill Huey	Subcontractor Oversight
Melissa Fritsch	Field Sampler

3. TRAINING

Prior to work, E & E team personnel shall have received training as indicated below. As applicable, personnel shall have read the project work plan, sampling and analysis plan, and/or quality assurance project plan prior to project work.

Training	Required
40-Hour OSHA HAZWOPER Initial Training and Annual Refresher (29 CFR 1910.120)	X
Annual First Aid/CPR	X
Hazard Communication (29 CFR 1910.1200)	X
40-Hour Radiation Protection Procedures and Investigative Methods	
8-Hour General Radiation Health and Safety	
Radiation Refresher	
DOT and Biannual Refresher	X
Other:	

4. MEDICAL SURVEILLANCE

4.1 MEDICAL SURVEILLANCE PROGRAM

E & E field personnel shall actively participate in E & E's medical surveillance program as described in the CHSP and shall have received, within the past year, an appropriate physical examination and health rating.

E & E's health and safety record (HSR) form will be maintained on site by each E & E employee for the duration of his or her work. E & E employees should inform the site safety officer (SSO) of any allergies, medical conditions, or similar situations that are relevant to the safe conduct of the work to which this SHASP applies.

Is there a concern for radiation at the site? ☐ Yes ☒ No
If no, go to 5.1.

4.2 RADIATION EXPOSURE

4.2.1 External Dosimetry

Thermoluminescent Dosimeter (TLD) Badges: TLD badges are required to be worn by all E & E field personnel on all E & E sites.

Pocket Dosimeters: _____

Other: _____

4.2.2 Internal Dosimetry

☐ Whole body count ☐ Bioassay ☐ Other

Requirements: _____

4.2.3 Radiation Dose

Dose Limits: E & E's radiation dose limits are stated in the CHSP. Implementation of these dose limits may be designated on a site specific basis.

Site-Specific Dose Limits:

ALARA Policy: Radiation doses to E & E personnel shall be maintained as low as reasonably achievable (ALARA), taking into account the work objective, state of technology available, economics of improvements in dose reduction with respect to overall health and safety, and other societal and socioeconomic considerations.

5. SITE CONTROL

5.1 SITE LAYOUT AND WORK ZONES

Site Work Zones: Refer to the map or site sketch, attached at the end of this plan, for designated work zones.

Site Access Requirements and Special Considerations: Fenced in areas will be accessed by client.

Illumination Requirements: All work done in daylight.

Sanitary Facilities (e.g., toilet, shower, potable water): Nearest convenience gas station

On-Site Communications: Verbal, vehicle horn if needed

Other Site-Control Requirements: None.

5.2 SAFE WORK PRACTICES

Daily Safety Meeting: A daily safety meeting will be conducted for all E & E personnel and subcontractors and documented on the Daily Safety Meeting Record form or in the field logbook. The information and data obtained from applicable site characterization and analysis will be addressed in the safety meetings and also used to update this SHASP, as necessary.

Work Limitations: Work shall be limited to a maximum of 12 hours per day. If 12 consecutive days are worked, at least one day off shall be provided before work is resumed. Work will be conducted in daylight hours unless prior approval is obtained and the illumination requirements in 29 CFR 1910.120(m) are satisfied.

Weather Limitations: Work shall not be conducted during electrical storms. Work conducted in other inclement weather (e.g., rain, snow) will be approved by project management and the regional safety coordinator or designee.

Other Work Limitations:

Buddy System: Field work will be conducted in pairs of team members according to the buddy system.

Line of Sight: Each field team member shall remain in the line of sight and within verbal communication of at least one other team member.

Eating, Drinking, and Smoking: Eating, drinking, smoking, and the use of tobacco products shall be prohibited in the exclusion and contamination reduction areas, at a minimum, and shall only be permitted in designated areas.

Contamination Avoidance: Field personnel shall avoid unnecessary contamination of personnel, equipment, and materials to the extent practicable.

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Sample Handling: Protective gloves of a type designated in Section 7 will be worn when containerized samples are handled for labeling, packaging, transportation, and other purposes.

Vermiculite Handling: Sample packaging materials other than vermiculite will be used.

Other Safe Work Practices:

6. HAZARD EVALUATION AND CONTROL

6.1 PHYSICAL HAZARD EVALUATION AND CONTROL

Potential physical hazards and their applicable control measures are described in the following table for each task.

Hazard	Task Number	Hazard Control Measures
Biological (flora, fauna, etc.)	1, 2, 3	• Potential hazard: Poison ivy, snakes. • Establish site-specific procedures for working around identified hazards. • Other: Insects; areas of "grown-up" vegetation
Cold Stress		• Provide warm break area and adequate breaks. • Provide warm noncaffeinated beverages. • Promote cold stress awareness. • See *Cold Stress Prevention and Treatment* (attached at the end of this plan if cold stress is a potential hazard).
Compressed Gas Cylinders		• Use caution when moving or storing cylinders. • A cylinder is a projectile hazard if it is damaged or its neck is broken. • Store cylinders upright and secure them by chains or other means. • Other:
Confined Space		• Ensure compliance with 29 CFR 1910.146. • See SOP for Confined Space Entry. Additional documentation is required.
Drilling	2,	• See SOP for Health and Safety on Drilling Rig Operations. Additional documentation may be required. • Other: • Other:
Drums and Containers	1, 2, 3	• Ensure compliance with 29 CFR 1910.120(j). • Consider unlabeled drums or containers to contain hazardous substances and handle accordingly until the contents are identified. • Inspect drums or containers and assure integrity prior to handling. • Move drums or containers only as necessary; use caution and warn nearby personnel of potential hazards. • Open, sample, and/or move drums or containers in accordance with established procedures; use approved drum/container-handling equipment. • Other:

Hazard	Task Number	Hazard Control Measures
Electrical		• Ensure compliance with 29 CFR 1910 Subparts J and S. • Locate and mark energized lines. • De-energize lines as necessary. • Ground all electrical circuits. • Guard or isolate temporary wiring to prevent accidental contact. • Evaluate potential areas of high moisture or standing water and define special electrical needs. • Other:
Excavation and Trenching		• Ensure that excavations comply with and personnel are informed of the requirements of 29 CFR 1926 Subpart P. • Ensure that any required sloping or shoring systems are approved as per 29 CFR 1926 Subpart P. • Identify special personal protective equipment (PPE) (see Section 7) and monitoring (see Section 8) needs if personnel are required to enter approved excavated areas or trenches. • Maintain line of sight between equipment operators and personnel in excavations/trenches. Such personnel are prohibited from working in close proximity to operating machinery. • Suspend or shut down operations at signs of cave in, excessive water, defective shoring, changing weather, or unacceptable monitoring results. • Other: • Other:
Fire and Explosion		• Inform personnel of the location(s) of potential fire/explosion hazards. • Establish site-specific procedures for working around flammables. • Ensure that appropriate fire suppression equipment and systems are available and in good working order. • Define requirements for intrinsically safe equipment. • Identify special monitoring needs (see Section 8). • Remove ignition sources from flammable atmospheres. • Coordinate with local fire-fighting groups regarding potential fire/explosion situations. • Establish contingency plans and review daily with team members. • Other:
Heat Stress	1, 2, 3	• Provide cool break area and adequate breaks. • Provide cool noncaffeinated beverages. • Promote heat stress awareness. • Use active cooling devices (e.g., cooling vests) where specified. • See *Heat Stress Prevention and Treatment* (attached at the end of this plan if heat stress is a potential hazard).
Heavy Equipment Operation	2	• Define equipment routes, traffic patterns, and site-specific safety measures. • Ensure that operators are properly trained and equipment has been properly inspected and maintained. Verify back-up alarms. • Ensure that ground spotters are assigned and informed of proper hand signals and communication protocols. • Identify special PPE (Section 7) and monitoring (Section 8) needs.

Hazard	Task Number	Hazard Control Measures
Heavy Equipment Operation (Cont'd)		• Ensure that field personnel do not work in close proximity to operating equipment. • Ensure that lifting capacities, load limits, etc., are not exceeded. • Other:
Heights (Scaffolding, Ladders, etc.)		• Ensure compliance with applicable subparts of 29 CFR 1910. • Identify special PPE needs (e.g., lanyards, safety nets, etc.) • Other:
Noise	, 2,	• Establish noise level standards for on-site equipment/operations. • Inform personnel of hearing protection requirements (Section 7). • Define site-specific requirements for noise monitoring (Section 8). • Other:
Overhead Obstructions		• Wear hard hat. • Other:
Power Tools Weed eater		• Ensure compliance with 29 CFR 1910 Subpart P. • Other:
Sunburn	1, 2, 3	• Apply sunscreen. • Wear hats/caps and long sleeves. • Other:
Utility Lines	1, 2, 3	• Identify/locate existing utilities prior to work. • Ensure that overhead, underground, and nearby utility lines are at least 25 feet away from project activities. • Contact utilities to confirm locations, as necessary. • Other:
Weather Extremes	1, 2, 3	• Potential hazards: heat • Establish site-specific contingencies for severe weather situations. • Provide for frequent weather broadcasts. • Weatherize safety gear, as necessary (e.g., ensure eye wash units cannot freeze, etc.). • Identify special PPE (Section 7) needs. • Discontinue work during severe weather. • Other:
Site Debris	1, 2, 3	• Trip/fall hazard – walk carefully. • Flying debris.
Water Hazard		•
Other:		• •
Other:		• •

6.2 CHEMICAL HAZARD EVALUATION AND CONTROL

6.2.1 Chemical Hazard Evaluation

Potential chemical hazards are described by task number in Table 6-1. Hazard Evaluation Sheets for major known contaminants are attached at the end of this plan.

Table 6-1

CHEMICAL HAZARD EVALUATION

Task Number	Compound	Exposure Limits (TWA)			Dermal Hazard (Y/N)	Route(s) of Exposure	Acute Symptoms	Odor Threshold/ Description	FID/PID	
		PEL	REL	TLV					Relative Response	Ioniz. Poten. (eV)
1, 2, 3	Lead	0.05 mg/m^3	0.05 mg/m^3	0.1 mg/m^3	Y	Inhalation, absorption, ingestion, dermal contact	Headache, visual disturbance, skin redness	Unknown/ odorless	0	0
1, 2, 3	Benzene	1 ppm	0.1 ppm	0.5 ppm	Y	Inhalation, absorption, ingestion, dermal contact	Skin, eye, nose irritant, nausea, dizziness	4.68 ppm / aromatic odor	Unknown	9.25
1, 2, 3	Chromium	1.0 mg/m^3	0.5 mg/m^3	0.5 mg/m^3	Y	Inhalation, absorption, ingestion, dermal contact	Ulceration of skin & nasal mucosa, eye & skin irritant	None	Unknown	0
1, 2, 3	Cadmium*	.005 mg/m^3	0.1 mg/m^3	.005 mg/m^3	Y	Inhalation & ingestion	Nose, throat irritation, nausea, vomiting, diarrhea	None	Unknown	0
1, 2, 3	Aldrin*	0.25 mg/m^3	0.25 mg/m^3	0.25 mg/m^3	Y	Absorption, ingestion, dermal contact	Irritate skin	None	Unknown	0
1, 2, 3	Dieldrin	0.25 mg/m^3	0.25 mg/m^3	0.25 mg/m^3	Y	Inhalation, absorption, ingestion, dermal contact	Headache, nausea, dizziness, vomiting	0.041 ppm	Unknown	0

Note: Use an asterisk (*) to indicate known or suspected carcinogens.

6.2.2 Chemical Hazard Control

An appropriate combination of engineering/administrative controls, work practices, and PPE shall be used to reduce and maintain employee exposures to a level at or below published exposure levels (see Section 6.2.1).

Applicable Engineering/Administrative Control Measures: **Not Applicable**

PPE: See Section 7.

6.3 RADIOLOGICAL HAZARD EVALUATION AND CONTROL

6.3.1 Radiological Hazard Evaluation

Potential radiological hazards are described below by task number. Hazard Evaluation Sheets for major known contaminants are attached at the end of this plan.

Task Number	Radionuclide	DAC (μCi/ml)	Route(s) of Exposure	Major Radiation(s)	Energy(s) (MeV)	Half-Life
		Not	Applicable			

6.3.2 Radiological Hazard Control

Engineering/administrative controls and work practices shall be instituted to reduce and maintain employee exposures to a level at or below the permissible exposure/dose limits (see sections 4.2.3 and 6.3.1). Whenever engineering/administrative controls and work practices are not feasible or effective, any reasonable combination of engineering/administrative controls, work practices, and PPE shall be used to reduce and maintain employee exposures to a level at or below permissible exposure/dose limits.

Applicable Engineering/Administrative Control Measures: Not applicable

PPE: See Section 7.

7. LEVEL OF PROTECTION AND PERSONAL PROTECTIVE EQUIPMENT

7.1 LEVEL OF PROTECTION

The following levels of protection (LOPs) have been selected for each work task based on an evaluation of the potential or known hazards, the routes of potential hazard, and the performance specifications of the PPE. On-site monitoring results and other information obtained from on-site activities will be used to modify these LOPs and the PPE, as necessary, to ensure sufficient personnel protection. The authorized LOP and PPE shall only be changed with the approval of the regional safety coordinator or designee. Level A is not included below because Level A activities, which are performed infrequently, will require special planning and addenda to this SHASP.

Task Number	B	C	D	Modifications Allowed
1			X	
2		(X)*	X	
3		(X)*	X	

Note: Use "X" for initial levels of protection. Use "(X)" to indicate levels of protection that may be used as site conditions warrant.
* - Air monitoring detects above limits.

7.2 PERSONAL PROTECTIVE EQUIPMENT

The PPE selected for each task is indicated below. E & E's PPE program complies with 29 CFR 1910.120 and 29 CFR 1910 Subpart I and is described in detail in the CHSP. Refer to 29 CFR 1910 for the minimum PPE required for each LOP.

PPE	Task Number/LOP				
	1	2	3		
Full-face APR	(X)	(X)	(X)		
PAPR					
Cartridges:					
H/P-100					
GMC-H/GMC-P-100/GME-P-100	(X)	(X)	(X)		
GMA-H/GMA-P-100					
Other:					
Positive-pressure, full-face SCBA					
Spare air tanks (Grade D air)					
Positive-pressure, full-face, supplied-air system					
Cascade system (Grade D air)					
Manifold system					
5-Minute escape mask					
Safety glasses	X	X	X		
Monogoggles					
Coveralls/clothing	X	X	X		
Protective clothing:					

PPE	Task Number/LOP				
	1	2	3		
Tyvek	(X)	(X)	(X)		
Saranex					
Other:					
Splash apron					
Inner gloves:					
Cotton					
Nitrile	(X)	(X)	(X)		
Latex	X	X	X		
Other:					
Outer gloves:					
Viton					
Rubber					
Neoprene					
Nitrile					
Other:					
Work gloves (type: Leather)	X	X	X		
Leather safety boots (as per ANSI Z41)	X	X	X		
Neoprene safety boots (as per ANSI Z41)					
Boot covers (type: Latex)	X	X	X		
Hearing protection (type:)	X	X	X		
Hard hat	X	X	X		
Face shield					
Other:					
Other:					

8. HEALTH AND SAFETY MONITORING

Health and safety monitoring will be conducted to ensure proper selection of engineering/administrative controls, work practices, and/or PPE so that employees are not exposed to hazardous substances at levels that exceed permissible exposure/dose limits or published exposure levels. Health and safety monitoring will be conducted using the instruments, frequency, and action levels described in Table 8-1. Health and safety monitoring instruments shall have been appropriately calibrated and/or performance-checked prior to use.

Table 8-1

HEALTH AND SAFETY MONITORING

Instrument	Task Number	Contaminant(s)	Monitoring Location	Monitoring Frequency	Action Levels[a]	
☒ PID (e.g., HNu IS-101) ☐ FID (e.g., OVA 128-GC) ☐ PID/FID (e.g., TVA)	1, 2, 3	Benzene	Breathing zone	Periodic	**Unknown Vapors** Background to 1 ppm: Level D 1 to 5 ppm above background: Level C 5 to 500 ppm above background: Level B >500 ppm above background: Level A	**Contaminant-Specific**
Oxygen Meter/Explosimeter					**Oxygen** <19.5% or >22.0%: Evacuate area; eliminate ignition sources; reassess conditions. 19.5 to 22.0%: Continue work in accordance with action levels for other instruments.	**Explosivity** ≤10% LEL: Continue work in accordance with action levels for other instruments; monitor continuously for combustible atmospheres. >10% LEL: Evacuate area; eliminate ignition sources; reassess conditions.
Radiation Alert Monitor (Rad-mini or RAM-4)					<0.1 mR/hr: Continue work in accordance with action levels for other instruments. ≥0.1 mR/hr: Evacuate area; reassess work plan and contact radiation safety specialist.	
Mini-Ram Particulate Monitor					**General/Unknown** Evaluate health and safety measures when dust levels exceed 2.5 milligrams per cubic meter.	**Contaminant-Specific**
Single-gas personal Monitors (Monitox, STX-70)					Contaminant-specific:	
Draeger Colorimetric Tubes					**Tube** **Action Level** **Action**	

Table 8-1

HEALTH AND SAFETY MONITORING

Instrument	Task Number	Contaminant(s)	Monitoring Location	Monitoring Frequency	Action Levels[a]
Air Monitor/Sampler Type:_____ Sampling medium: _____					**Action Level** **Action**
Personal Sampling Pump Type:_____ Sampling medium: _____					**Action Level** **Action**
Micro R Meter					<2 mR/hr: Continue work in accordance with action levels for other instruments. 2 to 5 mR/hr: In conjunction with a radiation safety specialist, continue work and perform stay-time calculations to ensure compliance with dose limits and ALARA policy. >5 mR/hr: Evacuate area to reassess work plan and evaluate options to maintain personnel exposures ALARA and within dose limits.
Ion Chamber					See micro R meter action levels above.
Radiation Survey Ratemeter/Scaler with External Detector(s)					**Detector** **Action Level** **Action**
Noise Dosimeter (Sound Level Meter)					≤85 decibels as measured using the A-weighed network (dBa): Use hearing protection if exposure will be sustained throughout work shift. >85 dBA: Use hearing protection. >120 dBA: Leave area and consult with safety personnel.
Other:					

[a] Unless stated otherwise, airborne contaminant concentrations are measured as a time-weighted average in the worker's breathing zone. Acceptable concentrations for known airborne contaminants will be determined based on OSHA/NIOSH/ACGIH and/or NRC exposure limits. As a guideline, ½ the PEL/REL/TLV, whichever is lower should be used.

9. DECONTAMINATION PROCEDURES

All equipment, materials, and personnel will be evaluated for contamination upon leaving the exclusion area. Equipment and materials will be decontaminated and/or disposed and personnel will be decontaminated, as necessary. Decontamination will be performed in the contamination reduction area or any designated area such that the exposure of uncontaminated employees, equipment, and materials will be minimized. Specific procedures are described below.

Equipment/Material Decontamination Procedures (specified by work plan): Nondedicated equipment will be cleaned with paper towels, distilled water and soap. Dedicated equipment will be disposed of per client's instructions.

Ventilation: All decontamination procedures will be conducted in a well-ventilated area.

Personnel Decontamination Procedures: Disposable PPE to be collected in plastic bags and sealed.

PPE Requirements for Personnel Performing Decontamination: NA. Each worker doing own decontamination

Personnel Decontamination in General: Following appropriate decontamination procedures, all field personnel will wash their hands and face with soap and potable water. Personnel should shower at the end of each work shift.

Disposition of Disposable PPE: Disposable PPE must be rendered unusable and disposed as indicated in the work plan.

Disposition of Decontamination Wastes (e.g., dry wastes, decontamination fluids, etc.): As designated by client.

10. EMERGENCY RESPONSE

This section contains additional information pertaining to on-site emergency response and does not duplicate pertinent emergency response information contained in earlier sections of this plan (e.g., site layout, monitoring equipment, etc.). Emergency response procedures will be rehearsed regularly, as applicable, during project activities.

10.1 EMERGENCY RESPONSIBILITIES

All Personnel: All personnel shall be alert to the possibility of an on-site emergency; report potential or actual emergency situations to the team leader and SSO; and notify appropriate emergency resources, as necessary.

Team Leader: The team leader will determine the emergency actions to be performed by E & E personnel and will direct these actions. The team leader also will ensure that applicable incidents are reported to appropriate E & E and client project personnel and government agencies.

SSO: The SSO will recommend health/safety and protective measures appropriate to the emergency.

Other:

10.2 LOCAL AND SITE RESOURCES (including phone numbers)

Ambulance: 911

Hospital: Mercy Regional Medical Center 1515 Logan Ave. Laredo, TX 78040 (956) 718-6222

Directions to Hospital (map attached at the end of this plan): From airport, go left on W. Aurora St., ¼ mile left on San Fancisco, ¼ mile right on W. Calton Rd., ¼ mile left on Santa Ursula Ave., bear left onto I-35 S, 2 ½ miles left onto US 83 S, ¾ mile left onto Logan Ave., hospital ¼ mile on right.

Poison Control: National Center 1-800-441-0040

Police Department: 911

Fire Department: 911

Client Contact:

Site Contact:

On-Site Telephone Number: Not available

Cellular Telephone Number: 214-793-4884

Radios Available: To be taken to site as needed.

Other:

10.3 E & E EMERGENCY CONTACTS

E & E Emergency Response Center (24 Hours):	716/684-8940
Corporate Health and Safety Director, Dr. Paul Jonmaire:	716/684-8060 (office) 716/655-1260 (home)
Assistant Corporate Safety Officer, Tom Siener:	716/684-8060 (office) 716/662-4740 (home)
Regional Safety Officer: Frances Verhalen	214/245-1000 (office) 214/319-9985 (home)
Other: Kevin Jaynes	214/763-3276 (home)

10.4 OTHER EMERGENCY RESPONSE PROCEDURES

On-Site Evacuation Signal/Alarm (must be audible and perceptible above ambient noise and light levels): Verbal or car horn

On-Site Assembly Area: Main gate. Several gates at site. Gates assembly areas to be determined/designated in the field.

Emergency Egress Route to Get Off Site: Any direction; if in fenced area, go through gate nearest work area. Exact routes to be determined in the field

Off-Site Assembly Area: Corner of McPherson and Hillside Dr. (to be verified after initial site visit)

Preferred Means of Reporting Emergencies: On site: verbal; off site: telephone

Site Security and Control: In an emergency situation, personnel will attempt to secure the affected area and control site access.

Spill Control: Appropriate absorbent will be kept on site for use as a sorbent material for liquid spills.

Emergency Decontamination Procedures: Put equipment on plastic sheeting in decontamination area. Do as much personal decontamination as possible. Finish decontamination later if needed to discontinue to address life-threatening emergency.

PPE: Personnel will don appropriate PPE when responding to an emergency situation. The SSO and Section 7 of this plan will provide guidance regarding appropriate PPE.

Emergency Equipment: Appropriate emergency equipment is listed on pages 19-22. Adequate supplies of this equipment shall be maintained in the support area or other approved work location.

Incident Reporting Procedures: Injuries, exposures: Follow usual E & E procedures. Notify RSO or H/S Coordinator in office. Call Buffalo (Mary Wdowka) in Personnel during business hours immediately; also contact H/S there. Non-business hours: Call Buffalo's 24-hour telephone number immediately. Both procedures will notify Buffalo H/S. Complete Form IR as soon as possible. Vehicle incidents: follow procedures attached to safety plan.

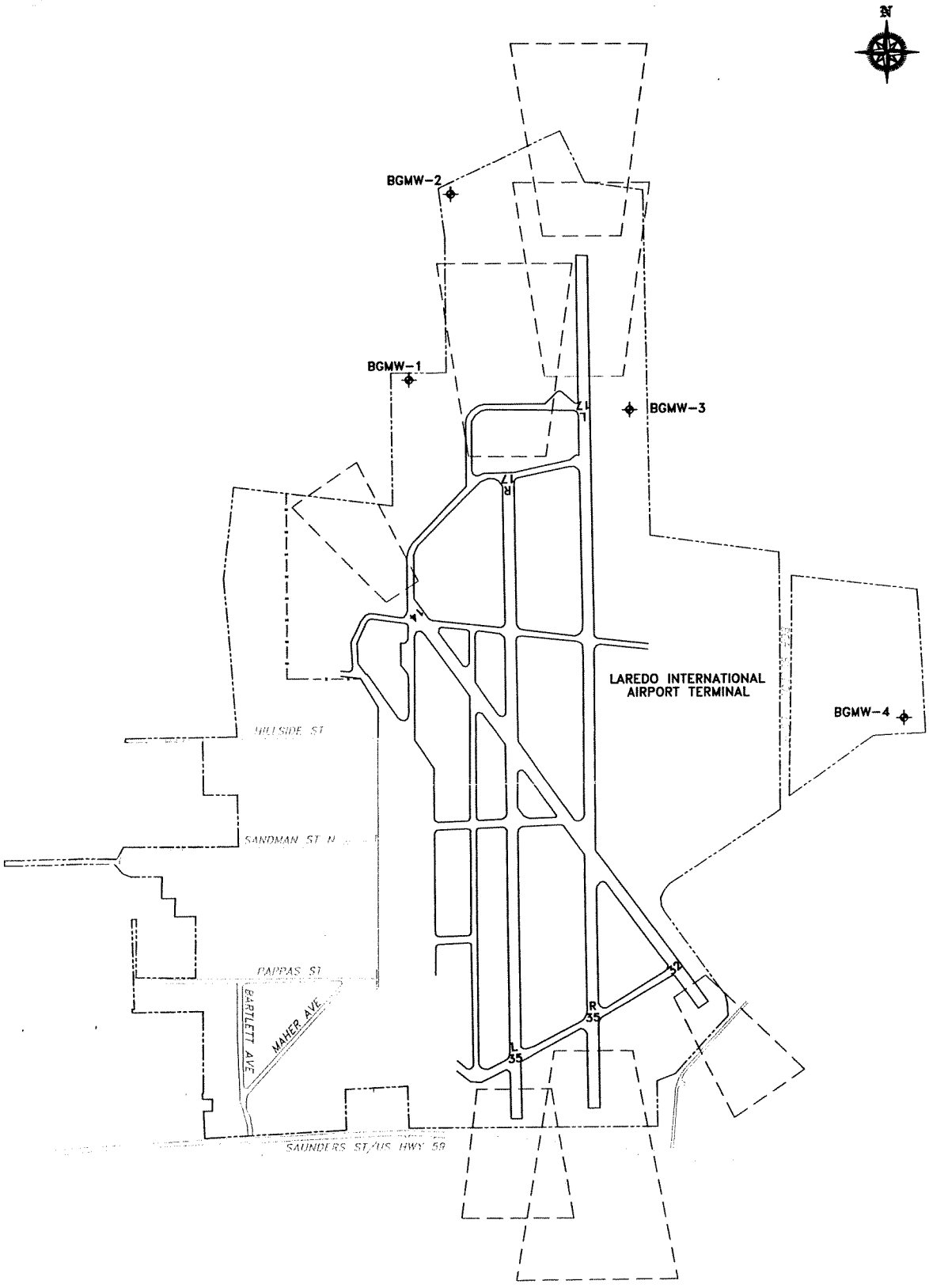
EQUIPMENT CHECKLIST REGION 6			
PROTECTIVE GEAR			
Level A	No.	Level B	No.
SCBA		SCBA	
SPARE AIR TANKS		SPARE AIR TANKS	
ENCAPSULATING SUIT (Type _____)		PROTECTIVE COVERALL (Type _____)	
SURGICAL GLOVES		BUTYL APRON	
SAFETY BOOTS (_NEOPRENE,_LEATHER)		SURGICAL GLOVES (Type _____)	
BOOTIES (Type _____)		OUTER GLOVES (Type _____)	
GLOVES (Type _____)		OUTER WORK GLOVES (Type _____)	
OUTER WORK GLOVES		SAFETY BOOTS (___NEOPRENE; ___LEATHER)	
HARD HAT		BOOTIES (Type _____)	
CASCADE SYSTEM		HARD HAT (___FACE SHIELD; ___LINER)	
5-MINUTE ESCAPE MASK		CASCADE SYSTEM; ___MANIFOLD SYSTEM	
COOLING VEST		SCBA/AIR-LINE SYSTEM	
		HIP AIR SYSTEM	
Level C		COTTON CLOTHING	
ULTRA-TWIN RESPIRATOR		COOLING VEST	
POWER AIR PURIFYING RESPIRATOR		HEARING PROTECTION	
CARTRIDGES (Type GMC-H/P-100)	X		
5-MINUTE ESCAPE MASK		Level D	
PROTECTIVE COVERALL (Type: Tyvek)	X	ULTRA-TWIN RESPIRATOR (Available)	X
RAIN SUIT		CARTRIDGES (Type GMC-H)	X
BUTYL APRON		5-MINUTE ESCAPE MASK (Available)	
SURGICAL GLOVES (Type nitrile)	X	PROTECTIVE COVERALL (Type Tyvek)	
OUTER GLOVES (Type nitrile)		RAIN SUIT	X
OUTER WORK GLOVES (Type _____)		SAFETY BOOTS (_X_NEOPRENE; _X_LEATHER)	X
SAFETY BOOTS (_X_NEOPRENE; _X_LEATHER)	X	BOOTIES (Type Latex)	
HARD HAT (___FACE SHIELD; ___LINER)	X	WORK GLOVES (Type heavy work)	X
BOOTIES (Type)		SURGICAL GLOVES (Type latex)	X
COTTON CLOTHING	X	HARD HAT (___FACE SHIELD; ___LINER)	X
COOLING VEST		SAFETY GLASSES	X
LIFE JACKET/VEST		COTTON CLOTHING	X
INSULATED: (___COVERALLS; ___BOOTS)		JACKET (___LIGHT; _ _HEAVY)	
HEARING PROTECTION	X	LIFE JACKET/VEST	
		INSULATED; (___COVERALLS; ___BOOTS)	
		HEARING PROTECTION (available)	X

INSTRUMENTATION	No.
OVA WITH CALIBRATION GAS	
HNu (Probe _____) WITH CAL. GAS	
TVA	
PASSPORT	
O_2/EXPLOSIMETER WITH CAL. KIT	
MONITOX: ___PHOSGENE; ___HCN; ___H_2S	
DRAEGER PUMP, TUBES _____	
ACCURO PUMP, TUBES _____	
RAM-1 / DATA RAM	
MINIRAM / PDR	
AIM	
XRF	
HAZARD CATEGORIZATION KIT, WITH GAS	
PHOTOVAC	
MIRAN	
INFRARED PYROMETER	
GROUND PENETRATING RADAR	
MAGNETOMETER	
MAGNETIC CABLE LOCATOR	
GROUND CONDUCTIVITY METER	
TERRAIN RESISTIVITY METER WITH ACC's	
___EM-31 __EM-34	
SEISMOGRAPH	
WEATHER STATION; ___DATALOGGER	
NOISE MONITOR	
HEAT STRESS MONITOR	
WIND GAUGE, HAND HELD	
pH: ___PAPER; ___PEN; ___METER	
CONDUCTIVITY PROBE /PEN	
HORIBA U-10 WATER QUALITY TESTER	
DISSOLVED OXYGEN METER	
RELATIVE HUMIDITY: ___PROBE; ___PEN	
H_2S TEST STRIPS	
SPILFYTER TEST STRIPS	
ANEMOMETER	
SLING PSYCHROMETER	
INTERSCAN ___SO_2 ___H_2S	
LIGHT METER	
CHLOR-N-OIL KIT	
CHLOR-N-SOIL KIT	
BATTERIES FOR EQUIPMENT	X

INSTRUMENTATION (Continued)	No.
CHARGERS FOR EQUIPMENT	
EQUIPMENT LOG BOOKS	
TOTAL STATION	
MOBILE DRILL RIG B-24	
JEROME MERCURY METER	
STX-70: (Types: _____)	
PID	X
RADIATION EQUIPMENT	
TLD BADGE	X
RADIATION ALERT MONITOR 4 (RAM-4)	
POCKET DOSIMETERS WITH CHARGER	
DOCUMENTATION FORMS	
PORTABLE RATEMETER:	
Bicron Surveyor M with Probes	
Victoreen Model Thyac III	
MICRO R METER:	
LUDLUM Model 19	
SCALER/RATEMETER:	
NaI PROBE	
ZnS PROBE	
GM PANCAKE PROBE	
GM SIDE WINDOW PROBE	
ION CHAMBER	
Victoreen 450P	
FIRST AID EQUIPMENT	
FIRST AID KIT	X
PORTABLE EYE WASH	
BLOOD PRESSURE MONITOR	
PORTABLE SHOWER	
FIRE EXTINGUISHER	
FIRE BLANKET	

SAMPLING EQUIPMENT	No.	SAMPLING EQUIPMENT (Continued)	No.
GLASS BOTTLES (Sizes Amber)	X	PERSONAL AIR SAMPLING PUMPS WITH CALIBRATOR, CHARGERS, SUPPLIES	
GLASS BOTTLES, WIDE MOUTH (_____)			
VOA BOTTLES (40 ml)		BIOS AIR-FLOW CALIBRATOR	
POLYETHYLENE BOTTLES (Size _____)	X	GENERATOR(S), PORTABLE	
SAMPLE PRESERVATIVES: ___ HNO_3, ___ NaOH, ___ H_2S0_4 ___ HCL		HIGH-VOLUME AIR SAMPLERS WITH CALIBRATORS: ___ PM-10; ___ PS-1; ___ PM-2.5	
HAND BAILERS	X	HIGH-FLOW AIR SAMPLER	
STRING AND/OR ROPE	X	ANALYTICAL BALANCE	
WATER LEVEL INDICATOR	X	BOAT	
WELL PUMP			
CAT HEAD/MOTOR/PULLEY			
SAMPLING PLATFORM (BOAT)			
KEMMERER WATER SAMPLER			
ECKMAN DREDGE			
TROWELS AND/OR SPOONS	X	**DECON EQUIPMENT**	
SHOVEL AND/OR POST HOLE DIGGER		WASH TUBS	
MIXING PLATES AND/OR BOWLS		BUCKETS	
SIEVES		SCRUB BRUSHES	
HAND AUGER (Size _____)		PRESSURIZED SPRAYER	
SLEEVES/CAPS/EXTENSIONS		DETERGENT (Type Alconox or commercial detergent)	X
SPLIT SPOON SAMPLER		SOLVENT (Type _____)	
GEOPROBE		DISTILLED WATER	X
MINUTEMAN DRILL WITH ACCESSORIES		DEIONIZED WATER	
SOIL GAS TILE PROBE WITH ACCESSORIES		DISPOSABLE FACEPIECE SANITIZER WIPES	X
SLAM BAR (Size _____)		FACE MASK SANITIZER POWDER	X
EXTENSIONS/SLEEVES/CAPS		WIRE BRUSH	
JMC SOIL PROBE		SPRAY BOTTLE	X
KNIVES AND/OR SCISSORS	X	BANNER/BARRIER TAPE	
BANNER TAPE		PLASTIC SHEETING	X
PLASTIC SHEETING	X	TARPS AND POLES	
SURVEYING: ___FLAGS; ___TAPE		TRASH BAGS	X
AUTO LEVEL; ___TRIPOD		TRASH CANS	
FIELD TRANSIT; ___TRIPOD		MASKING TAPE	X
BRUNTON COMPASS; ___ROD, 25 FT.		DUCT TAPE	X
CHAIN OR TAPE (200 FT.) AND/OR ROLATAPE		PAPER TOWELS	X
HAND HELD GPS (_____)	X	FOLDING CHAIRS	
NON-SPARKING TOOLS		STEP LADDERS	
THIEVING RODS WITH BULBS		5-GALLON WATER JUGS	
COLIWASA SAMPLERS WITH TUBES		TABLES	
SLUDGE JUDGE		STEAM CLEANER WITH GENERATOR	
SPRAY PAINT/MARKER	X		

VAN EQUIPMENT		MISCELLANEOUS	No.
TOOL KIT	X	35-mm CAMERA, FILM, BATTERIES	X
HYDRAULIC JACK		VIDEO CAMERA, TAPE, BATTERIES	
LUG WRENCH		LOG BOOK (___LARGE, ___SMALL)	X
TOW CHAIN		LOG BOOK STAMPS	
VAN CHECK OUT		CHEMICAL REFERENCES	
Gas		MSDS'S REQUIRED	
Oil		QASP IF REQUIRED	
Antifreeze		SITE SAFETY PLAN	X
Battery		BINOCULARS	
Windshield Wash		MEGAPHONE; __AIR HORN; __BULL HORN	X
Tire Pressure		INTRINSICALLY SAFE FLASHLIGHTS	
Lights		WITH BATTERIES	
WELLS CARGO TRAILER		WEATHER RADIO	
		2-WAY RADIOS WITH CHARGERS	X
		CELLULAR TELEPHONE WITH CHARGER	X
SHIPPING EQUIPMENT		SATELLITE TELEPHONE	
COOLERS	X	COMPUTER WITH PRINTER, BATTERIES	
PAINT CANS WITH LIDS, 7 CLIPS EACH SIZE _____		COMPUTER DISKS, PAPER, CORDS	
		MODEM; ___TELECOUPLER	
VERMICULITE		EXTENSION CORDS; ___OUTLET STRIP	
BUBBLE WRAP	X	REGULAR HAND TOOLS	
FEDERAL EXPRESS AIRBILLS	X	BOLT CUTTERS; ___KEYS, _ PADLOCKS	X
ADDRESS LABELS	X	MAPS	X
DOT LABELS: "DANGER"		COMPASS	
"THIS SIDE UP"		TAPE MEASURE	X
"INSIDE CONTAINER COMPLIES..."		PERMANENT MARKERS; PENS, PENCILS	X
OTHER HAZARD GROUP(S)		SCISSORS; ___KNIVES	X
BAGGIES; GARBAGE BAGS	X	WATER COOLER; ___STRESS DRINKS	X
CUSTODY SEALS	X	APR FOG PRUF, NOSECUP (Cold Weather)	
CHAIN-OF-CUSTODY FORMS	X	SNAKE LEGGINGS	
SAMPLE TAGS	X	WADERS (___HIP, ___CHEST)	
SAMPLE LABELS	X	INSECT REPELLENT	X
CLP FORMS/STICKERS		SUNSCREEN	X
CLEAR PACKING TAPE	X	HEARING PROTECTION	X
STRAPPING TAPE	X	FALL PROTECTION SAFETY BELT W/LANYARD	
DUCT TAPE	X	SAFETY PLAN	X
PERMANENT MARKERS	X		
SHARPIE PENS	X		
BALLPOINT PENS	X		



600 0 600 1200
SCALE IN FEET

LEGEND	
—·—·—·—	SECURITY FENCE
————	PROPERTY BOUNDARY OF LAREDO INTERNATIONAL AIRPORT
— — — —	AVIGATION EASEMENT (RESTRICTED VERTICAL CONSTRUCTION)
⌖	APPROXIMATE MONITORING WELL AND SOIL SAMPLE LOCATION

ecology and environment, inc.
Dallas, Texas
International Specialists in the Environment

FIGURE 5-1
PROPOSED LOCATIONS OF BACKGROUND MONITORING WELLS AND SOIL SAMPLES
FORMER LAREDO AFB
LAREDO, WEBB COUNTY, TEXAS

JOB#: 001289.UX06	Date: OCTOBER 2001
File Name: 001289UX06H.DWG	P.M.: D. SMITH

HEAT STRESS PREVENTION AND TREATMENT

Elevated temperatures are potentially hazardous, especially when work is conducted without appropriate precautions. The following sections describe heat stress prevention and the recognition and treatment of heat emergencies.

Effects of Heat

A predictable amount of heat is generated as a result of normal oxidation processes within the body. If heat is liberated rapidly, the body cools to a point at which the production of heat is accelerated, and the excess heat brings the body temperature back to normal.

Interference with the elimination of heat leads to its accumulation and to the elevation of body temperature. This condition produces a vicious cycle in which certain body processes accelerate and generate additional heat. Afterward, the body must eliminate not only the heat that is normally generated but also the additional quantities of heat.

Most body heat is brought to the surface by the bloodstream and escapes to cooler surroundings by conduction and radiation. If moving air or a breeze strikes the body, additional heat is lost by convection. When the temperature of the surrounding air becomes equal to or rises above the body temperature, all the heat must be lost by vaporization of the moisture or sweat from skin surfaces. As the air becomes more humid (contains more moisture), vaporization from the skin decreases. Weather conditions including high temperatures (90 to 100 degrees F), high humidity, and little or no breeze cause the retention of body heat. Such conditions or a succession of such days (a heat wave) increase the chances of a medical emergency due to heat.

Preventing Emergencies Due to Heat

When working in situations where the ambient temperatures and humidity are high, and especially in situations where protection levels A, B, or C are required, the site safety officer should:

- Ensure that all employees drink plenty of fluids (Gatorade or its equivalent);
- Ensure that frequent breaks are scheduled so overheating does not occur; and
- Revise work schedules, when necessary, to take advantage of the cooler parts of the day (i.e., 5:00 a.m. to 11:00 a.m. and 6:00 p.m. to nightfall).

When protective clothing is required, the suggested guidelines correlating ambient temperature and maximum wearing time per excursion are:

Ambient Temperature	Maximum Wearing Time per Excursion
Above 90 degrees F	15 minutes
85 to 90 degrees F	30 minutes
80 to 85 degrees F	60 minutes
70 to 80 degrees F	90 minutes

60 to 70 degrees F	120 minutes
50 to 60 degrees F	180 minutes

One method of measuring the effectiveness of an employee's rest-recovery regime is by monitoring the heart rate. The "Brouha guideline" is one such method and is performed as follows:

- Count the pulse rate for the **last** 30 seconds of the first minute of a 3-minute period, the **last** 30 seconds of the second minute, and the **last** 30 seconds of the third minute; and
- Double each result to yield beats per minute.

If the recovery pulse rate during the last 30 seconds of the first minute is 110 beats/minute or less, and the deceleration between the first, second, and third minutes is **at least** 10 beats/minute, then the work-recovery regime is acceptable. If the employee's rate is above the rate specified, a longer rest period will be required, accompanied by an increased intake of fluids.

Heat Emergencies

Heat Cramps. Heat cramps usually affect people who work in hot environments and perspire a great deal. Loss of salt from the body causes very painful cramps in leg and abdominal muscles. Heat cramps may also result from drinking iced water or other drinks either too quickly or in too large a quantity. The symptoms of heat cramps are:

- Painful muscle cramps in legs and abdomen;
- Faintness; and
- Profuse perspiration.

To provide emergency care for heat cramps, move the patient to a cool place. Give him or her sips of liquids such as Gatorade or its equivalent. Apply manual pressure to the cramped muscle. Move the patient to a hospital if there is any indication of a more serious problem.

Heat Exhaustion. Heat exhaustion also may occur in individuals working in hot environments and may be associated with heat cramps. Heat exhaustion is caused by the pooling of blood in the vessels of the skin. The heat is transported from the interior of the body to the surface by the blood. The skin vessels become dilated and a large amount of blood is pooled in the skin. This condition, plus the blood that is pooled in the lower extremities when in an upright position, may lead to an inadequate return of blood to the heart and eventual physical collapse. The symptoms of heat exhaustion are:

- Weak pulse;
- Rapid and usually shallow breathing;
- Generalized weakness;

- Pale, clammy skin;
- Profuse perspiration;
- Dizziness/faintness; and
- Unconsciousness.

To provide emergency care for heat exhaustion, move the patient to a cool place and remove as much clothing as possible. Have the patient drink cool water, Gatorade, or its equivalent. If possible, fan the patient continually to remove heat by convection, but do not allow chilling or overcooling. Treat the patient for shock and move him or her to a medical facility if there is any indication of a more serious problem.

Heat Stroke. Heat stroke is a profound disturbance of the heat-regulating mechanism and is associated with high fever and collapse. It is a serious threat to life and carries a 20% mortality rate. Sometimes this condition results in convulsions, unconsciousness, and even death. Direct exposure to sun, poor air circulation, poor physical condition, and advanced age (over 40) increase the chance of heat stroke. Alcoholics are extremely susceptible. The symptoms of heat stroke are:

- Sudden onset;
- Dry, hot, and flushed skin;
- Dilated pupils;
- Early loss of consciousness;
- Full and fast pulse;
- Deep breathing at first, followed by shallow or faint breathing;
- Muscle twitching, growing into convulsions; and
- Body temperature reaching 105 to 106 degrees F or higher.

When providing emergency care for heat stroke, remember that it is a life-threatening emergency. Transportation to a medical facility should not be delayed. Move the patient to a cool environment, if possible, and remove as much clothing as possible. Ensure an open airway. Reduce body temperature promptly by dousing the body with water or, preferably, by wrapping the patient in a wet sheet. If cold packs are available, place them under the arms, around the neck, at the ankles, or any place where blood vessels that lie close to the skin can be cooled. Protect the patient from injury during convulsions.

COLD STRESS PREVENTION AND TREATMENT

Cold temperatures are potentially hazardous, especially when work is conducted without appropriate precautions. The following sections describe cold stress prevention and the recognition and treatment of cold stress emergencies.

Preventing Emergencies Due to Cold Stress

When working in situations where the ambient temperature is low, especially if low temperatures are accompanied by windy conditions, personnel should use the following cold-stress prevention measures:

- Wear warm, dry, loose-fitting clothing that is preferably worn in layers. Outer clothing should be waterproof and windproof. Inner clothing should be capable of retaining warmth even when it is wet (e.g., wool or polypropylene) or have wicking capabilities (to draw moisture and perspiration away from the skin).
- Wear lined and insulated footwear and warm gloves or mittens.
- Alternately remove and don clothing layers as necessary to regulate body temperature and reduce excess perspiration.
- Drink warm fluids as often as desired.
- Take frequent breaks to provide for cold stress monitoring.

Cold Stress Emergencies

Hypothermia. Exposure to cold can cause the body's internal temperature to drop to a dangerously low level. Hypothermia occurs when a person's body loses heat faster than it can be produced. The body's normal deep-body temperature is approximately 98.6 degrees Fahrenheit. If body temperature drops to 95 degrees Fahrenheit, uncontrollable shivering may occur. If cooling continues, these other symptoms may occur:

- Vague, slow, slurred speech;
- Forgetfulness, memory lapses;
- Inability to use hands;
- Frequent stumbling;
- Drowsiness;
- Exhaustion, collapse;
- Unconsciousness; and
- Death.

Hypothermia impairs the judgment of the victim. Hypothermia is possible even in temperatures above freezing and can be prevented by remaining warm and dry and avoiding overexposure to the cold.

If a person shows symptoms of hypothermia, perform the following:

- Remove the victim from exposure to wet and cold weather.
- Remove wet clothing.
- If the victim is only mildly affected, provide warm drinks and dry clothing.
- If the victim is more seriously affected (clumsy, confused, unable to shiver), begin safe-warming procedures such as hugging, wrapping in dry blankets, and the use of warm objects such as hot water bottles or heat packs, and arrange for evacuation. Do not give the victim warm drinks until he or she exhibits a clear level of consciousness and appears to be warming up.

Frostbite. Frostbite occurs when body tissue freezes. Severe frostbite can lead to reduced circulation and the possible need for amputation. To prevent frostbite, maintain good circulation and keep extremities warm and dry. In extreme cold, it is important to prevent heat loss from as many areas of the body as possible. Exposed limbs and the head are major areas of heat loss.

Tall, thin people; those in poor physical condition; people with chronic diseases; heavy smokers; children; the elderly; and those who have been drinking alcohol are more susceptible to frostbite than other people due to poor circulation, poor production of body heat, or both.

There may be no pain or numbness experienced with gradual freezing of body tissues. While in the cold, it is important to test extremities for sensation and ensure that clothing is loose-fitting and warm. Exposed parts of the body should be inspected routinely. Just before freezing, skin becomes bright red. As freezing continues, small white patches will appear and the skin will become less elastic, often remaining pitted after it is touched or squeezed.

Serious freezing is most common in the feet because people are less aware of them, circulation and sensation are poorer, and warm footwear is difficult to obtain. Hands are usually the next to freeze. Exposed parts of the head will freeze less rapidly because they are conditioned to exposure and have a better blood supply.

In very cold weather, avoid touching cold metal with bare body parts. In the event that this happens, release the skin gently using heat, warm water, or urine. Avoid handling gasoline, kerosene, or similar liquids which, when handled in cold weather, can cause immediate frostbite.

If a person shows symptoms of frostbite, consult a medical professional, if possible, and perform the following:

- Initiate rewarming only if subsequent refreezing is not a possibility (thawing and refreezing should always be avoided because this is very injurious to tissue). Rewarm body parts in water that is approximately 100 to 105 degrees Fahrenheit. Do not try to thaw the body parts using cold water, snow, or

intense heat from fires or stoves. The whole body may be immersed in warm water if necessary.

- If a large portion of an extremity is frozen when rewarming is initiated, the deep body temperature may drop as cooled blood begins to circulate throughout the body. Provide warm liquids to alleviate this situation.

- Move the afflicted part gently and voluntarily during rewarming.

- Use pain medication if it is available. Rewarming can be acutely painful. After thawing is completed, a deep pain may persist for several days, depending on the severity of the frostbite. Pain may be a good sign as it indicates that nerve function is present.

- A dull purple color, swelling, or blisters indicate serious injury and the need for medical attention. Consult a medical professional.

VEHICLE ACCIDENTS, PROPERTY LOSS, THEFT, ETC.

When an accident does occur, it is very important to notify HQ and Regional Office as soon as possible! We have designed a form that covers all the required information for the company and our insurance carrier (see attached). The importance of timely notification of detailed information to HQ cannot be stressed enough.

Call/fax the form to HQ as soon as possible with accident information. Include how and when we can reach you with further instructions or questions.

Here are some pertinent steps that should be followed:

INJURIES
-Notify E & E Personnel Department of Emergency Response Center

RENTAL CAR
- Call police if accident involves another vehicle or injuries
- Exchange license and registration information
- Call car rental agency and inform them of the accident. Give them HQ address, phone number and Administrative Services Department (716/684-8060, Ext. 2802) for future contact
- If car is not drivable, inform rental agency to provide towing
- Complete rental agency's accident report form and send copy to HQ along with copy of rental agreement (both sides).

CORPORATE VEHICLE
- Call police if accident involves another vehicle or injuries
- Exchange license and registration information
- Notify Headquarters or Region to receive instructions
- Complete E & E's "Notice of Loss" report and sent to Headquarters/Region
- If appropriate, send at least three estimates and P.R., etc. to Headquarters/Region

GOVERNMENT EQUIPMENT
The local police need to be summoned regardless of the extent of damage.

All capital equipment items that are missing, stolen, or damaged need to be reported to the ZPMO so the appropriate documentation (see attached Report of Theft/Loss, etc.) and reporting to EPA can be conducted. Timely reporting will also facilitate replacement or repair to the item.

Attached to this form should be any police reports, photographs, estimates for replacement or repair and logbook entries related to the condition of the equipment. If appropriate, include PRs and 7-point justification documents for quick replacement. Equipment that is missing, stolen or damaged should be reported to the ZPMO within 5 working days of the incident.

PERSONAL VEHICLE
Personal vehicles used for work are not insured by the company. Though the company pays for mileage, coverage for damage to your car, personal injury or other involved property is your own responsibility. Moving violations and traffic tickets received while on the job are entirely the responsibility of the employee.

** Remember - if you are involved in an accident, give only the facts, do not admit fault.

ECOLOGY AND ENVIRONMENT, INC.
368 Pleasantview Drive
Lancaster, NY 14086
(716)684-8060/(716)684-0844

NOTICE OF LOSS REPORT
Call/Fax to HQ ASAP

FX _____
Claim Number _____

TYPE OF LOSS
☐ Automobile ☐ Other Liability ☐ Fire ☐ Theft

OWNERSHIP OF VEHICLE
☐ Rental ☐ Government ☐ E & E

Date and Time of Loss: _____

Police or Fire Department to whom reported: _____

Location of Accident or Loss: _____

Road Conditions: _____

Description: _____

Vehicle Information: _____
(Year, Make, Model, License Plate #, VIN #, Rental Agreement #)

Driver Information: _____
(Name, Address, License #, Date of Birth)

Other Vehicle and Driver Information: _____
(Year, Make, Model, License Plate #, Name, Address, License #, Phone, Alternate Phone #)

Damage to your vehicle: _____

Damage to other vehicle: _____

Other vehicle or property insured? _____ If yes, Name of Insurance Company, Address and

Policy Number: _____

Injured: _____
(Name, Address, Age, Extent of Injuries, Hospital)

Witnesses: _____
(Name, Address, Phone Numbers)

Additional Information or Remarks: _____

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*Please attach the following information: Police Report, Photographs, Copy of Rental Agreement (both sides), and Rental Accident Report. If applicable, send Estimates, Repair Bills, PR, etc. as soon as possible.

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TAT - Zone II

REPORT OF THEFT/LOSS/DAMAGE OF GOVERNMENT-OWNED PROPERTY

Instructions: This report is to be completed to the fullest extent possible, as soon as possible. In the event of positive discovery of theft or loss, the appropriate Regional and Zone Program Management Office program and equipment managers must be notified, immediately. The ZPMO representative will then notify the EPA-HQ Program Contract Officer and Property Administrator.

EPA Program: _____ EPA Region: _____

1. Event currently being reported (circle one):

   (a) THEFT ... Notify ZPMO by telephone, immediately, and follow up with police report and employee statement describing related details, to include summary of corrective action taken to prevent future thefts.

   (b) LOSS.... Provide ZPMO with employee statement describing related details and attempts made to locate lost items(s). This type of report should be made after exhausting hope of locating item(s).

   (c) DAMAGE .. In the event of damage beyond economical repair, provide ZPMO with statement describing how item(s) became damaged.

2. Inventory information for item(s) now being reported:

   (a) Item Description: (1) _____
   (2) _____
   (3) _____

   (b) Serial Number: (1) _____
   (2) _____
   (3) _____

   (c) EPA Decal Number: (1) _____
   (2) _____
   (3) _____

3. Details (as appropriate):

   (a) Status/location of item(s) at time of event: _____
   _____

   (b) Date/time of event: _____

   (c) Person in charge of item(s), if signed out: _____

   (d) Other individuals on job (for signed-out item(s): _____
   _____

(e) Individual who discovered event: _____

(f) Date/time of discovery of event: _____

(g) Date/time/location of last sighting of item(s): _____

(h) Person last sighting item(s): _____

(i) Other details: _____

_____

_____

_____

(j) Actions taken to locate item(s): _____

_____

(k) Authorities notified/reports filed: _____

_____

(l) Measures taken to prevent repeat of event: _____

_____

(m) Estimated cost of repair, for damaged items: _____

4. Name of individual filing report: _____

   Signature/Date: _____

5. Name of regional equipment coordinator: _____

Attachments: police report _____
log book entry _____
estimate for replacement/repair _____
other (specify)

ZPMO use:

1. Report filed with EPA-HQ:

   (a) Date: _____
   (b) To attention of: _____
   (c) Filed by/signature: _____
   (attach copy of report)

2. Other follow up action taken: _____

   _____

ecology and environment, inc.

DAILY SAFETY MEETING RECORD

GENERAL INFORMATION

Project: Former Laredo Air Force Base

Project No: 001289.UX06.01 | TDD/PAN No.:

Project Location: Laredo, Webb County, TX

Date: Time: Weather:

Specific Location:

Planned Activities:

SAFETY TOPICS PRESENTED

Chemical Hazards Update:

Physical Hazards Update:

Radiation Hazards Update:

Review of Previous Monitoring Results:

Protective Clothing/Equipment Modifications:

Special Equipment/Procedures:

Drilling Safety Issues (including testing the operation of drill rig emergency stop switches):

Emergency Procedures:

Additional Topics/Observations:

Team Members' Comments/Suggestions:

DAILY SAFETY MEETING RECORD

INITIAL PROJECT SAFETY CHECKLIST

1. Emergency information reviewed? ___ and made familiar to all team members? ___
2. Route to nearest hospital driven? ___ and its location known to all team members? __
3. Health and safety plan readily available and its location known to all team members? __
4. E & E Drilling SOP on site? ___ and available for team member review? __

ATTENDEES

Meeting shall be attended by all personnel who will be working within the exclusion area. Daily informal update meetings will be held prior to work and when site tasks and/or conditions change.

Name (Printed)	Name (Signature)	Date	Representing (Company/Agency)
Meeting Conducted By:			

ecology and environment, inc.

SITE-SPECIFIC HEALTH AND SAFETY PLAN ACCEPTANCE

Project: Former Laredo Air Force Base	
Project No.: 001289.UX06.01	TDD/PAN No.:
Project Location: Laredo, Webb Co., TX	
Project Manager: Don Smith	Project Director: Frances Verhalen

The undersigned acknowledge that they have read and understood and agree to abide by the health and safety plan.

Name (Printed)	Name (Signature)	Date

Appendix B
Field Sampling Plan

001289.UX06

Soil and Groundwater Background Study
Field Sampling Plan
Former Laredo Air Force Base
Laredo, Webb County, Texas

Contract No. DACA56-01-D-2001
Task Order No. 0004

July 2001

Prepared for:

United States Army Corps of Engineers, Tulsa District
Tulsa, OK

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List of Acronyms

ASC	Analytical Services Center
BGL	below grade level
BGMW	background monitoring well
CQC	contractor quality control
DoD	United States Department of Defense
DQOs	data quality objectives
E & E	Ecology and Environment, Inc.
FSP	field sampling plan
GPS	global positioning system
GW-Res	groundwater MSC for residential use
GWP-Ind	soil MSC for industrial use based on groundwater protection
GWP-Res	soil MSC for residential use based on groundwater protection
ID	inside diameter
IDWs	investigation-derived wastes
IWTP	industrial waste treatment plant
LAFB	Laredo Air Force Base
LIA	Laredo International Airport
mg/kg	milligrams/kilogram
MSCs	media-specific concentrations
PCBs	polychlorinated biphenyls
PID	photoionization detector
PVC	polyvinyl chloride
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RCRA	Resource Conservation and Recovery Act
RRS2	TNRCC Risk Reduction Standard No. 2
SAI-Ind	soil MSC for industrial use based on inhalation, ingestion, and dermal contact
SAI-Res	soil MSC for residential use based on inhalation, ingestion, and dermal contact
SGR	shotgun range
SLF	sanitary landfill

SOPs	standard operating procedures
SPLP	synthetic precipitation leachate procedure
SVOC	semivolatile organic compound
TAC	Texas Administrative Codes
TDS	total dissolved solids
TNRCC	Texas Natural Resource Conservation Commission
TOC	total organic carbon
TPHs	total petroleum hydrocarbons
TWDB	Texas Water Development Board
USACE	United States Army Corps of Engineers
USCS	Unified Soil Classification System
UST	underground storage tank
VOCs	volatile organic compounds

1 Project Background

This field sampling plan (FSP) was prepared by Ecology and Environment, Inc., (E & E) for the United States Army Corps of Engineers (USACE), Tulsa District, under Contract No. DACA56-01-D-2001, Task Order No. 0004. E & E was tasked to perform a soil and groundwater Background Study at the former Laredo Air Force Base (LAFB) in Laredo, Webb County, Texas, which is classified as a Formerly Used Defense Site. The specific areas of concern to be addressed at the former LAFB are the sanitary landfill (SLF), shotgun range (SGR), and industrial waste treatment plant (IWTP). The locations of the SLF, SGR, and IWTP are shown in Figure 1-1. The avigation easements are areas in which drilling activities are not permitted without specific permits from the Federal Aviation Administration.

1.1 Site History and Contaminants

The former LAFB is located in Laredo, Texas, near the Texas/Mexico border and approximately 150 miles south-southwest of San Antonio. The former LAFB airfield facilities are operated as the Laredo International Airport (LIA).

Site investigation reports were generated for the SLF, SGR, and IWTP in 2000 (USACE, 2000b; USACE, 2000c; USACE, 2000a). The site history and contaminants presented in these site investigation reports are summarized in this section.

On May 7, 1942, the United States Government acquired 2,085 acres for the construction of the Laredo Army Air Corps Base (now known as *Laredo International Airport*). The government constructed runways and numerous facilities from 1942 - 1974. The base initially was deactivated in June 1947; however, it was reactivated in 1952 during the Korean conflict. The base was again deactivated in March 1974. Approximately 309 acres of the original base area either were deeded or sold to other federal, state, and county agencies or private firms. The remainder of the base was deeded to the City of Laredo in 1974.

Sanitary Landfill

The SLF site is located approximately 0.5-mile west of the north end of the LIA. The SLF site is generally bordered by McPherson Road on the west, Hibiscus Avenue on the south, Daugherty Road on the east, and Los Ebanos Drive on the north. Land use in the vicinity of the SLF area is a combination of residential, commercial, and industrial. Residential development exists on on the SLF area. There is commercial development along McPherson Road. Industrial development is mainly north and east of the SLF area.

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Historic aerial photography of the former LAFB indicates that the SLF area west-northwest of the base was used for landfill activities beginning in the mid 1950s and apparently continuing until the early 1970s. After the base was deactivated in 1974, the SLF area apparently remained undeveloped until the early 1980s.

Residential development of the SLF area began in the early 1980s. Housing developments and an elementary school (Newman Elementary School) were constructed within the SLF area. Many homes and a portion of the elementary school apparently were constructed over the locations of the former landfill trenches. In 1988, the engineering firm of Raba-Kistner Consultants, Inc., discovered sanitary refuse in a foundation boring drilled during an investigation for the construction of an addition to the elementary school. Recent excavations in the neighborhood for swimming pools and utility connections for new homes indicated that the refuse consists of old bottles, cans, clothing, and other similar types of sanitary refuse.

Based on observations of historic aerial photography, land use was mainly agricultural and military before the current development on the properties surrounding the SLF area. A small portion of the SGR, described below, was operated in the SLF area. Land west and north of the SLF site appears to have been utilized for agricultural purposes until the late 1970s. McPherson Road, along with residential development west of McPherson Road, was visible for the first time in a 1979 aerial photograph. Land use south and east of the SLF was military. Residential development of the area immediately south of the SLF area appears to have begun in the late 1970s. Land north of the SLF area appears to have been developed for industrial use beginning in the early 1980s. Land east of the SLF area generally appears to have been unchanged or industrial since the closing of the former LAFB.

Shotgun Range

The SGR consists of two areas. The larger, main portion of the SGR area is bordered on the south by present-day Hillside Road, on the west by present-day McPherson Road, on the east by mostly vacant city-owned property, and on the north approximately by present-day Hibiscus Avenue. The smaller portion of the SGR is a rectangular area centered just west of the present-day intersection of Hemlock Drive and Poinsetta Drive. The main portion of the SGR is located immediately south of the SLF, while the smaller portion is located within the area of the SLF.

The SGR is generally flat, with a gentle west-northwestward slope. An intermittent stream/drainage ditch is located approximately 0.25-mile north of the SGR site. No other creeks, ponds, or drainage features are present. The area is developed for residential use (the Vista Hermosa and Alta Vista subdivisions). Construction is mainly single-family dwellings, although some condominiums and a church are present along Hillside Road. Commercial properties are located along McPherson Road, on the west side of the site.

The SGR was part of the original base construction and was operated as a gunnery training area. At the SGR, crews were trained in the use of the type of machine guns and turrets utilized on military aircraft, as well as small arms. Based on historic aerial photography, the original firing line was located along present-day Hillside Road, with the weapons fired northward. The firing line extended from present-day McPherson Road on the west to north of present-day Bartlett Avenue, on the east.

The use of the SGR from 1947 - 1952, while the base was operated as Laredo's municipal airport, is unknown. However, based on historic aerial photography from 1952, the SGR apparently was either unused during that time period or possibly used as a public firing range, because no obvious changes in land use are visible between the 1952 and earlier photographs.

During the 1960s, much of the SGR area was developed as a golf course. A 1973 aerial photograph indicates that the original firing line area had been incorporated into the golf course. Another smaller firing line had been constructed farther north, approximately in the present-day location of Poinsetta Drive and Hemlock Drive. The direction of fire for the new firing line was apparently north-northeast.

Based on historic aerial photography, the residential development of the south portion of the SGR began in approximately 1980, with the construction of the Vista Hermosa subdivision. Development of the Alta Vista subdivision began approximately in the mid-1980s.

Industrial Waste Treatment Plant

The parcel on which the IWTP is situated is bordered on the south by commercial property (a grocery store, a former dry cleaner, and a vacant retail/automobile service building), on the east by the current LIA fuel farm , on the north by the newly constructed Arkansas Street Extension, and along the west by a drainage ditch and Jarvis Street. The site slopes slightly west toward a ditch that drains to the southwest. Development in the vicinity of the IWTP site is generally commercial and industrial. Warehouses are located along the west side of Jarvis Street.

One small concrete building, a concrete clarifier tank, a sanitary sewer manhole structure, and a former underground storage tank (UST) are present at the IWTP site. Steel pipe is exposed where it crosses the drainage ditch northwest of the concrete building. The site is grass-covered, with a few small trees along the site boundaries and a moderate-size tree just east of the clarifier.

The concrete building is approximately 18 foot by 18 foot with a narrow platform and a concrete drive on the northeast side. The building was originally the control house for the IWTP and is now empty. No indications of piping or electrical controls are present inside the building.

The concrete clarifier is round, approximately 20 feet in diameter and approximately 8 feet in depth. The clarifier is empty, except for some soils and trash in the bottom, and is situated within a raised mound of soil approximately 5 feet high. The sanitary sewer manhole is located adjacent to the northwest clarifier tank.

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An UST rests on the ground east of the clarifier. The steel tank is approximately 6 feet or 7 feet in diameter and approximately 15 feet long. During investigation activities in 1998, no indications of holes in the tank were observed. According to Jose Flores, the airport director, the tank had no connection to the operation of the IWTP, and apparently had been dumped there by unknown people many years ago.

According to various historical maps of the LAFB, a sludge lagoon measuring approximately 40 feet by 80 feet was located approximately 120 feet southwest of the clarifier. No evidence of the lagoon's former location is visible at the site. Based on historic photographs, the lagoon appears to have been at least partially located on what is now the adjacent commercial property south of the IWTP site.

Historic maps also indicate subsurface piping associated with the IWTP. The piping included a 10-inch-diameter influent line connecting the underground storm sewer northwest of the drainage ditch to the control house, a pipe from the control house to the clarifier, a 6-inch-diameter pipe connecting the clarifier to the manhole and then to the sanitary sewer northwest of the drainage ditch, a 6-inch-diameter pipe connecting the clarifier to the sludge lagoon, and a 1.5-inch-diameter potable water line connecting the control house to the 12-inch water main northwest of the drainage ditch. No information regarding the removal of these pipes is available, and the IWTP structures remain on site. Therefore, it is presumed that at least some of the piping formerly associated with the IWTP remains on site. The small-diameter steel pipe, visibly crossing the drainage ditch at the site, is presumed to be the former potable water line.

Based on historic maps of the LAFB, the property on which the IWTP was constructed formerly had been mostly vacant. The general plan map indicates a small fenced storage yard approximately in the area just east of the present-day IWTP building and clarifier. Another fenced storage yard is indicated farther southwest, partially located in the southwest portion of the IWTP site. Information regarding the materials that may have been stored in the areas is not indicated on the historical maps.

The general plan map also indicates facilities in adjacent and nearby areas. The base fuel farm was located east and northeast of the IWTP site. Several warehouses are indicated along the west side of present-day Jarvis Avenue. A motor pool facility with USTs and motor repair facilities is indicated approximately 100 yards northwest of the IWTP site. The storm water drainage plan map indicates that the drainage ditch along the west side of the IWTP site collects surface water runoff from the south end of the flight line area.

The IWTP was constructed by the Air Force in 1969. According to historic maps of the base, the IWTP received flow from the underground storm sewer that served the south portion of the flight line area. Effluent from the IWTP was discharged to the base sanitary sewer system. The IWTP was taken out of service in 1974 when the LAFB was closed. No information regarding the influent/effluent water quality, the amounts of waste processed by the system, or sludge disposal amounts or practices is available.

1.2 Summary of Existing Site Data

Sanitary Landfill

The USACE, Tulsa District, contractor, CH2M Hill, conducted a site investigation during June and July 1997. The purpose of the investigation was to determine the presence or absence of impacted environmental media at the SLF due to activities conducted during the time that the United States Department of Defense (DoD) operated the LAFB, and to identify potential further actions (if any) for the SLF (USACE, 2000b). The site investigation included reviewing aerial photographs; conducting an electromagnetic survey of the area; and collecting and analyzing soil gas, soil, groundwater, and sediment samples. Data presented in this section were taken from *Revised Site Investigation Report, Sanitary Landfill Site, Former Laredo Air Force Base* (USACE May 2000c).

The aerial photograph review identifies sixteen former landfill trenches within the SLF area and rectangular vegetation patterns in the west portion of the site. The rectangular patterns are similar to other rectangular patterns visible west of the SLF area. This suggests that before establishment of the LAFB, the SLF area probably was utilized for agriculture (USACE, 2000b).

The soil gas survey involved analysis of gas collected from two depths at 150 sample locations across the SLF area. The soil gas samples were analyzed for methane and volatile organic compounds (VOCs). Only two samples were found to contain detectable concentrations of methane, and only one sample was found to contain a detectable concentration of a VOC, chlorobenzene. All three soil gas samples were collected from the vacant commercial area at the west end of the SLF area. However, one of the two soil gas samples for which methane was reported contained the methane at a concentration approximately equal to the lower explosive limit for methane in air. Soils in this portion of the site were found to consist of organic-rich clays, which could have accounted for the methane in the soil gas.

Soil and groundwater samples were collected and classified lithologically using the USACE Site Characterization and Analysis Penetrometer System. The soils at the SLF area range from clays to silty sand, with organic-rich clays present in the northwest portion of the area. Groundwater was encountered at depths of approximately 3.4 ft to approximately 15.2 ft. The groundwater flow gradient at the SLF area was found to slope generally toward the west.

The soil, groundwater, and sediment samples were analyzed for VOCs, semivolatile organic compounds (SVOCs), pesticides, polychlorinated biphenyls (PCBs), herbicides, and metals. Results of the analyses were compared to the Texas Natural Resource Conservation Commission (TNRCC) Risk Reduction Standard No. 2 (RRS2) media-specific concentrations (MSCs) for residential soil and groundwater (GWP-Res, SAI-Res, and GW-Res).

Except for metals constituents, only two soil samples, three groundwater samples, and one sediment sample contained a concentration of a parameter that exceeded the applicable TNRCC RRS2 MSC for residential area soils or groundwater. The soil samples contained chlordane (an herbicide) and bis(2-ethylhexyl)phthalate (a plasticizer, which is also a common laboratory contaminant). The groundwater samples contained alpha-benzene hexachloride and 2-methyl-4-chlorophenoxyacetic acid. The sediment sample contained chlordane. All of the soil and groundwater samples that exceeded RRS2 residential criteria were collected from the commercially zoned vacant area on the west side of the SLF area. The detections appeared to be isolated and showed no particular pattern of occurrence across the SLF area.

Except for aluminum, lead, and beryllium, the detected metals concentrations that exceeded the GWP-Res standards in SLF site soils occurred sporadically across the site. Although most of the aluminum, lead, and beryllium sample concentrations exceeded the respective GWP-Res standards, the concentrations were generally consistent across the site and of the same order of magnitude as the GWP-Res standards.

The detected metals concentrations that exceeded the GW-Res standards in SLF site groundwater included aluminum, arsenic, beryllium, chromium, manganese, lead, and vanadium. Barium, cadmium, and mercury also were detected in several groundwater samples at concentrations exceeding the GW-Res standards. The detected metals concentrations were generally consistent across the site and generally did not significantly exceed the GW-Res standards. Many of the detections that exceeded GW-Res standards corresponded to locations where the soil samples did not exceed the GWP-Res standards. A goal of this study is to confirm or deny this conclusion for soil.

It is noted that turbidity was a problem for many of the groundwater samples. The degree of turbidity could have affected the metals analytical results. The groundwater samples were not filtered before placement in the sample containers. For metals in sediment samples, only aluminum and lead exceeded the GWP-Res standards. However, the detected concentrations only slightly exceeded the standards and were fairly consistent. It is therefore likely that the detected metals concentrations reflect natural conditions (USACE, 2000b). Agoal of this study is to confirm or deny this conclusion for groundwater.

Shotgun Range

The USACE Tulsa District contractor, CH2M Hill, conducted site investigation activities during May and June 1999. The purpose of the investigation was to determine the presence or absence of impacted environmental media at the SGR due to activities conducted during the time that DoD operated the LAFB, and to identify potential further actions (if any) for the SGR (USACE, 2000c). The site investigation included collection of 33 soil samples. Data presented in this section were taken from *Site Investigation Report, Shotgun Range Site, Former Laredo Air Force Base* (USACE July 2000b).

The 33 soil samples were analyzed for total lead, and eight of the samples were analyzed for lead using the synthetic precipitation leachate procedure (SPLP). Results of the analyses were compared to the RRS2 MSCs for lead in soils and groundwater.

Lead was present in the soil at concentrations exceeding the GWP-Res standard. However, there was no site-specific background concentration for lead (USACE 2000c). The detected lead concentrations were generally low, with nine soil samples and one quality control (QC) sample containing more than 20 milligrams per kilogram (mg/kg) of total lead. Only one soil sample, SGRBHSO013000N1, exceeded the SAI-Res standard. The high concentration of 36,200 mg/kg in this sample possibly was a result of the inclusion of a piece of lead shot or bullet fragment within the aliquot of the sample. Samples collected from this boring at a greater depth contained an estimated 8.8 mg/kg of total lead.

Samples exhibiting the highest concentration of total lead were collected from the vicinity of the original firing line north of present-day Hillside Road. The detected levels of lead in samples collected from 2 feet and below were lower than those in samples collected from 0 feet to 0.5 feet. The detected lead concentrations in the SPLP extract for five of the eight samples analyzed, excluding the QC duplicate, were found to exceed the GW-Res standard of 0.015 milligram per liter. All of the SPLP samples were shallow samples. It was determined by the CH2M Hill investigation that significant leaching of lead into the subsurface was not taking place. This is due largely in part to findings showing a total lead concentration in the deeper samples being less than that in the shallow samples.

Industrial Waste Treatment Plant

The USACE contractor, CH2M Hill, conducted a site investigation during May 1999. The purpose of the investigation was to determine the presence or absence of impacted environmental media at the IWTP due to activities conducted during the time that the DoD operated the LAFB, and to identify potential further actions (if any) for the IWTP (USACE 2000a). The site investigation included drilling seven soil borings, installing three groundwater monitoring wells, and collecting and analyzing soil and groundwater samples. Data presented in this section were taken from *Final Site Investigation Report, Industrial Waste Treatment Plant Site, Former Laredo Air Force Base*, (USACE 2000a).

Twenty-one soil samples and three groundwater samples were collected from the IWTP area and analyzed for VOCs, SVOCs, pesticides, herbicides, PCBs, total petroleum hydrocarbons (TPH), and total Resource Conservation and Recovery Act (RCRA) metals. The soil and groundwater samples were compared to the RRS2 MSCs, and groundwater pH and total dissolved solids (TDS) were compared to state and federal drinking water standards.

Of the 21 soil samples analyzed for VOCs, 20 samples contained detectable concentrations of at least one VOC. None of the detected concentrations exceeded the GWP-Ind or SAI-Ind standards. Of the 21 samples, four samples contained detectable limits of SVOCs. Most of the detected SVOC concentrations were in samples collected from boring No. IWTPMW001 at 7 feet below grade level (BGL).

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Of the 21 samples, six contained detectable limits of pesticides, and none of the samples contained detectable limits of herbicides. Detectable limits of PCBs were measured in two of the 21 samples. Only one of the samples exceeded the GWP-Ind standard, and neither samples exceeded the SAI-Ind standard. Four samples contained detectable concentrations of TPHs as gasoline and six samples contained detectable limits exceeding the TPH as diesel.

All 21 of the soil samples contained detectable concentrations of one or more RCRA metals, including arsenic, barium, chromium, and lead. Chromium exceeded the GWP-Ind standard in six samples, cadmium exceeded the GWP-Ind standard in three samples, and barium exceeded the GWP-Ind standard in one sample. None of the samples exceeded the SAI-Ind standard. In the site investigation report, it was noted that a background study was not performed for metals and that the background concentration levels were unknown.

Of the three groundwater samples, only those collected from one of the wells contained detectable concentrations of VOCs. Two of the three groundwater samples contained detectable concentrations of SVOCs. No PCBs, herbicides or pesticides were detected in the groundwater samples. TPH was detected in only one groundwater sample.

RCRA metals were found at detectable limits in all three groundwater samples. Arsenic, mercury, selenium, and silver were not detected in any of the groundwater samples. It was noted in the report that the non detect results for arsenic and selenium for all groundwater samples were rejected in the data validation process because of low analytical spike recoveries.

During this investigation, groundwater was encountered approximately 6.5 feet to 9 feet BGL. According to water level measurements taken from the groundwater monitoring wells, the groundwater flow is south-southeast, with a gradient of approximately 0.01 foot.

1.3 Site-Specific Definition of Problems

During the site investigations conducted by the USACE, Tulsa District, contractor in 1997 and 1999, limited background samples were collected. The samples were compared to the RRS2 MSCs for residential and industrial soils and groundwater. Because of the limited collection of background samples, additional sampling will be performed to determine the extent of contamination and to establish site-specific cleanup goals.

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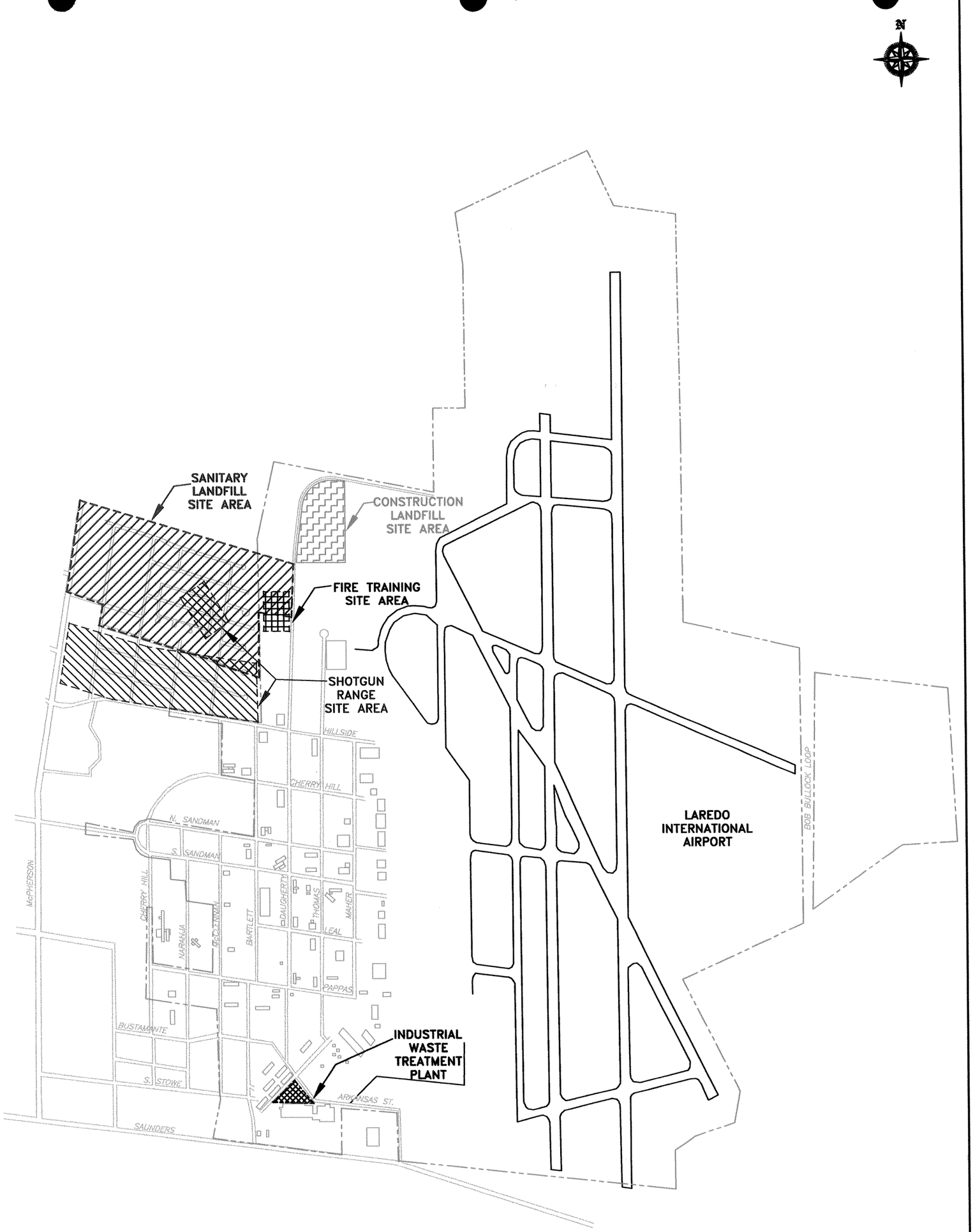

1000 0 1000

SCALE IN FEET

NOTE: SCALES ARE APPROXIMATE

LEGEND

- EXISTING BUILDING
- ROAD
- PROPERTY BOUNDARY OF LAREDO INTERNATIONAL AIRPORT
- INDUSTRIAL WASTE TREATMENT PLANT SITE AREA
- SHOTGUN RANGE SITE AREA
- SANITARY LANDFILL SITE AREA
- FIRE TRAINING SITE AREA
- CONSTRUCTION SITE SITE AREA

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Dallas, Texas
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FIGURE 1-1
LOCATIONS OF AREAS OF INTEREST
FORMER LAREDO AFB
LAREDO, WEBB COUNTY, TEXAS

JOB#: 001289.UX06	Date: OCTOBER 2001
File Name: 001289UX06G.DWG	P.M.: D. SMITH

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2 Project Organization and Responsibilities

The project planning personnel are delineated in Section 2 of the work plan. In this section, key field personnel and subcontractors are identified and their responsibilities are defined.

2.1 Field Personnel Information

David Anderson, the E & E task order manager, will be responsible for assigning the personnel required to complete the field activities associated with the Background Study. Mr. Anderson also will be the Quality Assurance (QA) chemist and will be responsible for the validation of the analytical results.

Don Smith, the E & E Project Manager, will be responsible for the coordination with E & E's Analytical Services Center (ASC). His specific responsibilities will include collection of groundwater and soil samples and adherence to standard operating procedures (SOPs) and (QA/QC) requirements. Mr. Smith will be responsible for the disposal of investigation-derived wastes (IDWs) generated from the collection of soil and groundwater samples.

Paul James will be responsible for procurement of the drilling subcontractor and the coordination and oversight of the drilling field activities. Mr. James also will oversee the development of the monitoring wells and will measure and record water quality measurements. He will be responsible for the development of the lithologic boring logs and the well development records, and for the disposal of IDWs generated from the drilling activities.

E & E field personnel will have training commensurate with the site safety and health plan and the E & E SOPs.

2.2 Subcontractor Information

Big Tex Drilling, Inc., is tentatively selected to install the monitoring wells required for the background study. David Rogers is the contact for the drilling subcontractor. Big Tex Drilling, Inc., is located in San Benito, Texas.

The E & E ASC will analyze soil and groundwater samples collected at the former LAFB. Gary Hahn is the contact. The ASC is located in Lancaster, New York.

3 Project Scope and Objectives

The extent of contamination at the former LAFB SLF was addressed in *Revised Site Investigation Report, Sanitary Landfill Site, Former Laredo Air Force Base* (USACE 2000c). Similarly, the extent of contamination at the former LAFB SGR was addressed in *Site Investigation Report, Shotgun Range Site, Former Laredo Air Force Base* (USACE 2000b). The extent of contamination at the former LAFB IWTP was addressed in *Final Site Investigation Report, Industrial Waste Treatment Plant Site, Former Laredo Air Force Base* (USACE 2000a).

The concentrations of specific metals, pesticides, and herbicides exceeded the TNRCC RRS2 MSCs at the SLF, SGR, and IWTP. Data from the previous site investigations were compared to the TNRCC Texas Risk Reduction Program protective concentration levels (PCLs), which became effective on May 1, 2000.

In these site investigations, limited background samples were collected for naturally occurring and anthropogenic-occurring analytes. In this background study, additional background data will be collected to augment the SLF, SGR, and IWTP site investigations.

The primary objectives of the Background Study include the following:

- Determining soil and groundwater background concentrations of metals, pesticides, and herbicides relevant to the SLF, SGR, and IWTP at the former LAFB;
- Recommending supplemental statistical and risk analyses if a localized "hot spot" is encountered in the background samples.
- Determining the statistically-based threshold concentrations of metals, pesticides, and herbicides necessary to define the extent of contamination;

3.1 Task Description

The specific tasks to be performed in this Background Study include the following:

- Install four shallow monitoring wells in locations upgradient of the areas of concern, based on local topography and potentiometric maps in previous reports ;
- Collect soil samples from two-foot intervals (four per well) plus two field QC for a total of 18 samples. Analyze for herbicides, chlorinated pesticides and the following metals: arsenic, beryllium, cadmium, manganese, and lead. In addition, analyze for Grain Size Distribution and TOC.

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- Collect initial groundwater samples and analyze for VOCs, SVOCs, pesticides, herbicides, TPH, and metals: arsenic, beryllium, cadmium, and lead. In addition, analyze the following water quality parameters: TSS, TDS, TOC, TKN, COD, ammonia,cyanide, and ions (chlorides, fluoride, nitrates/nitrites, phosphate, and sulfate). Field measurements will be conducted for temperature, pH, conductivity, turbidity and redox potential.

- Collect three subsequent sets of groundwater samples at one week intervals with analysis limited to pesticides, herbicides and metals. Field measurements will also be collected.

Potential locations of the monitoring wells and soil samples are shown in Figure 5-1. Specific details relating to the monitoring wells and the collection of groundwater and soil samples are provided in Section 5. Project QA/QC procedures and data quality objectives (DQOs) are described in the quality assurance project plan (QAPP).

3.2 Applicable Regulations and Standards

Because the former LAFB is actively operated as the LIA, all drilling activities will be coordinated with Airport Director, Jose Flores. Specific work permits will be obtained as warranted. Because sample collection will be performed at the LIA, standard access agreements will be generated for the USACE, Tulsa District, government technical manager. Monitoring wells will be constructed in accordance with Texas Administrative Codes (TACs; 30 TAC 338).

3.3 Project Schedule

The project schedule is summarized in the work plan. Field activities are anticipated to begin in the second week of November, 2001. Four monitoring wells will be installed and developed and soil samples collected during the first week of field activities. Following development and recharge, groundwater samples will be collected from the four monitoring wells. To collect subsequent water samples independently, a sufficient period of time must pass to allow for the wells to recharge and for groundwater to migrate past the well casings at the natural groundwater velocity. Subsequent water samples will be collected in the three weeks following monitoring well installation.

A seven-day-turnaround period will be requested for the samples collected during the first groundwater sampling event. The rapid turnaround is required to permit a reduction in the number of required analytical testing procedures, if appropriate. The remaining groundwater samples and all soil samples will be analyzed within a 28-day-turnaround period.

The draft Background Study is schedule for submission on April 8, 2002.

4 Non-Measurement Data Acquisition

From 1997 to 2000, site investigations at the former LAFB were completed for the SLF, SGR, and IWTP. The site investigation reports will be used as the basis for the Background Study investigation. The site investigation reports are as follows:

- *Revised Site Investigation Report, Sanitary Landfill Site, Former Laredo Air Force Base* (USACE 2000c);
- *Site Investigation Report, Shotgun Range Site, Former Laredo Air Force Base* (USACE 2000b); and
- *Final Site Investigation Report, Industrial Waste Treatment Plant Site, Former Laredo Air Force Base* (USACE 2000a).

Additional information regarding the background water quality may be obtained from groundwater reports published by the Texas Water Development Board (TWDB). Potential groundwater references include:

- Klemt, W.B., G.L. Duffin, and G.R. Elder, 1976, *Ground-Water Resources of the Carrizo Aquifer in the Winter Garden Area of Texas*, TWDB Report 210, two volumes; and
- McCoy, T.W., 1991, *Evaluation of the Ground-Water Resources of the Western Portion of the Winter Garden Area, Texas*, TWDB Report 334, 64 pages.

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5 Field Activities by Area of Concern

5.1 Groundwater Samples

5.1.1 Rationale and Design

This Background Study will determine statistically valid background concentrations for the shallow aquifer underlying the former LAFB. Four new monitoring wells will be constructed to collect analytical data to determine the background concentrations for the analytes of concern. These wells will be installed across the upper surface of the water table aquifer beneath the former LAFB. The new monitoring wells will be positioned upgradient of all areas of interest and in areas unaffected by the former operations of the areas of interest. A subcontractor will be obtained to drill and install four monitoring wells, to an approximate depth of 20 ft BGS. Four sampling events will be conducted to collect water quality data from the new monitoring wells. , The potential well locations are shown on Figure 5-1.

5.1.2 Monitoring Well Installation

The drilling methods for the installation of the monitoring wells are described in Section 5.1.2.1. Well construction material details are described in Section 5.1.2.2, and well installation details are described in Section 5.1.2.3.

5.1.2.1 Drilling Methods and Equipment

The well borings will be drilled with a hollow-stem-auger drilling rig. The monitoring wells will be sampled continuously. Sampling will be conducted using 2- to 3-inch-diameter, split-spoon samplers and/or core barrel samplers, depending on lithologic conditions. The project geologist will be responsible for classifying the soil samples according to the Unified Soil Classification System (USCS). The initial water level will be measured, and any staining of the soils will be noted. Because unexpected subsurface conditions may be encountered, the proposed methods may be altered in the field as directed by the project geologist. Monitoring wells will be installed in accordance with the *Standard Operating Procedure for Overburden Monitoring Well/Piezometer Installation*, provided in Attachment A.

5.1.2.2 Materials

Monitoring wells will be completed with the installation of 2-inch-diameter Schedule 40 polyvinyl chloride (PVC) solid riser piping, slotted screen, and well end caps. Specifications for each well are provided below. Sand filter packs (20/40-mesh) will be placed in the annular spaces around the screened intervals. Hydrated bentonite seals will be placed in the annular spaces on top of the sand packs. A 4-ft by 4-ft concrete well pad will be installed at each well

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location with a bolt down manhole cover. The well pads will be sloped to drain away from the wells. Four steel bollards will be placed at the respective corners of the well pad to serve as a protective barrier against vehicular traffic. Monitoring well material details are provided in Sections 5.1.2.2.1 through 5.1.2.2.5. A typical well construction diagram is shown on Figure 5-2.

5.1.2.2.1 Casing and Screen

- Riser pipe: 2-inch inside diameter (ID) PVC (Schedule 40).
- Well screen: 2-inch ID PVC (Schedule 40), machine-slotted, 0.010-inch well screen.

5.1.2.2.2 Filter Pack, Bentonite Seal, and Cement and Bentonite Grout

- Filter Pack: 20/40-mesh silica sand.
- Bentonite seal: Hydrated bentonite pellets.
- Cement/bentonite grout: None; grout is not required because of the shallow depth of the bentonite seal.

5.1.2.2.3 Surface Completion

Monitoring wells will be completed with 4-ft by 4-ft concrete at-grade pads with bolt down manholes and locking well caps installed in the concrete pad.

5.1.2.2.4 Water Source

Water for decontamination and bentonite seal hydration will be drawn from a nearby fire hydrant.

5.1.2.2.5 Delivery, Storage, and Handling of Materials

The drilling subcontractor will bring all well materials to the site immediately before use. Materials will be stored until use on subcontractor-owned trucks or trailers. Personnel will handle the materials with clean gloves. Well materials will be packaged in protective material prior to use.

5.1.2.3 Installation

5.1.2.3.1 Soil Sampling and Rock Coring During Drilling

The monitoring wells will be sampled continuously using 2- to 3-inch-diameter, split-spoon samplers and/or core barrel samplers, depending on lithologic conditions. The project geologist will be responsible for classifying the soil samples according to the USCS. The initial water level will be measured, and any staining of the soils will be noted. Because unexpected subsurface conditions may be encountered, the proposed methods may be altered in the field as directed by the project geologist. The wells will be logged in accordance with *Standard Operating Procedure for Geologic Logging*, provided in Attachment B.

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Four samples per well boring (plus field QC samples) will be collected during well installation. Table 5-1 indicates the analyses to be performed on these samples.

5.1.2.3.2 Borehole Diameter and Depth

All boreholes shall be drilled using nominally 6.75-inch-diameter, hollow-stem augers. The borehole depths are planned to not exceed 20 ft BGL.

5.1.2.3.3 Screen and Well Casing Placement

Monitoring wells will be constructed of 2-inch ID PVC (Schedule 40) casing and 2-inch ID PVC (Schedule 40), machine-slotted, 0.010-inch well screen. The well screens are planned to extend from 5 ft to 20 ft BGL. Blank casing will be placed from the tops of the well screen (5 ft BGL) to the surface.

5.1.2.3.4 Filter Pack Placement

Filter packs, consisting of 20/40-mesh silica sand, will be placed from the bottom of the borings to 2 ft above the well screens (3 ft BGL).

5.1.2.3.5 Bentonite Seal

Bentonite seals will be constructed of hydrated bentonite pellets. The Hydration water source is stated in section 5.1.2.2.4. Bentonite pellets shall be placed and subsequently hydrated from the top of the filter pack to 1 foot BGL. Bentonite hydration shall not begin until at least 0.5 foot of bentonite pellets is placed above the filter pack. After placement and hydration of the bentonite pellets, water will not be extracted from the wells for at least 12 hours to permit the proper hydration of the surface seal (bentonite pellets).

5.1.2.3.6 Cement and Bentonite Grout Placement

Because of the shallow depth of the bentonite seals (1 foot), cement and bentonite grout will not be utilized. The upper 1 foot of the boreholes will be filled with the concrete-and-gravel mixture used to construct the well pads.

5.1.2.3.7 Concrete-and-Gravel Pad Placement

Monitoring wells will be completed with 4-foot by 4-foot, concrete-and-gravel, at-grade pads with bolt down manholes. The concrete-and-gravel mixture will be placed using a continuous pour from the tops of the hydrated bentonite well seals to the final finished surface grades.

5.1.2.3.8 Protective Cover Placement

Each monitoring well will be protected with a bolt down manhole and internal locking well cap installed in the concrete-and-gravel, at-grade pad.

5.1.2.3.9 Well Identification

The background monitoring wells (BGMW) will be numbered sequentially as *BGMW-1*, *BGMW-2*, *BGMW-3*, and *BGMW-4*. Well identification shall be scribed into the concrete pad before it completes setting and shall be marked on the brass cap inside the bolt down manhole cover.

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5.1.2.3.10 Well Development

After each well is installed, it will be developed to remove fine sediments. Water will be bailed and surged from each well until a volume equal to at least three times the well volume is removed. Development water will be analyzed using hand-held pH, temperature, conductivity, and turbidity meter(s) which will be calibrated in accordance with the manufacturers specifications. Development will be considered complete after a minimum of three times the well volume is removed and stabilization of water parameters is achieved. If water removed from the well remains turbid after three well volumes is purged, bailing will be continued until visually clear water (i.e. water free of visible suspended solids) is discharged, up to a maximum of five borehole volumes. . Water level measurements will be recorded in accordance with the *Standard Operating Procedure for Measuring the Water Level and Well Depth*, provided in Attachment C, as well as purge volumes and field measurements. .

5.1.2.3.11 Well Survey

A surveying firm licensed in the State of Texas will be subcontracted to survey the locations of the four background monitoring wells at the former LAFB. The subcontractor will survey the ground surface and the top of casing of each monitoring well. For each monitoring well, the casing cap will be removed before measurement of the top of casing. Surveying will be undertaken to establish the following:

- Vertical Control: Elevations, to the nearest 0.01 ft, will be established for the ground surface for the points of interest. Elevations will be determined relative to 1929 Sea Level Datum; and

- Horizontal Control: All of the points of interest will be located to the nearest 0.1 ft. Coordinates are to be provided in the State Plane Coordinate System.

5.1.2.4 Documentation

The field documentation of the well logs, geologic logs, and well development records will be in accordance with *Standard Operating Procedure for Geotechnical Logbook/Trip Report Preparation*, provided in Attachment D.

5.1.2.4.1 Logs and Well Installation Diagrams

E & E will use the field documentation to prepare monitoring well installation diagrams detailing the soils encountered, initial and final depths to groundwater, well casing and screen types, diameters and depths, filter pack medium and placement depths, bentonite seal depths, surface completion details, and survey information. The drilling subcontractor will prepare and submit all required documentation for the monitoring wells to TNRCC. Copies of this documentation will be provided in the background study report.

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5.1.2.4.2 Development Records

A copy of the E & E development records for the new monitoring wells will be included in the appendices to the background study report. The development records will list the well name, global positioning position (GPS)-derived well location, surveyed elevation, development date, initial pre-development water depth, and final water depth. A table of groundwater characteristics during development will be included.

5.1.2.4.3 Photographs

E & E will provide photodocumentation of well drilling, installation, development, and sampling activities in the appendices of the background study report.

5.1.2.4.4 Water Level Measurement

E & E will measure the water levels in the four new monitoring wells, before initial development, after development, and before and after purging preparatory to well sampling. E & E also will measure the static water levels in any existing on-site monitoring wells at the former LAFB. E&E will coordinate with the USACE technical manager to obtain keys to existing wells which will be measured. A table of water level measurements will be included in the appendices to the background study report. SOPs for water level measurement are provided in Attachment C.

Water level measurements will be used to construct a potentiometric map of groundwater elevations. The potentiometric map will be used to confirm that background soil samples and the monitoring wells are located upgradient of the areas of concern. Potentiometric maps from the previous investigations, Figure 4-3 in the *Revised SI Report, Former LAFB Sanitary Landfill*, and Figure 4-1 in *SI Report, Former LAFB Industrial Waste Treatment Plant Site*, were utilized to establish groundwater gradient to locate the monitoring wells to be installed.

5.1.3 Field Measurement Procedures and Criteria

E & E will collect the necessary field measurements to document soil types encountered during drilling, well installation records, monitoring well development records, water table depths, and aquifer sampling. All pertinent descriptive soil information will be recorded, including soil color, grain size, sorting, compaction, plasticity, bedding, staining, calcium carbonate deposition, and odor. Descriptive aquifer information will include depth to water, pH, temperature, conductivity, visual turbidity, redox potential and odor.

5.1.4 Sampling Methods for Groundwater

Groundwater samples will be collected in accordance with *Standard Operating Procedure for Groundwater Well Sampling*, provided in Attachment E. Groundwater samples will be sent to the ASC, where they will be analyzed for the analytes listed in Table 5-1.

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Table 5-1: Analytical Methods for Soil and Groundwater Analytes of Concern,Laredo AFB

Event	Analysis	Analytes	Method
Well Installation /Soil Sampling			
	Metals		
		As, Be, Cd, Mn, Pb	SW846 3050B/6010B
		Hg	SW846 7471A
	Herbicides	All	SW846 8151A
	Chlorinated Pesticides	All	SW846 3550B/8081A
	Total Organic Carbon		SW846 9060
	Grain Size Distribution		ASTM D422
Initial Ground Water Sampling			
	Water Quality		
		TSS	SW846 160.1
		Cyanide	SW846 9010/9012
		TDS	SW846 160.2
		Ions: Chlorides, Fluoride, Nitrates/Nitrites, Phosphate, Sulfate	SW846 9056
		Total Organic Carbon	SW846 9060
		Ammonia	SW846 350
		TKN	SW846 356
		COD	SW846 410
	Total Petroleum Hydrocarbons		TX 1005
	Metals		
		As, Be, Cd, Mn, Pb	SW846 3050B/6010B
		Hg	SW846 7471A
	Volatiles	All	SW846 8260C
	Semivolatiles	All	SW846 8270B
	Herbicides	All	SW846 8151A
	Chlorinated Pesticides	All	SW846 3550B/8081A
Subsequent Ground Water Sampling	Metals		
		As, Be, Cd, Mn, Pb	SW846 3050B/6010B
		Hg	SW846 7471A
	Herbicides	All	SW846 8151A
	Chlorinated Pesticides	All	SW846 3550B/8081A

5.1.5 Sample Containers and Preservation Techniques

Groundwater samples will be handled in accordance with *Standard Operating Procedure for Sample Packaging*, provided in Attachment F. The specific sample containers and preservation times will be coordinated with the E & E ASC.

5.1.6 Field Quality Control Sampling Procedures

One QC replicate sample will be collected during each groundwater sampling event. A trip blank will be sent with all water samples that will be analyzed by United States Environmental

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Protection Agency Method SW846-8260B. In addition, a third replicate will be collected for QA purposes and submitted to a laboratoray designated by Tulsa District.

5.1.7 Decontamination Procedures

The hollow-stem-auger drilling rig, augers and split-spoon samplers, and all sampling equipment will be decontaminated before initial use and between borings and sampling. The sampling equipment will be decontaminated in accordance with *Standard Operating Procedure for Sampling Equipment Decontamination*, provided in Attachment G.

5.2 Soil Samples

5.2.1 Rationale and Design

Previous site investigations did not include collection of sufficient background data to determine the extent or target levels of soil contamination. This background study will determine the statistically valid background concentrations at the former LAFB. The sample collection effort will augment the site investigations at the SLF, SGR, and IWTP. In order to collect a sufficient number of background samples, E & E will collect 80 samples from 40 potential locations. The proposed locations for the background soil samples are shown in Figure 5-1. The samples will be collected from 0 to 0.5 foot and 0.5 to 2 feet BGL, in accordance with the *Standard Operating Procedure for Soil Sampling*, provided in Attachment H.

5.2.2 Field Procedures

5.2.2.1 Drilling Methods

All soil samples will be collected using decontaminated split spoon or core samplers in the process of drilling the four background monitoring wells. Decontamination of the sampling devices will be conducted *Standard Operation Procedure for Sampling Equipment Decontamination*, provided in Attachment G. The depths and soil characteristics of the samples will be noted in the log which will be maintained for installation of the monitoring wells.

5.2.2.4 Sampling for Physical and Geotechnical Analyses

Soil samples will be collected in accordance with *Standard Operating Procedure for Soil Sampling*, provided in Attachment H. The soil samples will be sent to the ASC, which will subcontract the geotechnical samples. Analyses to be performed are listed in Table 5-1.

5.2.2.5 Sampling for Chemical Analyses

Soil samples will be collected in accordance with the *Standard Operating Procedure for Soil Sampling*, provided in Attachment H. The samples will be sent to the ASC, where they will be analyzed for the analytes listed in Table 5-1.

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5.2.2.6 Sample Containers and Preservation Techniques

Soil samples will be handled in accordance with *Standard Operating Procedure for Sample Packaging*, provided in Attachment F. The specific sample containers and preservation times will be coordinated with the ASC.

5.2.2.7 Field Quality Control Sampling Procedures

Field QC replicates will be collected from every 10th sample. A QA sample will be collected along with each field QC sample.

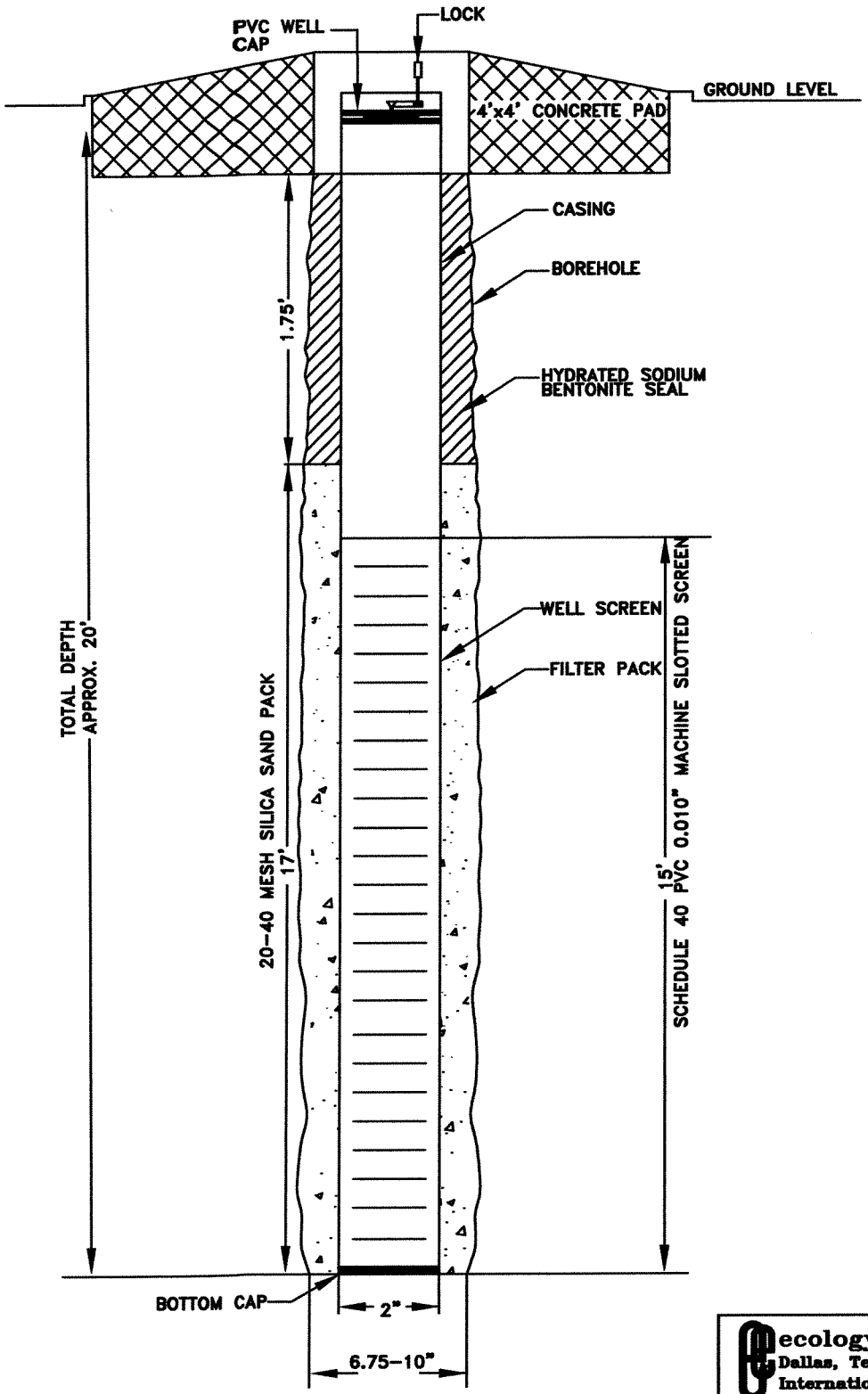
5.2.2.8 Decontamination Procedures

All sampling equipment will be decontaminated before initial use and between borings. Non-dedicated sampling equipment will be decontaminated in accordance with *Standard Operating Procedure for Sampling Equipment Decontamination*, provided in Attachment G. Rinsate blanks will be collected in sufficient number and frequency to demonstrate the effectiveness of decontamination, not to exceed 5% of total samples.

5.2.2.9 Sample Survey

E & E will survey the locations of the soil samples using a hand-held GPS unit.

TYPICAL COMPLETION DIAGRAM FOR EXTRACTION/MONITORING WELL



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NOT TO SCALE

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FIGURE 5-2
TYPICAL WELL SCHEMATIC
FORMER LAREDO AFB
LAREDO, WEBB COUNTY, TEXAS

JOB#: 001289.UX06	Date: OCTOBER 2001
File Name: 001289UX06D.DWG	P.M.: D. SMITH

Appendix C
Quality Assurance Project Plan

Soil and Groundwater Background Study
Quality Assurance Project Plan
Former Laredo Air Force Base
Laredo, Webb County, Texas

Contract No. DACA56-01-D-2001
Task Order No. 0004

July 2001

Prepared for:

United States Army Corps of Engineers, Tulsa District
Tulsa, OK

Approved by

Carol Wies, USACE Government Technical Manager	Date	Don Smith, E & E Project Manager	Date
		David Anderson, E & E Task Order Manager	Date
		Marcia Galloway, QC Director	Date

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A Ecology and Environment Inc., Analytical Services Center, Quality Assurance Manual

B Ecology and Environment Inc., Analytical Services Center, Analytical Standard Operating Procedures for Analytical Methods

C Data Qualifier Flags

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List of Acronyms

ASC	Analytical Services Center
DQOs	data quality objectives
E & E	Ecology and Environment, Inc.
EPA	United States Environmental Protection Agency
IWTP	industrial waste treatment plant
LAFB	Laredo Air Force Base
PCL	Protective Concentration Limit
PQLs	practical quantitation limits
QA	quality assurance
QAM	quality assurance manual
QC	quality control
SGR	shotgun range
SLF	sanitary landfill
SOPs	standard operating procedures
TNRCC	Texas Natural Resource Conservation Commission
TRRP	Texas Risk Reduction Program
USACE	United States Army Corps of Engineers

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1 Project Laboratory Organization and Responsibilities

The organization and management responsibilities of the Ecology and Environment, Inc. (E & E) Analytical Services Center (ASC) are described in detail in Section 2 and Appendix G of the laboratory quality assurance manual (QAM) included as Attachment A. Key laboratory staff includes Gary Hahn, laboratory director; Ray Piccione, laboratory quality assurance (QA) coordinator; and Caryn Wojtowicz, laboratory general manager.

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2 Data Assessment Organization and Responsibilities

E & E staff with the primary responsibility of conducting the data assessment include Don Smith, project manager; David Anderson, task order manager; chemists; and modeling specialists. Mr. Smith has the primary responsibility for all data assessment. Mr. Anderson is responsible for determining whether project and laboratory data quality objectives (DQOs) are achieved, and that data validation conducted by staff chemists are complete and accurate. Statisticians will be responsible for conducting calculations and assessments. Figure 2-1 shows the staff organization and lines of authority.

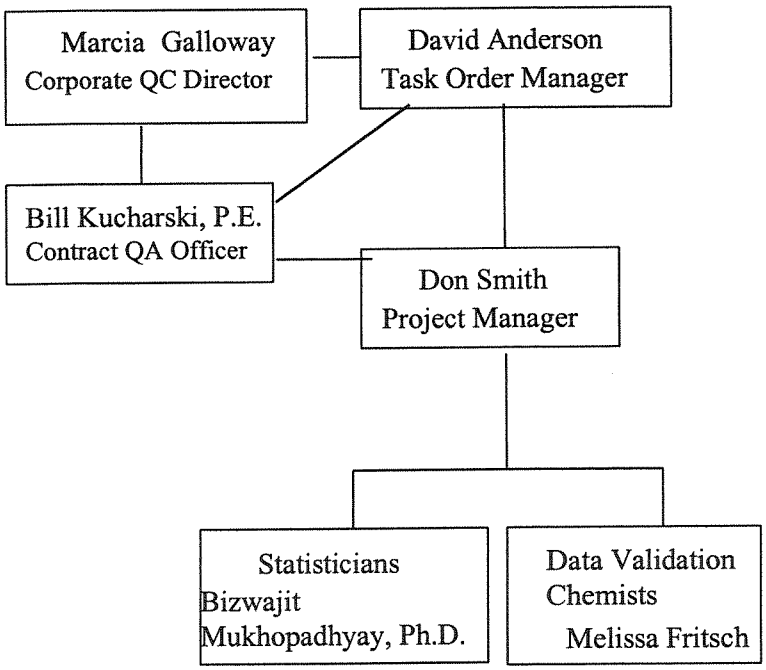

Figure 2-1
Data Assessment Staff Organization

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3 Data Quality Objectives

DQOs for the investigation are detailed in this section.

3.1 Data Use Background

DQOs have been developed for each of the two investigation tasks that are included in the scope of work: the background determination study and the site contamination assessment. Completion of the site contamination assessment depends on the results of the background determination study. *Guidance for the Data Quality Objective Process* (United States Environmental Protection Agency [EPA] QA/G-4, September 1994; EPA 1994) was used to develop the DQOs presented in the following sections.

3.2 Measurement Quality Objectives for Chemical Data Measurement

Measurement quality objectives for the chemical data are listed in detail in each of the analytical standard operating procedures (SOPs) in Attachment B. The DQOs for the background determination study and site contamination assessment are described below.

The planning team shall consist of the Tulsa ACE technical manager, Carol Wies; E & E Project Manager, Donald Smith; E & E Task Order Manager, David Anderson; E & E geologist, Paul James; and E & E statistician Biz Mukhopadhyay. Carol Wies is the primary decision maker.

3.2.1 Background Determination Study

Problem

Determine statistically valid background soil (surface and subsurface) and groundwater concentrations for metals, pesticides, and herbicides at the former Laredo Air Force Base (LAFB).

Decision

Have statistically valid background soil and groundwater concentrations for metals, pesticides, and herbicides been determined from the proposed sampling effort for use in the technical evaluation of the sanitary landfill (SLF), shotgun range (SGR), and industrial waste treatment plant (IWTP).

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Inputs to the Decisions

Groundwater. Data from previous site investigations (United States Army Corps of Engineers [USACE] 2000a, USACE 2000b, USACE 2000c) will be used to determine the effective porosity, hydraulic gradient, and hydraulic conductivity needed to calculate groundwater velocity and sampling frequency and to locate where the new monitoring wells are to be installed (EPA QA-5GS, February 1989 [EPA 1989], and 1992 addendum [EPA 1992]). The background concentrations will be calculated from the analytical data from the four sampling events conducted under this task order.

Soil. Data from previous site investigations will be used to determine the appropriate number of samples to be collected, based on the following: a) a preliminary estimate of the variability of the population expressed as standard deviation ; b) the two-sided confidence interval in the estimate of the average target concentrations; and c) the desired confidence in estimating the average target concentrations. Items b and c will be determined by the USACE government technical manager and 2 times the laboratory practical quantitation limit (PQL) and 95% confidence, respectively. The soil sample locations will be determined from current and documented land use (USACE 2000a, USACE 2000b, USACE 2000c). *Handbook for Statistical Analysis of Environmental Background Data* (U.S. Navy 1999) also will be used in the planning of the investigation. Analytical data from sampling will be used in statistical calculations to determine background concentrations.

Analytical procedures will be drawn from EPA publication SW-846, and other published methods.

Validation of sample results will be conducted using EPA Functional Guidelines as guidance on the individual parameters to check, with control limits being based on the USACE "Shell" document. USEPA QA-9 will be used to determine whether the collected analytical data meet DQOs. *Statistical Analysis of Ground-Water Monitoring Data at RCRA Facilities, Interim Final Guidance* (USEPA QA-5GS, February 1989 [EPA 1989]); the July 1992 addendum to the guidance (EPA 1992); and *Handbook for Statistical Analysis of Environmental Background Data* (U.S. Navy 1999) will be used for the statistical evaluation of the data. Confidence limits for the background data set will be calculated to assess whether background concentrations have been adequately determined.

Study Boundaries

The spatial limits of the former LAFB will be the boundaries for this study. Existing data will be examined to determine whether the soils should be separated into lithologic strata for evaluation. The sampling data are assumed to represent current and future soil and groundwater background concentrations. Sampling will be conducted in October 2001, with the specific frequency of groundwater sampling to be determined from the existing data.

Decision Rules

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USACE is interested in the true mean background concentrations of metals, pesticides, and herbicides at the former LAFB. If an adequate background concentration is not determined based on calculated confidence limit, additional sampling may be necessary to determine the background concentration.

Limits on Decision Errors

The two decision errors are: (1) deciding that an accurate background concentration has been determined for an analyte when in fact an accurate background concentration has not been determined; and (2) deciding that an accurate background concentration has not been determined for an analyte when in fact an accurate background concentration has been determined.

- The true state of nature for decision error (1) is that an accurate background concentration has not been determined.

- The true state of nature for decision error (2) is that an accurate background concentration has been determined.

The consequence of deciding that an accurate background concentration has been determined when in fact it has not is that decisions made in subsequent site evaluations using the data may be incorrect. This could result in either action taken to address contamination or a decision that no action is needed.

The consequence of deciding that an accurate background concentration has not been determined when in fact it has is that additional sampling would be required for the determination before decisions could be made in subsequent site evaluations.

It is concluded that decision error (1) has the most severe consequences because incorrect decisions regarding future site evaluations could be made.

- The baseline condition or null hypothesis is "an accurate background concentration has been determined."

- The alternative hypothesis is "an accurate background concentration has not been determined."

A false positive decision error occurs when a decision is made that an accurate background concentration is not determined when it truly is. A false negative decision error occurs when a decision is made that an accurate background concentration is determined when it truly is not.

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The gray region is defined as the laboratory's practical quantiation limit.
The decision error limits, positive and negative, have been set at 10%.

3.2.2 Site Contamination Assessment

Problem

Determine whether risk associated with contamination at the SLF, SGR, and IWTP can be attributed to background concentrations. Determine whether the extent of contamination at the sites is defined and whether a no-further-action alternative is supported.

Decision

Is the site contamination attributable to background conditions, and is a no-further-action alternative justified based on Texas Natural Resource Conservation Commission (TNRCC) Texas Risk Reduction Plan (TRRP) Protective Concentration Limits (PCLs)?

Inputs to the Decisions

Analytical results from previous sampling will be compared. Background concentrations of target analytes will be determined from the preceding study.

Statistical Analysis of Ground-Water Monitoring Data at RCRA Facilities, Interim Final Guidance (USEPA QA-5GS, February 1989 [EPA 1989]), the July 1992 addendum to the guidance (EPA 1992), and *Handbook for Statistical Analysis of Environmental Background Data* (U.S. Navy 1999) will be used for the statistical evaluation of the data.

PCLs to be utilized are the Tier 1 residential soils, 30 acre source area, ground water protection and the Tier 1 Groundwater PCLs for residential use.

Study Boundaries

For the SLF, SGR, and IWTP to be evaluated, the spatial boundaries are assumed to be the physical boundaries determined during previous investigations; however, the evaluation will be used to determine whether these boundaries are accurate. Each site will be evaluated independently.

Decision Rules

If the concentration of a target analyte is not statistically greater than its background concentration, then a no-further-action alternative is supported. If the concentration of a target analyte is statistically greater than its background concentration but is less than the its PCL, then a no-further-action alternative is supported. If the concentration of a target analyte is statistically greater than its background concentration and is greater than its PCL, then further action may be required.

Limits on Decision Errors

The two decision errors are (1) deciding that a site concentration determined for an analyte is less than its background concentration and its PCL when it is actually greater than the background concentration and its PCL; and (2) deciding that a site concentration

determined for an analyte is greater than its background concentration and its PCL when it is actually less than the background concentration and its PCL.

- The true state of nature for decision error (1) is that the site concentration is greater than the background concentration and PCL.

- The true state of nature for decision error (2) is that the site concentration is less than the background concentration and PCL .

The consequence of deciding that a site concentration is less than its background concentration and PCL when it truly is greater is that a no-action decision that could endanger human health and the environment may be made. Additional actions may have to be taken later to address the contamination not removed at this time.

The consequence of deciding that a site concentration is greater than its background concentration and PCL when it truly is less is that a decision to initiate remedial activities that are unnecessary may be made, thereby increasing costs.

It is concluded that decision error (1) has the most severe consequences because the risk of jeopardizing human health will outweigh the additional remediation costs.

- The baseline condition or null hypothesis is "the site concentration is greater than the background concentration and PCL."

- The alternative hypothesis is "the site concentration is less than the background concentration and PCL."

A false positive decision error occurs when a decision is made that the site concentration is less than the background concentration and PCL when it is truly greater. A false negative decision error occurs when a decision is made that the site concentration is greater than the background concentration and PCL when it truly is less.

The gray region is defined as 75% of to the PCL for pesticides and herbicides and 90% of to the PCL for metals . T

The decision error limits, positive and negative, have been set at 10%.

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4 Sample Receipt, Handling, Custody, and Holding Time Requirements

ASC procedures for sample management are described in Section 10 of the attached QAM (see Attachment A). Holding time requirements for specific analytical procedures are detailed in Appendix E of the QAM and in the individual method SOPs, provided in Attachment B.

4.1 Verification and Documentation of Cooler Receipt Condition

The ASC sample custodian examines each shipment of samples for discrepancies in documentation or condition as described in Section 10 of the QAM and applicable SOPs.

4.2 Corrective Action for Incoming Samples

Any discrepancies in documentation or problems with sample condition are noted and immediately brought to the attention of the E & E project manager, per Section 10.7 of the QAM and applicable SOPs. The E & E project manager will communicate with the USACE government technical manager regarding the corrective actions.

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5 Analytical Procedures

Analytical procedures to be conducted on samples collected during this investigation are detailed in Table 5-1. Extraction and analysis methods are included. Copies of the listed ASC SOPs are in Attachment B.

Table 5-1: Analytical Procedures:
Soil and Groundwater Background Study
Former Laredo Air Force Base; Laredo, Webb County, Texas

Analytes	Matrix	Method	SOP
Volatile organic compounds	Water	SW846-8260B	GCMS.17
Semi-volatile organic compounds	Water	SW846-3510C/8270C	EXT.20/GCMS.19
Chlorinated pesticides	Water	SW846-3510C/8081A	EXT.4/GC.72
Chlorinated pesticides	Soil	SW846-3550B/8081A	EXT.19/GC.72
Polychlorinated biphenyls	Water	SW846-3510C/8082	EXT.4/GC.73
Herbicides	Water	SW846-8151A	EXT.8/GC.60
Herbicides	Soil	SW846-8151A	EXT.7/GC.60
Total petroleum hydrocarbons	Water	TX1005	GC.78
Metals	Water	SW846-3010A/6010B	Metals.72/Metals.18
Metals	Soil	SW846-3050B/6010B	Metals.73/Metals.18
Mercury	Water	SW846-7470A	Metals.70/Metals.67
Mercury	Soil	SW846-7471A	Metals.71/Metals.67
PH	Water	SW846-9040B	
PH	Soil	SW846-9045C	GAC.100
Total cyanide	Water	SW846-9012A	GAC.65
Fluoride	Water	SW846-9056	GAC.145
Nitrate/nitrite	Water	SW846-9056	GAC.145
Chloride	Water	SW846-9056	GAC.145
Sulfate	Water	SW846-9056	GAC.145
Total organic carbon	Water	SW846-9060	GAC.20
Total Organic Carbon	Soil	Lloyd Khan	GAC.76
Ammonia	Water	SM4500	GAC.2
Chemical oxygen demand	Water	SM5220C	GAC.12
Total Kjeldahl nitrogen	Water	SW846-351.3	GAC.4
Total suspended solids	Water	SW846-160.2	GAC.38
Total dissolved solids	Water	SW846-160.1	GAC.37
Total phosphorus	Water	SW846-365.2	GAC.145
Grain size distribution	Soil	ASTM D422	*

* - To be provided by subcontract laboratory.
ASTM - American Society for Testing and Materials.
SM - *Standard Methods for the Examination of Water and Wastewater.*
SOP - Standard Operating Procedure.
SW - *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, EPA SW846.
TX - Texas Natural Resource Conservation Commission.

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5.1 Preventive Maintenance

General preventive maintenance guidance for the ASC is described in Section 7 of the QAM. Instrument-specific procedures are included in the method-specific SOPs. The ASC maintains a stock of spare parts and consumables for all analytical instruments. Back-up instrumentation is available for most analytical equipment. Service contracts are maintained for major instrumentation in the event that routine maintenance procedures do not eliminate problems that are encountered.

5.2 Calibration Procedures and Frequency

General calibration procedures are described in Section 8 of the QAM. Method-specific calibration procedures, frequencies, and acceptance criteria are listed in the method-specific SOPs.

5.3 Laboratory Quality Control Procedures

Specific quality control (QC) procedures are detailed in the individual method SOPs. The SOPs list the specific internal QC measures used, including method blanks, laboratory control samples, spikes, duplicates, or any other QC measure. Acceptance criteria for these QC procedures also are listed in the SOPs.

5.4 Performance and System Audits

Internal audits conducted by the laboratory QC administrator and audits by clients and accrediting agencies are conducted at the ASC. The ASC also participates in Performance Evaluation (Proficiency Test) Sample programs. Section 4 of the QAM describes audits, and Appendix C of the QAM lists the ASC's certifications and the accreditations.

5.5 Nonconformance and Corrective Action

Section 4.5 of the QAM describes the ASC's approach to implementing corrective actions as a result of nonconformances in data and services. The ASC is proactive in taking actions to prevent or correct nonconformances.

5.6 Sample Collection

Sample collection procedures are described in the Field Sampling Plan.

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6 Data Reduction and Calculation of Data Quality Indicators

Section 12.1 of the QAM describes the data reduction procedures conducted by the ASC. The analyst and peer review analyst conduct a complete review (100%) of QC elements. The project manager reviews the reported information for compliance with project requirements. The E & E task order manager conducts reviews on approximately 10% of the data reports generated by the laboratory. Data quality indicators are discussed in Section 4.6.1 of the QAM.

6.1 Precision

Precision, the reproducibility of measurements under a defined set of conditions, is evaluated using duplicates, matrix spike duplicates, or replicate measurements. *Relative percent differences*, *standard deviation*, and *coefficient of variation* are the terms used to state precision.

6.2 Bias

Bias, or accuracy, the relationship of reported data compared to true or expected values, is evaluated using laboratory control samples, surrogate and/or matrix spikes, performance evaluation samples, and blanks. *Percent recoveries* is used to state bias.

6.3 Sample Quantitation and Reporting Limits (Limit of Detection)

The ASC calculates and verifies method detection limits annually for all analytical methods. The ASC uses practical quantitation limits (PQLs) as reporting limits. The lowest standard used for calibration is typically at or below the PQL.

6.4 Completeness

A completeness goal of 90% is used for this project. Completeness is defined as the percentage of performed measurements that are valid, *valid data* being data not rejected for failure to meet QC goals.

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7 Laboratory Operations Documentation

7.1 Sample Management Records

Section 11 of the QAM and the SOP referenced in the QAM detail the ASC's document control and record procedures, including laboratory notebooks, data records, QA records, and archives.

7.2 Data Reporting Procedures

The ASC's procedures for reporting data are discussed in Section 12 of the QAM.

7.2.1 Data Package Format and Contents

Analytical data for this project will be reported in a format equivalent to the current EPA Contract Laboratory Program format.

7.2.2 Electronic Deliverables

An electronic deliverable will be prepared for this project using the electronic format standard developed by the ASC, as modified to meet USACE, Tulsa District, requirements. Acceptance criteria for QC measurements are included in the electronic data deliverable.

7.3 Data Management Procedures

Section 12.1 of the QAM details data management procedures utilized by the ASC.

7.3.1 Laboratory Turnaround Time

A turnaround time of 28 days from receipt of the samples has been specified for delivery of the final hard copy and electronic deliverables for this project. Preliminary data results for the initial groundwater samples will be provided with a seven-calendar-day turnaround time to facilitate the final three rounds of groundwater sampling.

7.3.2 Data Archival and Retention Requirements

Section 11.2.6 of the QAM describes the ASC's archival procedures.

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8 Data Assessment Procedures

8.1 Data Quality Control Review

Section 12.2 of the QAM describes the data QC review procedures conducted by the ASC.

8.2 Data Verification and Validation

Analytical data generated by the laboratory will be validated independently by qualified chemists utilizing EPA's organic and inorganic national functional guidelines as the basis for the validation. Criteria for evaluation of QC parameters will be the laboratory-determined 95% confidence intervals. These intervals will be compared to guides in the method to determine whether the intervals are consistent with those listed in the method. Standard qualifier flags as described in functional guidelines will be used, with supplemental flags being added to indicate the direction of any bias, if applicable. Attachment C contains a complete list of data validation qualifiers utilized and their meanings.

8.3 Data Quality Objective Reconciliation

After verification and validation of the analytical data are complete, statistical calculations will be conducted on the data to determine whether project DQOs are achieved. These calculations include:

- Determination of valid background concentrations for the analytes of concern; and
- Comparison of the background results to previous sample results to determine contaminant concentrations exceeding background concentrations and extent of contamination.

Additional information regarding DQO reconciliation is provided in the data reduction plan, which is in Section 3.2 of the work plan.

8.4 Project Completeness Assessment

As part of the validation process, chemists will conduct a completeness assessment, verifying that all required deliverables, hard copy and electronic, are provided.

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9 References

United States Environmental Protection Agency (EPA), n.d., *Contract Laboratory Program National Functional Guidelines for Inorganic Data Review.*

n.d., *Contract Laboratory Program: National Functional Guidelines for Organic Data Review, Multi-Media, Multi-Concentration (ILMO 1.0) and Low Concentration Water (OLCO 1.0).*

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Quality Assurance Project Plan
Attachment A
Ecology and Environment Inc., Analytical Services Center, Quality Assurance Manual

Quality Assurance Manual

Revision No. 3
September 2000

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The quality assurance system described in this Quality Assurance Manual has the support of the management at Ecology and Environment, Inc., Analytical Services Center. Every member of the Analytical Services Center staff is tasked to provide quality data to our clients. It is the responsibility of all personnel to use and follow this manual as a guide for quality improvement.

Signature: **Date:**

Gary Hahn
Laboratory Director

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General Manager

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List of Acronyms

% D	percent difference
% R	percent recovery
AA	atomic absorption spectrophotometers
APHA	American Public Health Association
ASC	Ecology and Environment's Analytical Services Center
ASTM	American Society for Testing and Materials
AWWA	American Water Works Association
BFB	bromofluorobenzene
BOD	biochemical oxygen demand
BOD_5	5-day biochemical oxygen demand
CAD	computer-aided design
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	chain-of-custody
COD	chemical oxygen demand
DFTPP	decafluorotriphenylphosphine
DOT	United States Department of Transportation
DQOs	data quality objectives
E & E	Ecology and Environment, Inc.
EDD	electronic data deliverables
EPA	United States Environmental Protection Agency
GAC	general analytical chemistry
GB	gigabyte

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GC/MS	gas chromatography/mass spectrometry
HMTA	Hazardous Materials Transportation Act
HP	Hewlett Packard
HPLC	high performance liquid chromatography
IC	ion chromatography
ICP	inductively coupled plasma
ICS	interference check samples
ICV	initial calibration verification
IPR	initial precision and recovery
KCl	potassium chloride
LCS	laboratory control sample
LIMS	laboratory information management system
LRS	Laboratory Report System
MB	megabyte
MDL	method detection limit
mg/L	milligrams per liter
MS	matrix spike
MSD	matrix spike duplicate
MSDS	material safety data sheets
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NFPA	National Fire Protection Association
NIST	National Institute of Standards and Technology
NYCRR	New York Code of Rules and Regulations
OSH	Occupational Safety and Health Act
OSHA	Occupational Safety and Health Administration
PARCC	precision, accuracy, representativeness, completeness, comparability
PE	performance evaluation
ppm	parts per million

PQL	practical quantitation limit
QA	quality assurance
QAM	Quality Assurance Manual
QAPP	quality assurance project plan
QC	quality control
RAM	random access memory
RCRA	Resource Conservation and Recovery Act
RPD	relative percent difference
SOP	Standard Operating Procedure
SOW	statement of work
TKN	total Kjeldahl nitrogen
TOC	total organic carbon
TOX	total organic halogen
TRPH	total recoverable petroleum hydrocarbons
UPS	uninterruptible power source
VIM	International Vocabulary of Basic and General Terms in Metrology

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Distribution Policy

The Quality Assurance Manual (QAM) distribution is controlled by the Ecology and Environment, Inc. (E & E) Analytical Services Center (ASC) Document Control Officer. The document is not released without approval by the Quality Assurance (QA) Officer or QA Coordinator. Uncontrolled copies are generally released to outside clients or agencies. Internal copies are controlled and maintained by the Document Control Officer.

When this QAM is revised and approved, it is distributed to each laboratory section. The distributed copies are recorded, along with the current revision number, in a distribution logbook or database maintained by the Document Control Officer. The names of the QAM recipients outside the laboratory are also recorded in the logbook. The recipients will be sent all revisions for a period of one year.

The attached revision table lists the revision type, date, applicable revision number, and distribution.

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Revision Table (Only most recent revisions shown.)

Section Number	Description	Revision Date

1

Introduction

QA
quality assurance

ASC
Analytical Services Center

QAM
Quality Assurance Manual

QC
quality control

SOPs
standard operating procedures

This manual describes the quality assurance (QA) program employed by Ecology and Environment's Analytical Services Center (ASC). It is intended to provide employees, accrediting agencies, and clients with an understanding of how ASC maintains an effective QA system. The Quality Assurance Manual (QAM) is divided into 16 sections. The sections provide general descriptions of ASC objectives, policies, facilities, organization, personnel, services, and specific QA and quality control (QC) procedures as practiced within each area of operation. Referenced figures, tables, and standard operating procedures (SOPs) support the descriptions in the QAM sections and related documentation. The content of the QAM sections is not modified for specific clients or agencies unless the requirements of these clients become standard policy. Client-specific requirements are included in project-specific documents or in the client-specific sections of the operational SOPs, as appropriate.

SOPs are maintained written procedures for implementing the activities described in this manual. The SOPs are made available to the staff in hard copy or as electronic documents as defined in the Document Control Procedure (see Section 11). All laboratory personnel assigned responsibilities outlined in this QAM adhere to the procedures and policies described here and in the laboratory SOPs.

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2

Definitions

NELAC
National Environmental Laboratory Accreditation Conference

EPA
United States Environmental Protection Agency

Definitions for this QAM are taken from the National Environmental Laboratory Accreditation Conference (NELAC) *Quality Systems Manual* (Revision 12, July 1, 1999); "Test Methods for the Evaluating of Solid Waste, Physical/Chemical Methods," United States Environmental Protection Agency (EPA) SW-846, Update III, June 1997; and other relevant state and federal guidance. The NELAC manual quotes relevant definitions from ISO/IEC Guide 2; ISO 8402; ANSI/ASQC E-4, 1994; the EPA "Glossary of Quality Assurance Terms and Acronyms"; and the *International Vocabulary of Basic and General Terms in Metrology* (VIM). These definitions are provided in Appendix A. The list of standard definitions is referenced in internal SOPs. SOPs include any definitions related specifically to that procedure.

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3

Organization and Management

3.1 Company

The location, mailing address, and phone numbers for ASC are:

Ecology and Environment, Inc.
Analytical Services Center
4493 Walden Avenue
Lancaster, New York 14086
Telephone: (716) 685-8080
Facsimile: (716) 685-0852

E & E
Ecology and Environment, Inc.

GC/MS
gas chromatography/ mass spectrometry

HPLC
high performance liquid chromatography

Ecology and Environment, Inc. (E & E) is a publicly held corporation registered and incorporated in the State of New York. E & E has been in business since 1970; ASC is a division of E & E. ASC serves environmental businesses; local, state, and federal agencies; industry; and various international projects.

3.2 Organization

ASC's core services include analysis of samples and interpretation of results. The primary contact at ASC for analytical testing is the Laboratory Director. In addition to standard laboratory analyses, ASC provides other services in conjunction with E & E's corporate staff.

The QA and management structure at ASC is presented on Figure 3-1. Appendix G shows the organizational chart with current staffing. ASC is divided into the following production sections: organic sample preparation, gas chromatography/mass spectrometry (GC/MS) semivolatiles, GC/MS volatiles, GC volatiles and semivolatiles and high performance liquid chromatography (HPLC), inorganics and sample custody. Project management, information systems, QA, and administrative units operate in conjunction with production work groups but report independently to the Laboratory General Manager.

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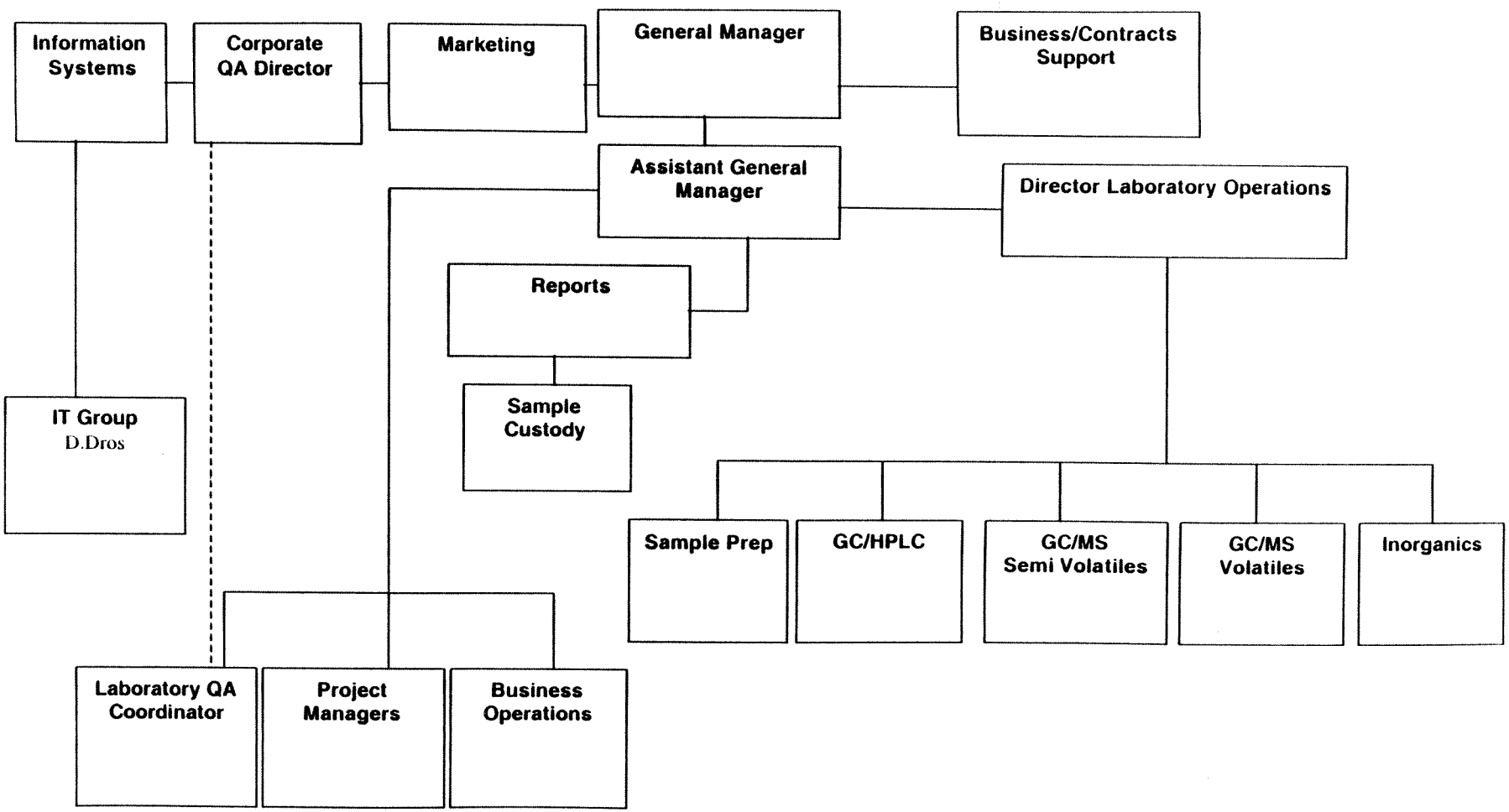

ASC ORGANIZATIONAL PLAN

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Responsibilities and authority of key personnel are summarized in this section. Resumes of key ASC personnel are found in the company's training record files.

3.2.1 Responsibility and Authority

All personnel are responsible for establishing, implementing, maintaining, and enforcing procedures for data quality management and control as defined in the QAM and related documentation. Each person is responsible for the day-to-day quality of data and the services performed. The overall responsibilities of the ASC staff are defined below. Additional specific job functions and duties are listed in the job descriptions for the following positions. Job descriptions are maintained in the QA files.

General Manager

- Reports to E & E ASC Executive Committee.
- Designates the Quality Assurance Coordinator.
- Ensures adequate resources are available to all staff to meet the objectives of the QA program.

Laboratory Director

- Reports to the General Manager and is responsible for all technical operations.
- Designates Section Supervisors.
- Responsible for:
  - implementing the QA program and technical operations at ASC;
  - overseeing of the supervision of personnel employed by the laboratory;
  - overseeing that criteria for accepting client samples are met and logged into the tracking system;
  - understanding client and market needs, requirements, or standards;
  - ensuring that data reported meet the method or client-specific requirements;

- overseeing data quality issues that affect the client; and
- participating in quality decisions affecting any client.

- Conducts an annual management review of all quality systems in conjunction with the General Manager.

- Names, in the event of a temporary absence, a chief-level project manager to fulfill all duties.

QA Coordinator

- Reports to the General Manager. May report directly to the Corporate QA Director any QA concerns.

- Implements and reviews all QA/QC practices, including corrective actions, internal audits, training, and other activities as defined in Section 4.3 and throughout the QAM.

- Monitors and reports to the Laboratory Director and General Manager on the status of all QA/QC related matters.

- Oversees maintenance of the QAM, the QA files, and other version-controlled documents as current documents.

- Oversees retrieval of all laboratory records as defined for archiving in the document control procedure.

- Is independent of all production responsibilities.

Section Supervisors

- Report to the Laboratory Director for all technical and production matters.

- Coordinate with the Laboratory Director to evaluate the resources available for daily work production.

- Review data and evaluate the QC elements for recurring or continuing nonconformances.

- Work with the Laboratory Director and General Manager to resolve process and system nonconformances.

Project Managers

- Reports to the General Manager for all matters related to client services.
- Implements and reviews all QA and QC practices as defined in the project- or client-specific requirements.
- Maintains all project files and client QA/QC documentation.
- Communicates relevant information to the appropriate staff.

Technical Staff

- Reports to the Section Supervisor for work assignments and scheduling of samples to be analyzed.
- Responsible for performing work according to the SOP, meeting agreed-upon time scale, and achieving the QC criteria defined in the SOP.
- Records information on a corrective action form when QC criteria are not within defined limits, or when procedures deviate from the SOP or client requirements.
- Performs all necessary administrative operations according to the documented SOP, including corrective action, document control, data review, traceability, and sample management.
- Performs all necessary ancillary technical operations according to the documented SOP or good laboratory practice, including routine preventative maintenance, standard preparation and inventory, and calibration of instruments and equipment.

Sample Custody Staff

- Reports to the General Manager or designee for all matters related to sample receipt, handling, and disposal.

- Responsible for performing work according the sample management SOP(s) and performing all operations within agreed-upon time scale.

- Records information on a Project Manager notification form when samples or materials are received that do not conform with client or method requirements.

- Performs all necessary administrative operations according to the documented SOP, including corrective action, document control, data review, and traceability.

Quality Assurance Staff

- Reports to the QA Coordinator for the job duties assigned.

- Records information on a corrective action form when data or other information is received that does not conform with the SOP or client requirements.

- Performs all necessary administrative operations and record keeping according to the documented SOP, including corrective action, document control, and data review.

Administrative Staff

- Reports to the General Manager or designee for the work area assigned.

- Responsible for performing work according to the assigned SOP and performing all operations within agreed-upon time scale.

- Records information on a corrective action form when data or other information is received that does not conform with the SOP or client requirements.

- Performs all necessary administrative operations and record keeping according to the documented SOP, including corrective action, document control, and reporting.

Corporate Quality Assurance Director

The corporate QA Director ensures compliance with the Corporate QA Program. The QA Director remains independent of day-to-day, direct project activities, but is responsible for ensuring that all QA/QC requirements are met. The parent company assists with QA oversight at the laboratory and utilizes staff with appropriate training and expertise to evaluate those operations.

3.2.2 Verification

Verification activities include inspection and monitoring of process and data quality, and auditing the quality system, processes, and data. Effective verification is achieved by providing personnel with adequate resources, including adequate training, supplies, and equipment; time for verification activities; knowledge of requirements and documented procedures; and access to quality records.

Technical personnel, under the direct supervision of the Section Supervisors, are responsible for the inspection and monitoring of in-process and final data. Audits of the quality system and process are performed by personnel who do not have direct responsibility for the work being performed. Quality system audits are carried out by the QA Coordinator. Process audits are scheduled by the QA Coordinator and carried out by designated personnel and trained internal auditors (see Section 4.4).

3.2.3 Management Review

The QA Coordinator reports directly to the General Manger, functions independently of production, and has the authority to implement and maintain the quality system. The QA Coordinator meets at least monthly with the Section Supervisors to determine the status of quality systems in their laboratory section. The QA Coordinator attends the biweekly management meetings and reports on the status of all QA activities, including open and closed corrective actions, internal audit results, external audit results, status of certifications and approvals, client complaints, and major quality issues. The QA Coordinator interacts frequently with personnel at all levels throughout the organization.

A management review of the quality system is conducted annually by the Laboratory Director, General Manager, and corporate QA. The management review must ensure that the quality system remains effective, meets quality objectives and policies, and satisfies the requirements of state and federal certifications or approvals held by ASC. Management review includes review of the annual QA systems audit and changes in laboratory operations, staff activities, and client concerns. The management review includes areas for improvement in relation to quality systems.

4

Quality System

4.1 Objective

The objective of the staff at ASC is to provide data and services that are legally defensible, scientifically valid, timely, and to the satisfaction of clients' expectations.

4.2 Policies

The management of ASC supports the following policies in order to achieve the objective and promote the overall QA program:

- Laboratory procedures, including client requirements, are documented in SOPs that clearly communicate these procedures to laboratory personnel.
- Management emphasizes quality improvement through commitment and leadership.
- A comprehensive QC system has been established and is maintained to verify and assure continued precision and accuracy of analytical results.
- Adequate training on laboratory operations is provided for all employees whose decisions affect the quality of laboratory data and services.
- A comprehensive program of documentation has been implemented and is maintained to ensure accountability and traceability throughout the analytical process.
- Measures are taken to ensure that sample integrity is protected.
- Studies are performed to demonstrate the capabilities for each analytical method.

- The instrumentation, equipment, and materials used in the production process are controlled to ensure that required quality standards are within specifications.

- A comprehensive program for data reduction, review, reporting, and archiving is maintained.

- Preventive and corrective actions are taken, as necessary, to eliminate the causes of potential or actual nonconformance.

- Measures are taken to fulfill the requirements of agencies from whom certifications and accreditations have been granted.

- Client-specific requirements are incorporated in the overall QA program by addendum to appropriate SOPs.

Quality control: A system of activities applied at each stage of the data production process designed to ensure that data meet defined quality standards. This system includes the following: system, operational, and performance audits; reference materials; statistical evaluations; re-analysis; and measurement bias investigation (when measurements may be operator-, instrument-, or methodology-dependent).

Quality assessment: A system of activities designed to ensure that quality control takes place at each stage of the production process. This system includes the following: employee education, training, and experience; documentation (e.g., instructions, document control, records); instrument calibration and maintenance; traceability of samples, data, and methods; laboratory facili-

4.3 Quality Assurance

Through a formal QA system, ASC provides testing services that meet specific quality standards. These quality standards are defined to meet the needs and requirements of clients, the analytical methods used, government agencies, and E & E's senior management.
Quality Assurance is composed of **quality control** and **quality assessment**.

ASC management is accountable for the quality of the services provided, and each employee is an integral part of achieving the system objectives. This section is limited to a discussion of the major activities performed and administered by the QA unit; policy details are discussed throughout this manual.

The QA program at ASC is affected by the requirements of certification agencies. The QA Coordinator is directly responsible for coordination of the following external quality assessment activities:

- On-site audits by outside agencies;

- Analysis of performance evaluation (PE) samples;

- Corrective action responses to deficiencies cited in external audit reports and external PE results; and

PE
performance evaluation

- Dissemination of requirements and status of certifications to relevant laboratory personnel and project managers.

4.3.1 Quality Assurance Unit

At ASC, the QA unit monitors the QA system and reports the results of its observations to the Laboratory Director, General Manager, and QA Officer. The QA Coordinator has no direct responsibility for production, scheduling, or workflow in the laboratory. The objective of this independence is to eliminate all conflicts of interest in the performance of QA duties. Major activities performed and administered by the QA unit are summarized below. Each activity is discussed in greater detail elsewhere in the QAM.

- Performance of internal audits and coordination of external audits (Section 4.4);
- Administration of laboratory certification/accreditation programs (Section 4.7);
- Administration of a system for formal corrective action reports (Section 4.5);
- Administration of the document control system, including the publication of the QAM (Section 11) and other documents that describe ASC's QA system; and
- Administration of the in-house training program (Section 5.5).

4.4 Audits

ASC conducts internal audits in which the laboratory reviews and examines itself, and undergoes external audits in which the laboratory is audited by outside organizations. Internal system audits are defined in the internal audit SOP QA.19 as operational and system audits.

4.4.1 Internal Audits

The internal audit is a review of the quality system as it is employed in the laboratory. Auditors verify that adequate written instructions are available for use; that analytical practices performed in the laboratory are consistent with SOPs; that the QC practices are applied during production; that corrective actions are applied as necessary; that devia-

tions from approved protocols occur only with proper authorization and documentation; and that SOPs, quality records, analytical records, and magnetic tape are properly maintained.

System Audit

Internal system audits are implemented by the QA Coordinator at least once per year. The system audit is a review of the overall system of operations and is used to verify that corrective actions are being implemented effectively. The system audit is a sampling of all operations and must include sample tracking from receipt to disposal, a data audit of a completed report, and audits of ancillary operations as necessary to support the data audit. Data audits ensure that data comply with method and SOP requirements, deliverable specifications and standard traceability. The internal system audit report is provided to laboratory management and forms the basis for management review of the quality systems described in Section 3.2.3.

Operational Audit

The QA Coordinator schedules internal operational audits for all sections at least once per year. The operational audits are a more detailed review of laboratory (procedure) operations. They are performed by analysts, Section Supervisors, or supervisors from sections other than those being audited. This manner of conducting operational audits allows for the exchange of ideas, improving production flow and operations.

Prior to a scheduled audit, checklists or the SOP specific to the area are reviewed by the assigned auditor. The checklist or notes from the audit are returned to the QA Coordinator as part of the audit report. The specific contents and process for conducting and reporting on the audit are detailed in the internal audit SOP QA.19. Any deficiencies or nonconformances found during the audit are recorded on a corrective action form. The corrective action procedure is followed to ensure the nonconformance is corrected.

4.4.2 External System Audits

Representatives sent by clients, government, or accrediting agencies often perform system audits at ASC. These audits are announced inspections. The QA Coordinator accompanies the external audit team through the laboratory. The auditors receive a brief overview of company objectives, activities, and facilities. Interviews with essential su-

pervisory and technical staff are arranged and any documentation pertinent to the audit is retrieved. Auditors typically provide a report on their findings shortly after the audit. This account is evaluated by the QA Coordinator and reported to management, along with a corrective action form responding to any cited deficiencies.

4.4.3 Performance Evaluation Samples (or Proficiency Test Samples)

A PE or PT sample analysis is the analysis of a fortified blank sample or real world sample for the purpose of evaluating laboratory or analyst performance. PE samples are submitted by external organizations and have analyte concentrations unknown to ASC. PE may be analyzed as part of inter-laboratory round-robin studies, in conjunction with accreditation programs, or as blind and double blind check samples submitted by clients.

Internal PE samples are fortified blanks with known analyte concentrations. On a continuing basis, an analyst must demonstrate performance in the method as described in the SOP on Continuing Staff Proficiency A.29. Performance requirements may be from external or internal PE samples or other means such as repetition of initial demonstration of capability as described in the SOP. Internal performance audits may be used to provide information to an accrediting agency on correction of past performance on an external performance audit.

As the sole laboratory for E & E, the ASC does not participate in its own interlaboratory comparisons except through external PE programs or client split samples.

4.5 Corrective Action

ASC takes the necessary actions to prevent or correct nonconformances in data and services. When corrective actions result in permanent procedural changes, pertinent documentation (e.g., SOPs) is modified to reflect these changes. A formal corrective action reporting system, administered by the QA unit, is in place at ASC. The process is described in the corrective action SOP QA.1 and is applicable to all nonconformances or client concerns.

Routine corrective actions are made by the analysts, technicians, or other personnel who detect problems or nonconformances during analysis of samples. SOPs for testing procedures contain instructions

for implementing and documenting corrective actions for typical problems. These actions are typically noted in the laboratory section documentation. Any data reported outside SOPs or project requirements must be described on a corrective action form submitted with the results for project manager approval.

Sample handling concerns, such as problems during cooler receipt or holding time violations, are reported immediately on a Project Manager Notification form to the project manager for resolution. Deviations from the SOP and other activities that require approval or actions by others also may be recorded on the corrective action form (see Appendix B).

All non-routine problems, deficiencies, or irregularities are reported to management as part of the corrective action process. The corrective action process is used to report all audit findings from both external and internal audits as well as any client comments or complaints from the review of analytical data.

The QA unit monitors the progress of corrective actions, maintains completed documentation, and provides reports to the Laboratory Director on the status of the corrective action activities. Corrective action forms may be originated by any ASC personnel. A completed form is sent to the person responsible for completing the corrective action. This person is responsible for recording the corrective action plans, implementations, and follow-up actions to be completed. During the corrective action process, several measures may be taken, including: determination of the root cause through careful analysis of processes, specifications, quality records, customer complaints, and use of statistical process control; implementation of process controls to ensure that effective corrective action is taken; application of remedial actions to reports or data affected by the identified problem; and revision of documentation for procedures that changed as a result of the corrective action. The goal of the corrective action process is to complete all corrective action forms before data are reported to the client.

4.6 Quality Control

QC is applied at every stage of the testing process at ASC. Its purpose is to assure that the data reported to clients are within defined acceptance and performance criteria. QC activities are described in many sections of this manual; this section presents those QC activities that are quantified with defined acceptance and performance criteria.

The criteria are derived from several sources, including requirements of the analytical methods, needs stated by clients, and standards established within ASC. During the generation of data, verification activities evaluate the performance of the measurement to the specified criteria. These verification activities include data review by a second analyst, proofreading of all data entry, project-specific review, and a data review by the QA Coordinator on a random selection of the final reports (see Section 12.2).

When defined quality standards have not been met (a nonconformance), the corrective action process is initiated to document and track the nonconformance and the corrective actions taken. The QA Coordinator and Laboratory Director or their designees review the information to determine the effectiveness of the QC system.

4.6.1 Data Quality Objectives

DQO
data quality objective

SOW
scope of work or statement of work

PARCC
precision, accuracy, representativeness, completeness, and comparability

RPD
relative percent difference

LCS
laboratory control sample

The data quality objectives (DQOs) are developed by the client during the design of a given study and typically presented to the laboratory as a QAPP or contract scope or statement of work (SOW). The DQOs are qualitative and quantitative indicators used to define the quality of the data needed to support specific project decisions. DQOs specify critical data points or acceptance criteria to be achieved to make the proper decision. The qualitative and quantitative indicators are often expressed as precision, accuracy, representativeness, completeness, and comparability (PARCC). Achievement of these quality indicators helps to demonstrate that the data are scientifically valid, legally defensible, and adequately meet client requirements as stated by the DQOs. The laboratory reviews all project-specific DQOs and identifies any areas in which internal QC criteria indicate project DQOs may not be achievable (see Section 13).

Precision

Precision measures the reproducibility of measurements under a defined set of conditions. It is a quantitative measure of the variability of a group of measurements compared to their average value. Precision is usually stated in terms of standard deviation, coefficient of variation, or relative percent difference (RPD) between two values. Precision is determined in the laboratory by evaluating laboratory duplicates, matrix spike duplicates, or replicate measurements of the sample or standard.

Precision measurements for overall sampling and analytical testing are evaluated through field duplicates or split samples.

Accuracy

Accuracy is a quantitative measure of the relationship of reported data compared to the true or expected values and includes a combination of random and systematic error. Accuracy is determined in the laboratory by evaluating the recoveries of laboratory control samples (LCS), through the use of surrogate spikes, and stated in terms of percent recovery or the average (arithmetic mean) of the percent recovery. Accuracy measures for overall sampling and analytical testing are evaluated through blank samples, background samples, or external PE samples.

Representativeness

Representativeness expresses the degree to which data represent a characteristic of a population, a parameter variation at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that is most concerned with proper design of the measurement program. Sample measurement locations may be biased (judgmental) or unbiased (random or systematic). For unbiased schemes, the sampling must be designed not only to collect samples that represent conditions at a sample location, but also to select sample locations that represent the total area to be sampled.

Representativeness is usually dependent upon sampling techniques not controlled by the laboratory; however, there is representativeness of sub-samples prepared within the laboratory from collected samples. Parent samples must be subjected to thorough homogenization prior to sub-sampling to ensure unbiased measurements within the laboratory. Sample duplicates prepared in the laboratory are a measure of the representativeness.

Completeness

Completeness is defined as the percentage of measurements performed that are judged to be valid. A quantitative goal defines how much valid data must be generated to make a decision regarding completeness. Critical samples typically have higher completeness goals than noncritical samples and require additional measures of QC. In the laboratory, the quantitative measure of the amount of valid data is obtained from the analytical process and compared to the amount that was expected to be

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obtained. Valid data are data that are not rejected for failure to meet project DQOs.

Comparability

Comparability is a qualitative measure of the confidence with which one set of data can be compared to another. Sample data should be comparable with other measurement data for similar samples and sample conditions. Characteristics that make comparison possible include method performance samples as defined in Section 4.6.2; QC samples as defined Section 4.6.3; standardized report format; consistency of units (e.g., milligrams per liter [mg/L], parts per million [ppm]); and standardized sample collection, preparation, and analysis procedures. PE samples also help establish comparability of results between laboratories.

mg/L
milligrams per liter

ppm
parts per million

4.6.2 Method Performance

For any given analytical method, ASC analysts initially demonstrate their ability to successfully perform the analytical method, and continue to demonstrate this ability as long as the method is used. Initially, the laboratory analysts demonstrate the ability to meet the precision and accuracy criteria stated in the method and defined in the initial demonstration of capability SOP A.23. The demonstration normally requires analysis of four replicate samples. Each individual analyst also demonstrates the ability to perform the method in accordance with the training SOP QA.5 and method requirements. On an ongoing basis, the laboratory analyzes calibration standards as defined in Section 8, QC samples as defined in Section 4.6.3, and PE samples as defined in Section 4.4.3 to demonstrate method capability.

Method sensitivity is defined as the concentration of an analyte in a given matrix necessary to produce a detectable response. The sensitivity is measured for each analytical method as defined below.

Method Detection Limits

The method detection limit (MDL) for all quantitative analyses is determined based on the reference procedure found in 40 CFR Part 136, Appendix B and is verified annually per the MDL SOP A.18. The MDL SOP details the calculations and iterative process for determining the MDL. An MDL is determined for each method and matrix during method start-up and whenever the basic chemistry of the procedures is changed.

MDL
method detection limit

PQL
practical quantitation limit

Practical Quantitation Limits

The practical quantitation limit (PQL) is the reporting limit used by the laboratory. The PQL must be greater than the MDL and is typically three to 10 times the MDL, depending on the method. The lowest standard used for calibration is at or below the PQL. Where client-specific criteria require reporting of a PQL that is below the lowest standard in the calibration range, a low level check standard is performed at the end of the analysis to validate the lower reporting limit. These client-specific reporting limits and the acceptance criteria for performance are defined in the client documentation.

4.6.3 Quality Control Samples and Splits

Analytical performance is monitored through QC samples and spikes, such as laboratory method blanks, surrogate spikes, QC check samples, matrix spikes, matrix spike duplicates, duplicate samples, and duplicate injections. Many of the QC measures applied at ASC are summarized below. Laboratory QC criteria are defined in the appropriate method SOP. A heirarchy is established in the application of QC criteria: project-specific, method then laboratory-derived requirements.

All QC samples are applied on the basis of a laboratory batch. The laboratory batch associates samples that are prepared or analyzed together as described in the batching SOP A.22. The laboratory defines two basic types of batches: the preparation batch and the run (i.e., analytical) batch. The preparation batch includes all samples processed as a unit during organic sample preparation, metals digestion, or General Analystical Chemistry preparation. Preparation batches do not exceed 20 samples excluding associated QC samples. The QC samples associated with sample preparation include method blanks, LCS, matrix spikes, and duplicates. The run batch is all samples analyzed together in the run sequence. The run sequence is typically limited to 24 hours unless defined differently for the analytical method. For some analyses, such as volatile organics, the run batch is equivalent to the preparation batch. The QC samples associated with the run sequence include calibration standards, instrument blanks, and verification. The specific procedures for documenting the laboratory batch are defined in the batching SOP. Additional batches may be defined for unique steps of the analytical process, such as a clean-up batch or reporting batch, and these additional batches are defined in the laboratory case narrative.

Laboratory Method Blank

A laboratory method blank is an analyte-free material processed in the same manner and at the same time as a client sample. The blank is prepared using American Society for Testing and Materials (ASTM) Type II water when analyzing water samples, and where practical, pre-cleaned sand or other solid material, such as sodium sulfate, when analyzing solid samples. The laboratory method blank sample is prepared along with the client samples at a frequency of one laboratory method blank per batch of 20 (or fewer) of client samples for the given matrix type.

ASTM
American Society for Testing and Materials

The laboratory method blanks serve to demonstrate a contamination-free environment in the laboratory. The goal is for method blanks to be free of contamination. Low level contamination may be present, but must be less than the level in the samples as defined by the method SOP or the client. If contamination is greater, the samples are reanalyzed. If contaminants are present in the method blank but not in client samples, no further action is required. All sources of contamination that are not common laboratory contaminants as defined in the method SOPs are investigated as part of the corrective action process. Sample results are not blank subtracted unless specifically required by the analytical method.

Surrogate Standards

For certain organic methods, all samples, including the laboratory method blank QC samples (MS, MSD, LCS) and standards, are spiked with a set of specific surrogate standards to monitor the accuracy of the analytical determination. Surrogate spikes are added at the start of the laboratory preparation process. Surrogate compounds are not typically found in environmental samples. QC criteria for surrogate recoveries are method-and matrix-specific. Laboratory QC criteria are established after a sufficient number of data points (30 or more) is performed and updated annually as defined in the Control Charting SOP QA.20.

The percent recovery (%R) is calculated as:

$$\%R = \frac{X_1 - X_2}{|\,TV\,|} x\ 100$$

where:

- % R = the percent recovery
- X_1 = the measured concentration of the sample with the spike addition
- X_2 = the measured concentration of the sample
- TV = concentration of

Surrogate recoveries within QC criteria for method blanks and LCS samples demonstrate acceptable method performance. If surrogate recoveries are outside QC criteria for method blanks or LCS samples, corrective action is required as defined in the appropriate method SOP Surrogate recoveries in the samples indicate the method performance

on the particular sample matrix. Surrogate recoveries that are outside QC criteria for a sample indicate a potential matrix effect. Matrix effects are verified based on review of recoveries in the method blank or LCS, sample reanalysis, or evaluation of interfering compounds. Sample clean-up procedures may be appropriate to alleviate potential matrix problems. Standard procedures for evaluating surrogate recoveries in samples are listed in the appropriate method SOPs. However, these procedures are often modified by project- or client-specific requirements.

Laboratory Control Sample

An LCS consists of ASTM Type II water, and where practical, precleaned sand, glass beads, or sodium sulfate for solid matrices, or a purchased performance evaluation sample. The LCS is spiked with the analytes of interest or a representative subset usually near the mid-point of the calibration range, followed by the same sample preparation, standard addition, and analysis as client samples. The LCS concentration may be specified in the reference method or project-specific requirements. LCSs are analyzed at the frequency of one per batch of every 20 samples or fewer. The recovery of target analytes in the LCS is an estimation of method accuracy.

LCS recoveries are monitored on control charts. Laboratory QC criteria are established for each method and matrix using a minimum of 30 points. QC criteria are updated annually. If all target analytes are not included in the LCS spike, then the spike mixture is rotated throughout the year to evaluate control limits for all parameters. When the LCS recovery is within the control limits, the method performance is considered acceptable.

If the number of LCS recoveries as specified in the SOP are outside QC criteria for the target analytes, corrective action is required. QC criteria and appropriate corrective actions are defined in the specific method SOPs. After corrective action is complete, sample re-analysis may be required for the failed parameters. If LCS recoveries exceed the QC criteria, and that parameter is not found in any of the samples, re-analysis is not necessary unless specifically required for the project.

Matrix Spike Sample

An MS sample consists of a client sample (often specified by the client) split into two parts and processed as two separate samples in a manner

identical to that of the rest of the samples. In addition to the regular addition of monitoring standards (internal standards, surrogate), spiking analytes are added to one aliquot of the chosen sample before preparation. Generally, all method target analytes, if compatible are added. A subset, as indicated in the method SOP, is reported unless specified otherwise by the client. The spike concentration levels for the target analytes are defined by the analytical method or by project-specific requirements or are specified by the laboratory in the analytical SOP. An MS is prepared for every extraction batch of 20 samples (or fewer) for a given matrix if sufficient sample allows. The sample chosen for matrix spike is random except that field and trip blanks are not chosen for spiking. Clients may specify the sample(s) to be spiked. The client must provide ASC with extra sample amounts for samples requiring matrix spikes.

Due to matrix effects, matrix spike recoveries are not a measure of the performance of the method on the sample being analyzed. In the absence of observable quantitative interferences, an MS sample recovery that exceeds QC limits indicates matrix effects. For these reasons, MS recoveries are not monitored on control charts. The results of the MS are evaluated, in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis.

Matrix Spike Duplicate Sample

The MSD sample is commonly prepared in conjunction with the MS sample. The MSD is prepared from a separate portion of the client sample and processed with the same additions as the MS. The sample chosen for matrix spike duplicate must be the same as that chosen for MS. The MSD is prepared for methods that do not typically show concentrations of target analytes above MDLs, such as organic methods. The RPDs between the recoveries in the MS and MSD measure the precision of the analytical method on the actual client samples. Laboratory QC criteria for RPDs may be established as specified in the Control Chart SOP QA.20 or by method requirements. RPD values may be tabulated in the analytical reports and not plotted on control charts. For the MSD sample, the client must provide ASC with extra sample amounts. If insufficient sample is available, the laboratory may run an LCS duplicate to measure precision, depending on the method.

The relative percent difference (RPD) is calculated as:

$$RPD = \frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2}\right)} \times 100$$

where:

RPD = the relative percent difference

X_1 = the concentration or percent recovery of the sample

X_2 = the concentration or percent recovery of the duplicate sample

Duplicate Sample

A duplicate sample consists of a set of two samples obtained in an identical manner from the same client sample. The collection of duplicate samples from a heterogeneous matrix requires homogenization to ensure that representative portions are analyzed. One sample per batch of 20 samples or fewer per matrix is analyzed. The sample chosen for duplicate analysis is random except that field and trip blanks are not used. Clients may designate the sample for duplicate analysis. The client must provide sufficient sample to allow for duplicate testing.

The duplicate is prepared for methods that typically show concentrations of target analytes above MDLs, such as metals and GAC methods. The RPDs between the recoveries in the original and duplicate measure the precision of the analytical method on the actual client samples. Laboratory QC criteria for RPDs are established for each method and matrix as defined in the Control Chart SOP QA.20. RPD values also may be tabulated in the analytical reports and not plotted on control charts.

If all other QC criteria are met, matrix effects may be established by observing the sample to determine any visual heterogeneity. If visual observation does not indicate a potential problem, the sample may be reanalyzed. Potential matrix effects are reported in the case narrative.

Splits

Clients may request that samples be split and sent to another laboratory, or they may split the samples and submit them to a minimum of two laboratories. Splits are often performed to evaluate the comparability and reproducibility of the data for critical decision making, or when sample re-collection is not practical. These inter-laboratory studies are defined in the client documentation. Data resulting from splits are evaluated by the client. Sample splits must be representative of the sample submitted. Errors from sample splitting must be minimized by ensuring that the sample is adequately mixed prior to splitting.

Other Laboratory QC Samples

The laboratory performs analysis of other QC samples or standards, depending on the analytical method. Standard QC samples or standards are documented in the specific method SOP. Method-specific QC samples or standards include internal standard spikes for GC/MS methods; post-digestion spikes and serial dilutions for metals analysis.

ICS
interference check sample

ICP
inductively coupled plasma

and interference check samples (ICSs) for inductively coupled plasma (ICP) analysis.

Field QC Samples

To evaluate the overall sampling and analytical data quality indicators, the client submits QC samples from the field. The field QC samples include trip blanks, field blanks (such as ambient condition blanks or equipment blanks), and field duplicates. Trip blanks may be prepared in the laboratory as described in Section 10. The project managers will normally track the field QC samples to ensure that the frequency of sampling and analysis is consistent with project requirements. In most cases, the analytical staff are typically not aware of which samples are the field QC samples. In cases when MS/MSD samples are not client-specific for water sample analysis, the analyst should review the client samples to ensure that MS/MSD analysis is not performed on field blanks.

4.6.4 Statistical Evaluation

Statistical evaluations are performed for the quality indicators, MDLs, surrogates, and LCSs. Control charts offer the most graphic representation of a statistical evaluation. Control charts identify potential problems, and the causes of these problems are promptly investigated and eliminated. Internal QC criteria are determined through historical trend analysis of data collected on QC charts. The control chart process is defined in the control chart SOP QA.20. The control chart procedure allows for identification of points outside QC criteria or potential QC problems by evaluation of trends.

Control charts are plotted for the percent recovery of surrogates, and LCS compounds, as determined by the individual method SOP or certifying agency requirement.

4.7 Accreditations, Certifications, Licenses, and Registrations

ASC has received approval from various state, federal, and national agencies for performing analytical testing. The laboratory has been granted numerous certifications and accreditations, based upon compliance with standards set forth by the granting agencies. The certifications are summarized in Appendix C. The scope of approval is based on each agency's requirements with regard to regulatory requirements, methodologies, or matrix. Specific information on the parameters ap-

proved is maintained in the QA files. The scope of evaluation as presented in Table C-1 in Appendix C is the process used for granting the approval.

5

Personnel

5.1 Job Qualifications for Key Technical Personnel

The following are the minimum experience, education, and qualifications required for key personnel. Specific job descriptions for each laboratory function are maintained in the QA files.

Laboratory Director: Experience in environmental laboratory managing administrative, business, personnel, and production activities. Minimum qualifications:

- Education: Bachelor's degree in chemistry or any related scientific/engineering discipline with at least 24 college semester hours in chemistry.
- Experience: Two years laboratory experience.

Laboratory Staff: The laboratory has sufficient personnel, having the necessary education, training, technical knowledge, and experience for their assigned functions.

All personnel are responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function. Each technical staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of his/her particular function and a general knowledge of laboratory operations, test methods, quality assurance/quality control procedures, and records management. Specific requirements for training are found in the training SOP QA.5

Some clients or programs may have specific requirements for specific positions. These requirements are specified in SOP QA.5.

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5.2 Recruitment Policy

ASC's recruitment policy is defined by the corporate program. Recruitment is the responsibility of the Laboratory Director and corporate human resources personnel. Orientation training of all new employees includes review of the QAM, Chemical Hygiene Plan, and all SOPs required for the job function.

5.3 Corporate Ethics Policy

Laboratory ethics and ASC's ethics policy are discussed during initial training for new employees. All new personnel receive ethics training and are required to sign an ethics agreement. Ethical standards training is reinforced on an annual basis. All employees are required to sign an ethics agreement following annual training. The corporate ethics policy is posted throughout the laboratory. Any employee found to have knowingly reported data values that are not actual values obtained, or improperly manipulated or intentionally reported dates and times of data analyses that are not the actual dates and times of analyses, is immediately terminated. Any laboratory personnel identifying an accidental or suspected intentional reporting of non-authentic data must report the finding immediately. Information can be provided to the Laboratory Director, Laboratory QA Coordinator, Laboratory General Manager, or Assistant General Manager, or in cases where anonymity is desired, to the Corporate QA Director. The project manager immediately notifies the client if any improper data are reported.

5.4 Corporate Confidentiality and Propriety Rights

All work is performed in the strictest confidence. During orientation training, new and contract employees are required to review the corporate policy and practices for protecting client confidentiality and proprietary rights. The policy and practices are designed to ensure that information from the laboratory is transmitted only to the appropriate and responsible parties.

5.5 Training

New employees receive all necessary training for their job function. Refresher training is provided for current employees as required for their current job function. Training is performed both on and off site by

members of ASC staff, contractors, equipment manufacturers, academic institutions, or regulatory authorities. Training procedures are detailed in the training SOP QA.5.
Off-site training takes place on an as-needed or availability basis. Completed studies are documented and updated in the training files of each employee. Off-site training may include the following:

- Courses taken at local colleges and universities.
- Workshops and seminars conducted by instrument manufacturers, software companies, and national associations specializing in analytical chemistry or laboratory QA.
- Specific training classes for instruments or software purchased at the laboratory.

Personnel provide the Document Control Officer with any attendance information and copies of any certificates earned during such training.

On-site training takes place for all new personnel according to the training procedure SOP. Personnel must demonstrate proficiency in the analytical and/or administrative procedures before independent operation in the job function. On-site training includes the following:

- Formal training in the QAM for all employees when newly hired and at yearly refresher training.
- Formal training on appropriate SOPs and all major revisions to those SOPs, including QC requirements.
- Assistance for the trainee performing a procedure.
- Observation and oversight for the trainee performing a procedure.
- Verification that the trainee demonstrates proficiency in the procedure. Each analyst is required to show an initial demonstration of capability through analysis of four known standards (SOP A.23) and an annual demonstration of proficiency through analysis of a PE sample, repetition of initial demonstration of capability or other means described in SOP A.29.

5.5.1 Training Records Maintenance

The Document Control Officer is responsible for maintaining the training record files and entering information into the tracking database. Reports from the database provide training summaries by individual, and by SOP. The Document Control Officer notifies appropriate personnel when a revision is finalized for the controlled versions of the documents that will require retraining of all appropriate personnel.

Resumés, job descriptions, and biographies are included in the training record files. Biographies are put in a uniform format by the corporate human resources department and are updated with additional education and experience as needed.

5.6 Safety and Health Policies

All laboratory personnel receive orientation training session upon initial employment as well as on-the-job training concerning health and safety issues related to the operations being performed. In compliance with the Occupational Safety and Health Administration (OSHA), ASC conducts safety and health training annually, with a careful introduction to new principles. Personnel have access to the master Chemical Hygiene Plan and are trained in reading and locating the Material Safety Data Sheets (MSDS). Personnel required to manage hazardous waste are trained in safety, legal, and shipping aspects of waste management.

OSHA
Occupational Safety and Health Administration

MSDS
Material Safety Data Sheets

6

Laboratory Facilities

6.1 Facility

The laboratory facility has an area of 25,000 square feet with over 800 linear feet of laboratory benching and over 120 linear feet of fume hood space. The ASC floor plan is provided on Figure 6-1. The laboratory is divided into the following areas: administration and offices; GAC; metals sample preparation; organic sample preparation; metals analysis; HPLC; GC and GC/MS analysis for volatile and semivolatile compounds; and other laboratory support functions. The sample management office is approximately 1,500 square feet and has a 390-square-foot walk-in cooler. A separate ventilated room is used to store laboratory waste prior to pickup by a licensed waste hauler.

Off-site facilities are used for material storage and file archiving. The warehouse is located behind the laboratory facility and the corporate headquarters is located within four miles of the laboratory facility.

Deionized water from a recirculating reverse osmosis/ion exchange system is piped to all laboratory benches. Gases for laboratory instruments and operations are distributed from a central source to the required areas through piping and manifolds. Laboratory benches have chemical resistant work tops and surface bench-level exhaust grills to remove vapors.

6.2 Security Systems and Environmental Controls

ASC provides a secure environment for our employees, guests, clients, samples, and analytical data.

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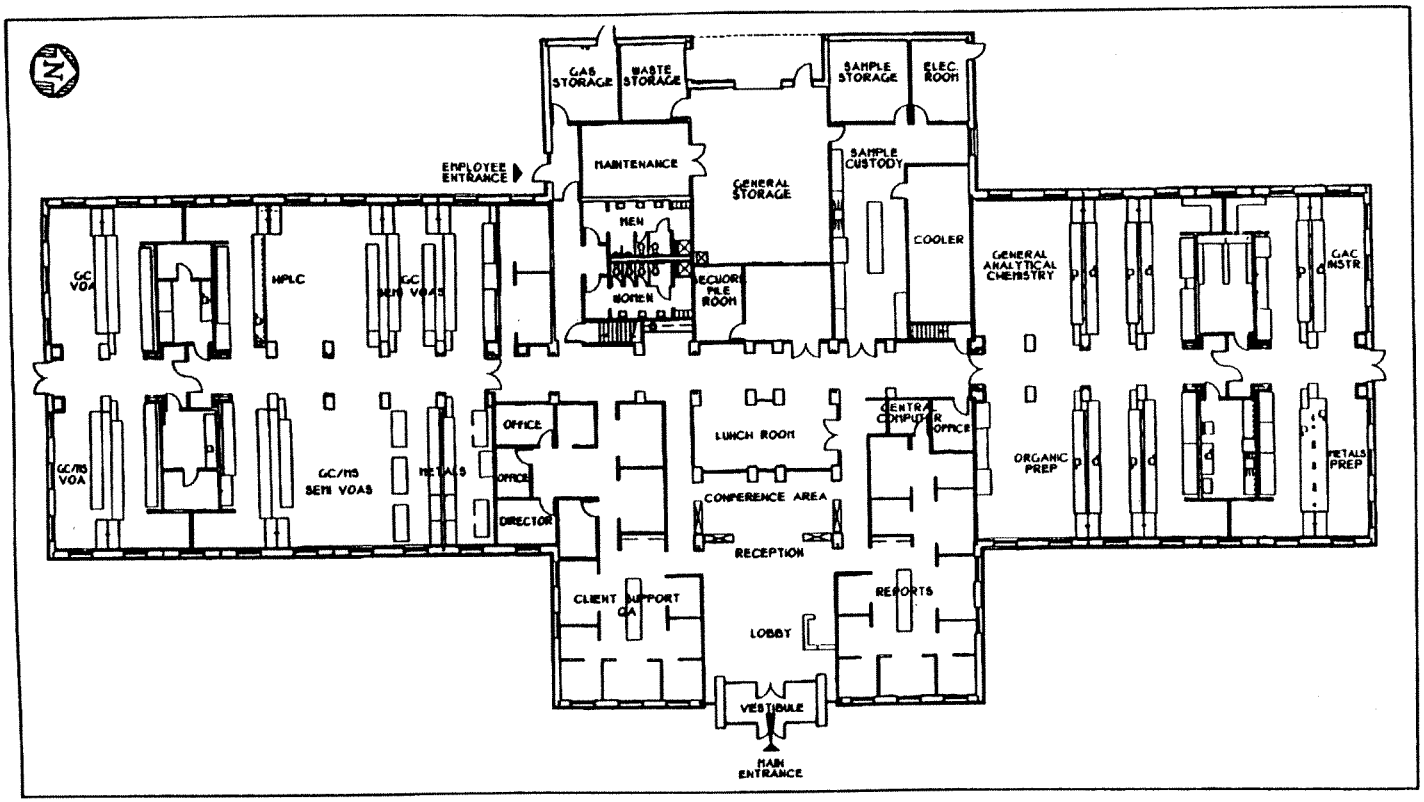
DELIVERY ENTRANCE ↓



EMERGENCY→ DOOR

←EMERGENCY DOOR

6.2.1 Access

To ensure security, all exterior doors remain locked unless directly monitored by personnel. Access to the laboratory is limited to employees and contractors. Visitors not under signed contract to E & E are required to sign the Visitor Log and must be accompanied by an E & E employee.

The sample management area is the defined high security area. Entry into this area is controlled by identification card activated locks on the internal and external entry doors. Personnel not assigned to the high security area must be accompanied by an employee assigned to the area.

The sample reports storage area is defined as a limited access area. Individuals requiring access must get a key from a designated staff member.

6.2.2 Security

All doors are locked after normal business hours and require a card for entry (SOP A.7). The security alarm at ASC continuously monitors for smoke, extreme fire-related heat, cold room temperatures, motion, and door contacts. When the alarm is activated, the appropriate emergency response offices are notified. An emergency contact list is provided to the local emergency offices for contact in case of an emergency.

6.2.3 Archives

Access to the archive facilities is limited to the QA Coordinator, Document Control Officer, or personnel designated by them (SOP A.27). The archives include all ASC copies of analytical reports, raw data, inactive logbooks, and other data that facilitate traceability of analytical results that are typically older than one year. Materials housed in the archives are labeled with identification numbers to ensure retrievability. Electronic storage tapes and disks are maintained by the system administrator.

6.2.4 Environmental Control

Lighting, noise, and other environmental factors at ASC are maintained at appropriate levels. Heating, ventilation, and air-conditioning systems satisfy the needs of the testing being performed. The laboratory building is designed to ensure constant temperature control for analytical equipment. The building has three completely separate air-handling systems to minimize airborne contaminants that may jeopardize sample integrity. Laboratory activities that involve use of large quantities of organic sol-

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vents or inorganic acids are conducted in the northern end of the building, well away from instrument areas.

The working and storage environments are maintained in a safe and appropriate manner. Sample and standard storage areas are monitored and temperature controlled. Separate storage areas are designated for samples, standards, and other identified sources of potential cross-contamination. Sample storage areas for volatiles are separated from other storage areas and monitored for any cross-contamination effects.

Safety measures and equipment which protect personnel and property from injury or illness include: fume hoods, fire extinguishers, fire blankets, alarm systems, safety training, protective clothing, emergency showers, eyewashes, and spill control kits.

6.3 Chemical Storage

All chemicals are stored in appropriate cabinets and are properly disposed of when necessary. All flammable solvents are kept in OSHA and National Fire Protection Association (NFPA)-approved areas. Acids are stored in OSHA-approved areas.

NFPA
National Fire Protection Association

All chemicals, samples, and wastes are accumulated in satellite areas in the laboratory as specified in the waste disposal SOP A.10 and Chemical Hygiene Plan. The samples and wastes are then transferred to the waste storage area. This area is specially designed and separately ventilated to accommodate waste storage prior to proper disposal.

An authorized waste carrier is contracted to pick up waste as needed and dispose of it, in accordance with all regulatory requirements. Post-analysis disposition of samples is dependent upon project-specific requests. Remaining sample material may be returned to the client, safely discarded, or archived for a specific period of time prior to disposal. Section 10.5 defines the specific requirements for sample disposal and other waste disposal operations.

7

Equipment and Materials

Accurate analysis consists of setting up proper instrument operating conditions, calibrating instruments, monitoring instrument performance tests, analyzing prepared samples, and collecting data from the analyses. Instrumental analysis procedures, frequencies, and acceptance criteria are described in the test method SOP. The basis for performance criteria, instrument conditions, and the steps of each procedure are derived from EPA or client-specified reference methods.

7.1 Instruments

Analytical instrumentation at ASC includes:

GC
gas chromatograph

GC/MS
gas chromatograph/
mass spectrometer

IC
ion chromatograph

AA
absorption
spectrophotometer

- Gas chromatographs (GC);
- Gas chromatographs/mass spectrometers (GC/MS);
- High performance liquid chromatographs (HPLC);
- Ion chromatographs (IC);
- ICP-atomic emission spectrophotometers;
- Atomic absorption spectrophotometers (AA);
- UV/visible and infrared spectrophotometers; and
- Flow injection analyzers.

ASC's major equipment is summarized in Table D-1 in Appendix D. All instrumentation is uniquely identified by an instrument code and serial number.

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UPS
uniterruptible power source

The laboratory has a large uninterruptible power source (UPS) capable of sustaining power to the instruments and computers for up to three hours in the event of a power failure. The UPS prevents problems caused by fluctuations in the power source.

Regular maintenance procedures and frequency for all equipment are defined in the test method SOP. A stock of spare parts and consumables are maintained for all analytical equipment. Backup instrumentation for most analytical equipment is available in the event of major equipment failure. The laboratory has a large uninterruptible power source (UPS) capable of sustaining power to the instruments and computers for up to three hours in the event of a power failure. The UPS prevents problems caused by fluctuations in the power source.

Service contracts are maintained for major instrumentation. Service technicians may be called for on-site service of major equipment if routine maintenance operations do not eliminate any problems. Procedures and schedules for preventive maintenance are available in the test method SOPs. All preventative and corrective instrument maintenance is recorded in the maintenance logbook assigned to the equipment. Records of external service are stored in files by the Section Supervisor. Instruments will be tagged as out-of-service until maintenance procedures are completed.

After instrument maintenance or repair, the instruments must be successfully recalibrated following the method SOP. Laboratory personnel must demonstrate acceptable QC performance on the instrument before sample analysis is conducted with it.

7.2 Instrument Operating Conditions

The reference methods for a given analysis define the instrument operating conditions. In many of the reference methods, a range or general guidance for the operating conditions is defined. The operating conditions may be modified to clarify the reference methods or improve the quality of the results. In all cases where the method is modified, the performance criteria from the reference method must be within the defined acceptance limits. Acceptable modifications to the operating conditions are stated in the method SOP. Any change in the operating conditions not stated in the SOP is documented on a corrective action form. When a change in the operating condition is proven to improve performance for all matrices, the method SOP is revised.

7.3 Computer Systems

E & E maintains an extensive network of computer systems centralized in the corporate headquarters building. ASC's domain includes an inte-

grated network of standalone instrument computers, instrument data systems, servers, and personal computers.

The GC/MS data system is a UNIX-based Hewlett Packard (HP) 9000/735 system running Target Chromatographic Analysis software by Thru-put. The central server (i.e., PA-RISC, 208 megabyte (MB) random access memory (RAM) and 8 gigabytes (GB) disk space) is supported with PC stations and three HP1000 minicomputers. The GC and HPLC data system is Perkin Elmer Turbochrom Client-server Chromatographic Analysis software running on an HP Netserver LH Pro/200 with 64 MB RAM and 4.2 GB of disk space. The GC data system supports seven PC stations. The PE Optima ICP stand-alone Winlab data system runs on a Windows 95 PC station. All other instruments operate standalone data systems.

HP
Hewlett Packard

MB
megabyte

RAM
random access memory

GB
gigabytes

LIMS
Laboratory Information Management System

DBMS
Database Management System

CLP
Contract Laboratory Program

CAD

Developed in Microsoft Access®, the Laboratory Information Management System (LIMS) Omega II is a Microsoft Windows™ based, multi-user Database Management System (DBMS). This system operates under a modified client/server model. The front-end application runs on a mix of Windows NT™ Workstation and Windows 95™ desktop PCs, the backend database is stored on a Windows NT™ Server with dual Pentium Pro dual processors. Omega II provides full-feature, flexible laboratory information management including sample management, work assignment, sample tracking, data reporting, QA, financial planning, and resource allocation. For Contract Laboratory Program (CLP) reporting, ASC runs Target software for organics analysis and Ward software for inorganic reporting on stand-alone PC stations.

In addition to the LIMS, all ASC support software is Microsoft Office 97 running in Windows 95. All the systems are linked to over 45 PCs and 20 printers.

E & E's Corporate Computer Support Center, publications network, graphics, and computer-aided design (CAD) departments also support ASC. Through these groups, ASC has complete access to the programming and office support software required by clients.

7.4 Sampling Materials

While ASC does not routinely perform sampling, ASC does provide clients with materials needed for sampling upon request. Materials are

assembled to meet project-specific requests. The materials provided may include the bottles, blanks, custody form(s), preservatives, and cooler(s) to be used during sample collection. Tables 7-1 and 7-2 list standard containers provided by

Table 7-1 Sample Containers and Volumes for Soil Samples

Type of Analysis	Type and Size of Container	Number of Containers and Sample Volume (per sample)	Minimum Volume per Test (grams)
Purgeable (volatile) organics	40-ml glass VOA vial with teflon-backed septum or 4-oz. VOA glass jar with Teflon-lined cap	Two 40 mL or one 4 oz; fill completely, minimal air space	Fill container
Extractable organics	8-oz. glass jar with Teflon-lined cap	One; fill completely	50
Metals	8-oz. glass jar with Teflon-lined cap	One; fill completely	25
General analytical tests	8-oz. glass jar with Teflon-lined cap	One; fill completely	15

Notes:

All sample bottles are prepared in accordance with generally accepted bottle-washing procedures.

Several types of analyses may be performed on samples from the same container, depending on the sample density and minimum volume requirements.

When project-specific laboratory QC samples are required, a double volume of the sample should be collected and labeled as MS/MSD, or duplicate, etc. Minimum volumes do not include QC samples.

Sample preservation requirements and holding times are presented in Appendix E.

Key:

VOA = Volatile organic analysis.

ASC. The sample containers or bottles for sample collection are purchased either precleaned, with a certificate of the test results, or precleaned but without a certificate for which ASC then tests lots for contamination. The certificates of analysis are maintained by shipping personnel. The lot numbers, expiration date, and amount of reagent used for sample preservation are recorded and tracked by shipment. Field and trip blanks are preserved with the same reagents as the samples and are tracked by lot number.

Materials used for sampling are stored in the warehouse building adjacent to the laboratory building. Reagents for preservation are stored in designated areas to avoid contamination during storage. Trip blanks are

prepared as needed in the laboratory volatiles area. All glass containers are packaged to prevent breakage. The containers are placed in plastic coolers, filled with additional packing material, and sealed with tape for shipping.

Table 7-2 Sample Containers and Volumes for Water Samples

Type of Analysis	Type and Size of Container	Number of Containers and Sample Volume (per sample)	Minimum Volume per Test (mL)
Purgeable (volatile) organics	40-ml glass vial with Teflon-backed septums	Two; fill completely; no air space	40
Extractable organics	0.5-gallon or 1-liter amber glass bottle with Teflon-lined caps	One 0.5-gallon or 1-liter per test; fill completely	1,000
Metals	1-liter polyethylene bottle with polyethylene-lined cap	One; fill 7/8 full	600
Cyanide	1-liter polyethylene bottle with polyethylene-lined cap	One; fill 7/8 full	50
TOC	125-mil polyethylene bottle	One; fill completely	50
Sulfide	1-liter polyethylene bottle	One; fill completely, no air space	200
Acidity, alkalinity, pH, dissolved solids, suspended solids	1-liter polyethylene bottle with polyethylene-lined cap (multiple analyses) all from one bottle)	One; fill completely	1,000
Hardness	1-liter polyethylene bottle with polyethylene-lined cap	Can take out of metals analysis bottles	100
Chloride	1-liter polyethylene bottle with polyethylene-lined cap	One; fill completely	100
Sulfate	1-liter polyethylene bottle with polyethylene-lined cap	One; fill completely	100
Nitrate	1-liter polyethylene bottle	One; fill completely	100
Nitrite	1-liter polyethylene bottle	One; fill completely	100
Total phenol	1-liter amber glass bottle	One; fill completely	500
TOX	1-liter amber glass bottle	One; fill completely, no air space	200
Ammonia-nitrogen	1-liter polyethylene bottle with polyethylene-lined cap	One; fill completely	500
BOD_5	1-liter polyethylene bottle with polyethylene-lined cap	One; fill completely	100
COD	1 125-ml polyethylene bottle with polyethylene-lined cap	One; fill completely	100
Color	1-liter polyethylene bottle with polyethylene-lined cap	One; fill completely	100
Fluoride	1-liter polyethylene bottle with polyethylene-lined cap	One; fill completely	100

Table 7-2 Sample Containers and Volumes for Water Samples

Type of Analysis	Type and Size of Container	Number of Containers and Sample Volume (per sample)	Minimum Volume per Test (mL)
TKN	1-liter polyethylene bottle with polyethylene-lined cap	One; fill completely	100
Hexavalent chromium	One 125-ml polyethylene bottle with polyethylene-lined cap	One; fill completely	200
Oil and Grease	1-liter amber glass bottle	One; fill completely	1,000
TRPH	1-liter amber glass bottle	One; fill completely	1,000
Orthophosphate	1-liter polyethylene bottle with polyethylene-lined cap	One; fill completely	50

Notes:

All sample bottles are prepared in accordance with generally acceptable bottle-washing procedures.

Several types of analyses may be performed on samples from the same container depending on the minimum sample volume and preservation requirements.

When project-specific laboratory quality control samples are required, a double volume of the sample should be collected and labeled as a matrix spike/matrix spike duplicate, duplicate, etc. Minimum volumes do not include QC samples.

Sample preservation requirements and holding times are presented in Appendix E.

Key:

BOD_5	=	Five-day biochemical oxygen demand.
COD	=	Chemical oxygen demand.
TKN	=	Total Kjeldahl nitrogen.
TOC	=	Total organic carbon.
TOX	=	Total organic halogen.
TRPH	=	Total recoverable petroleum hydrocarbons.

Sample containers are purchased from vendors using EPA-approved glass cleaning procedures. Vendors are qualified approximately every one to two years. The vendor selection process includes a pre-selection review of qualifications, preparation of a request for quotation, and receipt and review of proposals. Competitive bidders are required to send a series of bottles for testing. ASC tests a subset by batch of containers to ensure product quality prior to awarding contracts. Once a vendor is awarded the contract, the vendor secures large lots of bottles required by the laboratory. Bottles are shipped to ASC or directly to sampling sites from the lots allocated to E & E.

7.5 Glassware and Glassware Cleaning

Glassware subjected to heat or chemicals are of borosilicate glass. The laboratory also possesses Teflon™ ware for certain tests. Volumetric glassware is Class A.

All glassware used for the preparation of samples is cleaned as described in the SOP for glassware cleaning, A.31, defined for each laboratory preparation area. These procedures include pre-rinses and soapy water washes. The pre-rinse may be solvent, water, or acid solution, depending on the analysis. Glassware is washed with laboratory grade detergents, followed by multiple ASTM Type II water rinses. Cleaning procedures required by specific methods may include solvent, acid, or other rinses. The clean glassware is air dried unless other drying is recommended by the reference test method. The clean glassware is stored in the laboratory area as specified in the reference method or on designated shelves.

Cleaning areas and materials are segregated to avoid cross-contamination. Glassware used for high concentration analyses is kept segregated from glassware used for low concentration analyses. Dedicated glassware is used for volatiles, extractable organic compounds, GAC, and metals analyses.

8

Standards and Calibration

During the analytical process, a variety of measurements are required including volume, weight, concentration, pH, and temperature measurements. ASC follows established practices as documented in SOPs that ensure the calibration and verification of all equipment and the traceability of standards to national standards of measurement (where available).

8.1 Chemical Standards

The procurement, preparation, handling, storage, and documentation of chemical standards is critical to the analytical process. It is through these chemical standards that reported analyte measurements in samples are traceable to reference values. ASC uses only the highest quality chemicals as reference materials. Whenever possible, standard solutions must be traceable to national standards, such as National Institute of Standards and Technology (NIST) or NELAP-certified providers.

NIST
National Institute of Standards and Technology

Preparation of the chemical standards is documented in the method SOP. The documentation requirements for ensuring traceability are defined in the reagent and standard traceability SOP A.28. The procedures describe the management of these analytical standards and the reagents used in preparing the standards. All analysts are trained in the reagent and traceability SOP and are responsible for implementing the procedures.

8.1.1 Sources of Standards, Traceability, and Verification

ASC purchases chemical standards from approved suppliers. Information about the standards along with any client requirements are recorded in the pertinent standards logbook. The analyst receiving a chemical standard shipment verifies that the information on the standard label is consistent with that on the supplier's certificate or other paperwork. Information about the standard is recorded in a standards logbook. Codes that unambiguously identify the supplier materials and all derived

Primary Standard - A neat or concentrated standard received from a supplier. A primary standard is traceable to NIST or verified as equivalent to a certified reference material.

Secondary Standard - A neat or concentrated standard received from a supplier or prepared at a laboratory. The solution's concentration is determined against a primary standard..

Stock Standard - A solution of a standard at a high concentration, used to prepare working standards. These may be prepared in-house or received from a supplier. This standard may be a primary or secondary standard.

Working Standard - A solution of one or more standards or stock standards containing the analyte(s) of interest. The standard is prepared at the concentration defined in the appropriate method SOPs. The working standard is used to directly prepare instrument calibration or sample fortification. These may be prepared in-house from primary or stock standards, or pur-

preparations are also used to trace standard solutions. Non-certified materials are verified against certified reference standards, when certified reference materials are available.

8.1.2 Types of Standards

Analytical methodologies define a variety of QC samples which are used by the laboratory and include: surrogate and matrix spikes, laboratory control samples, internal standards, other QC check standards, and calibration standards. The composition and concentration of these QC samples must conform to method specifications. Standards used to prepare QC samples are categorized at ASC according to primary standard, secondary standard, stock standard, and working standard.

8.1.3 Preparation of Standards

Standard solutions are prepared by experienced analysts and documented in the appropriate standards logbooks. How a standard solution is prepared depends upon the amount required, the concentration required, and the solution's intended application. New standards are prepared as needed.

All standards are assigned an expiration date. If provided, the supplier's assigned expiration date is used for standard or stock standards, otherwise, the expiration date is assigned based on the supplier's date of preparation and the known stability of the analyte. Analytes known to be highly volatile or subject to rapid degradation or reaction are assigned shorter expiration dates. A standard mixture is assigned an expiration date no later than that of the shortest-lived components.

The expiration date is a latest use date. Standards are examined for deterioration and evaluated for their contribution to analytical problems during each analytical sequence. The QC parameters for each analysis are designed to monitor the working standards. Standard solutions showing signs of deterioration or with uncertain integrity are replaced immediately, prior to their expiration date.

Analyte or standard components common to calibration solutions and associated QC sample(s) may be from the same source or an independent source. QC samples such as the initial calibration verification (ICV) are typically prepared with reference material that is independent

ICV
initial calibration verification

of the associated calibration standards. Continuing calibration verifications are typically the same source as the calibrations standards.

8.1.4 Inventory and Storage

Documentation for all standards is carefully kept in relevant standards logbooks. Certificates from the manufacturer are taped in the bound logbook or placed in a ring binder. Each certificate includes the code number for the standard. The manner of storage for a standard is determined by its type, expiration date, shelf life, or manufacturer's specifications. All light sensitive standards are stored in amber vials or bottles in designated refrigerators, freezers, or chemical shelves. Analytical standards are never stored together with samples, extracts, or digestates.

8.2 Calibration Procedures and Frequencies

All equipment used for measurement and testing meets the specific requirements of pertinent analytical methods and applicable certification agencies. Calibration procedures and frequencies for specific types of equipment are briefly summarized below. Section 8.3 describes major instruments such as GC/MSs, GCs, HPLCs, and ICPs. Section 8.4 describes support equipment, such as thermometers, analytical balances, pH meters, autopipettors, and volumetric glassware. The specific calibration procedure and frequency are specified in separate SOPs for support equipment or the SOP for the test method using the instrument. Calibration criteria are listed in the method SOP. Except in the rarest of circumstances, the ASC does not report data for any samples unless the performance criteria for initial calibrations are satisfied. If it is necessary to report data, it is appropriately qualified or discussed in the report narrative after approval from the project manager.

Most instrumentation software indicates for the calibration curve the test method, date, time, analyte, and standard concentrations used and corresponding instrument responses. The time stamp is set to the network time by the System Administrator. Access is password protected. When prepared manually, the analyst records these. The software reports the equation of the calibration curve and the correlation coefficient. When the curve is recorded manually, the analyst records the equation of curve and the correlation coefficient.

The instrumental performance requirements of the published methods and/or manufacturer specifications are followed unless otherwise specified for a project. Other performance tests may also be executed to

further demonstrate proper functioning of instrumentation. Any instrument not functioning properly will be tagged out-of-service until proper operation is restored. SOPs for the operation of measurement equipment on the test method contain the following information, as applicable:

- Equipment used in the procedure;
- Calibration procedure, including all formulas and calculations;
- Acceptance criteria for the calibrations, including the accuracy and precision required;
- Corrective action for failed acceptance criteria, including assessment of previous calibration results;
- List of calibration standards;
- Frequency with which the equipment is calibrated, adjusted, and checked; and
- Maintenance of the equipment and record keeping to track performance before and after maintenance.

8.3 Instrumentation

8.3.1 Gas Chromatography/Mass Spectrometry

The GC/MS is hardware tuned prior to performing the initial and continuing calibrations and all sample analyses. Results are required to meet the peak ratio specifications of the analytical methods. For volatiles analyses, bromofluorobenzene (BFB) is used, and for semivolatliles analyses, decafluorotriphenylphosphine (DFTPP) is used.

BFB
bromofluorobenzene

DFTPP
decafluorotriphenylphosphin
e

The GC/MS is calibrated using internal standard techniques by analyzing a set of five or more initial calibration solutions, using procedures specified in the method and SOP.

Calibration criteria are listed in the method SOP. The initial calibration is verified by analysis of a second source standard.

Each day the initial calibration is verified through the analysis of a continuing calibration standard at the start of each clock sequence. The

concentration of the continuing calibration standard is dependent on the requirements of the specific method and SOP.

8.3.2 Gas Chromatography and High Performance Liquid Chromatography

External standard calibration is utilized for analysis by GC and HPLC. The method-specified number of calibration standards are used (e.g., generally 5 points) and specified in the SOP. For multiple response components (e.g., polychlorinated biphenyls, toxaphene) the quantitation consists of an average of the quantitated values for selected peaks. Calibration criteria are listed in the method SOP. The initial calibration is verified by analysis of a second source standard (ICV).

Each day the initial calibration is verified through the analysis of a continuing calibration standard (CCV) at the beginning of the analytical sequence at least every 20 samples. The concentration of the continuing calibration standard is dependent on the requirements of the specific method and SOP.

Other standard checks may be required for a specified reference method. Instrument performance checks specified in the reference method must be performed and within the acceptance limits stated in the reference method.

8.3.3 Atomic Absorption Spectrophotometry

An initial calibration is performed daily with standards as specified in the SOP. The initial calibration is verified at the beginning of the run sequence and every 10 samples. Continuing calibration blanks are run at the same frequency.

The continuing calibration must be within the range specified in the analytical method SOP utilized. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within the specified range of the true value (generally 10% of the expected value).

8.3.4 Inductively Coupled Plasma Emission Spectrophotometry

Initial calibration is performed daily and continuing calibrations are performed every 10 samples. The number of initial calibration standards used depends on the method employed. Samples are not analyzed until

an initial calibration verification has been performed and is found to be within specified range of the true value, depending on the method. Continuing calibration is required to meet the criteria of the analytical method.

8.4 Support Equipment

All equipment is calibrated according to the SOP used for the analysis. The most common support equipment used in the laboratory are thermometers, balances, pH meters, and conductivity meters. This section provides a general summary of the procedure used for calibration and measurement controls. Other equipment calibration is defined in the test method SOP.

8.4.1 Thermometers

Laboratory thermometers are routinely checked for accuracy against certified, NIST-traceable thermometers following the thermometer calibration procedure. These calibrations are performed annually (SOP A.38). Correction factors derived from the annual calibrations are applied to temperature readings. NIST-traceable thermometers are professionally calibrated and recertified every five years. Records of thermometer calibrations are retained in a logbook in the general analytical chemistry laboratory area. All thermometers are tagged with the thermometer ID number, correction factor to be applied to the measurement, and the expiration date of the calibration check. Thermometers past the calibration expiration date or thermometers not reading properly are not used. Replacement thermometers are calibrated, and the maintenance logbook is updated when a change in the thermometer is required due to breakage, damage, or expired calibration.

8.4.2 Balances

Calibration checks are performed for each day of use, for each balance. The calibration consists of a minimum of two weights which bracket the weight to be measured. The balance calibration procedure defines the acceptance criteria and performance criteria for the various balances used in the laboratory. Calibration weight measurements must meet the acceptance criteria listed in the SOP A.19 and balance calibration logbook. Each balance is serviced and calibrated by a certified professional at least annually. The accuracy of the calibration weights used by ASC is verified each year by an accredited calibration service. Balances are labeled with the balance number, date of service, and the expiration date for the annual service check. The balance number used

for any measurements requiring traceability is recorded with the measurement data. Balances with expired calibrations or that fail the weight check are not used.

8.4.3 pH Meters

Each day pH meters are calibrated prior to use. The meter is calibrated following the procedure for pH analysis (SOP GAC.34). Records of the calibration are documented in an instrument logbook or in the raw data for the analysis being performed. At least two buffer solutions that bracket the measurement range of the analysis are used for calibration. A check standard is used at the end of the analysis run to verify meter stability.

8.4.4 Conductivity Meters

KCl
potassium chloride

A five-point calibration curve using potassium chloride (KCl) solutions is analyzed annually. The calibration curve is used to determine the cell constant for the meter. The cell constant from the annual calibration curve is compared to the acceptance criteria defined by the manufacturer. If performance is unacceptable, the cell is cleaned and rechecked. The cell is not used until satisfactory performance is achieved.

A single KCl standard solution is used as a check standard each day the meter is used. The meter is labeled with the expiration date for the annual calibration.

9

Test Methods and SOPs

ASC is a full service analytical laboratory. Products include the preparation and analysis of a wide variety of sample matrices using standard, approved methods. In addition, ASC develops and validates non-routine analytical methods. ASC's standard tests are listed in Table E-1 in Appendix E. ASC's procedures for validation of new methods are described below.

9.1 Routine Analytical Methods

ASC utilizes a variety of in-house analytical methods that are based on the most recent versions of EPA or other published methods. In some cases, minor modifications of the reference methodology may be employed. Such modifications are listed in the corresponding SOP and must be validated prior to implementation. Accuracy, precision, and sensitivity must be within the acceptance criteria of the reference method. Compounds, elements, or parameters to be measured are chosen from the reference methods. Additional compounds, elements, or parameters may be added based on client or project specific requests. Reference methods are utilized from the latest version of the following references:

- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, EPA SW-846;
- *Standard Methods for the Examination of Water and Wastewater* (American Public Health Association [APHA]/American Water Works Association [AWWA]);
- *Methods for Chemical Analysis of Water and Wastewater* (EPA);
- Wastewater testing references cited in 40 Code of Federal Regulations (CFR) Part 136;

APHA
American Public Health Association

AWWA
American Water Works Association

CFR
Code of Federal Regulations

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- EPA Contract Laboratory Program Statements of Work for Organic and Inorganic Analyses;
- Drinking water testing references cited in 40 CFR Part 141;
- ASTM guidelines; and
- Specialty testing references cited by state regulatory programs.

The test methodologies are listed in Table E-1. An inventory of the ASC SOPs is provided in Appendix F.

SOPs are written for each analytical method as described in the SOP on SOPs. Analytical SOPs include a method summary; health and safety concerns; references; interferences; instrumentation and equipment; reagents and materials; preservation and container handling; a detailed description of the procedure including all calculations; data reduction and reporting; QA/QC procedures; and sample disposal. Any special client requirements are listed in a separate section. SOPs are developed, reviewed, and revised according to document control procedures described in Section 11. All SOPs must reflect actual laboratory practice and are used as a training tool for new and existing analysts. SOPs are reviewed annually or when the method is revised. All analysts receive training on the major revisions prior to the actual implementation date.

9.2 Target Compounds

Most of the organic analytical methods can be used to analyze multiple parameters that are either listed in the method or considered potentially detectable by the analytical technique. The target compounds for the method depend on the specific application or project. For each method, the laboratory maintains a standard target compound list as identified in the individual method SOPs. The standard compound list is made up of the compounds that are analyzed routinely, included in all standards, and used to generate internal control limits and method performance data. The laboratory may add or delete compounds from the method and will generate the QC data for the compound as required for the specific application or project. In some cases, the method SOPs list compounds that ASC has historical data demonstrating the use of the method for that compound.

9.3 Non-routine Analytical Methods

IPR
Initial Precision and Recovery

The process of development of non-routine analytical methods includes a literature search and review, development and approval of an experimental approach, testing of analytical techniques, and generation of method performance data such as an MDL or Initial Precision and Recovery (IPR) studies. Method development studies are conducted under Good Laboratory Practice procedures. Methods are documented in an SOP.

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Sample Management

10.1 Sample Receipt and Chain-of-Custody

The sample custodian or designee receives all samples. A work order number is assigned to each shipment of samples received from a client. In-house records for a new work order are initiated with a cooler receipt form. The sample custody form is signed by the client and sample custodian when the samples are received at the laboratory. Samples received via overnight courier are signed on the bill of lading. The bill of lading, cooler receipt form, and the sample receipt log are completed for samples delivered by courier (see examples in Appendix B).

The shipping containers, their contents, and accompanying client documentation are examined by the sample custodian. Information about the presence and condition of custody seals, the state of preservation of the samples, and other required information is noted on the cooler receipt form and/or package receipt log (SOP SM.1, SM.2). The USEPA form DC-1 is used for USEPA CLP samples (SOP SM.11). The cooler temperature, along with the presence or absence of ice, is recorded on the cooler receipt form and chain-of-custody (COC), if applicable. ASC recommends clients include a temperature blank in all coolers. Any discrepancies in documentation or problems with sample condition are noted and immediately brought to the attention of the client via the project manager notification form. The project manager provides the sample custodian with instructions on the processing of samples that are incomplete or missing required information.

COC
chain-of-custody

The sample custodian logs the samples into the LIMS, and a label for each container is printed. The custodian attaches each barcode label to the appropriate sample container. The following information is recorded for tracking samples: laboratory sample ID, client sample ID, sample matrix, and storage location. The sample receipt and handling SOPs describe procedures for sample receipt and log-in, COC, and handling of sample shipment containers provided by clients. All log-in

information is reviewed by a project manager before a sample can be released for analysis.

10.1.1 Sample Acceptance Policy

ASC only accepts samples that are appropriate for the requested tests, that are accompanied by sufficient documentation, and that pose no abnormal health and safety threats to facilities or employees.

Samples are appropriate for the requested tests if:

- they are in the proper containers,
- the matrix is compatible with the test,
- there is sufficient volume,
- they are properly preserved, and
- they are within hold times.

Documentation is sufficient if:

- the sample is identified correctly;
- the sample location, date, time of collection, and sampler's name are noted;
- labeling is durable (water resistant);
- preservatives added are noted;
- sample type is noted;
- information is legible;
- a COC form or equivalent documentation is used; and
- container contents match the documentation.

A sample is acceptable from a health and safety perspective if:

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- it is not from a site with known radioactivity ≥2 times background (a counter may be used on suspect samples);
- it is not from a site that contains dioxin or furan compounds in concentrations which will cause health, safety, or disposal problems;
- it is not explosive, shock sensitive, air or water reactive; and
- the outside of the sample container is clean.

It is the responsibility of the sample management staff to note when samples are unacceptable. It is the responsibility of project managers to make final determinations regarding samples, to notify clients, and to make appropriate comments in laboratory reports.

10.2 Sample Preservation and Security

Samples are stored in a secure manner to ensure their integrity. Samples are stored at temperatures that meet the specifications of the methodology, regulatory agencies, and client. Refrigerator temperatures are maintained at required temperatures (2° - 6°C) and monitored regularly. Preservatives, temperatures, holding times, and container storage requirements are listed in Appendix E. QAPPs may list preservation requirements differing from the laboratory. This information is provided to the sample custodians for project-specific handling.

Chemical preservatives are normally added to sample containers in the field at the time of sampling. Acid or base preservation and temperature preservation are checked upon receipt with the exception of preservation for volatile organic compounds, which is checked at analysis. Any differences from laboratory or client-specific requirements are recorded on corrective action forms, and the client is contacted by the project manager.

Sample storage facilities are located within the sample management area which is secured by locked doors. Extracts and digestates are stored in designated freezer units or storage cabinets, respectively, in the laboratory analytical areas. Internal procedures and documentation pertaining to sample possession, removal from storage, and transfer are outlined in the sample custody procedure SOP. Samples are returned to the sample management area after the required sample portion is removed for analysis.

Precautions are taken to ensure that cross-contamination does not occur during sample storage. The volatile storage refrigerator is monitored weekly for volatile compounds via storage blanks. Storage blanks from the volatiles sample storage refrigerator are replaced weekly and the previous blanks removed and analyzed. The storage blank information helps ASC personnel assess potential cross-contamination in the sample storage refrigerator. Additional project-specific storage blanks are used as required.

Temperatures of cold storage areas are monitored using thermometers calibrated to NIST-traceable thermometers and recorded each working day. Min/max or recording thermometers are used to verify cold storage temperatures on non-working days. Walk-in coolers in the Sample Management area are monitored on off-hours electronically by a security company. Corrective action is taken as necessary when temperatures are not within the control criteria. All temperature records document which thermometer was used to obtain the measurement. Details regarding sample storage and preservation are provided in the sample log-in SOP SM.2 and SM.4, and in the SOP A.43.

10.3 Sample Preparation and Analysis

All sample preparation and analysis procedures employed at ASC are covered by appropriate SOPs. Samples are prepared and analyzed following the method specified by client requirements and the matrix to be analyzed. Most samples must be prepared and/or analyzed within a method-specified holding time after sampling (see Appendix E). Any exceptions to the specified holding times are subject to the corrective action process and noted in the case narrative.

Holding times listed in the applicable project-specific documents are followed as required. Samples are occasionally received after holding times have expired. A project manager notification form is completed whenever holding times are exceeded. The project manager notifies the client, and the client decides to continue with the analysis or suspend the testing.

The LIMS tracks holding times and applicable analytical methods and helps ensure that all method and client requirements are met.

10.4 Sample, Extract, and Digestates Archiving

The sample custodian and other authorized personnel are responsible for archiving and disposing of raw samples, extracts, and digestates. Raw and prepared samples are not archived or disposed of until all of the designated analyses are complete and resultant analytical data is sent to clients. Samples in storage are retained for at least 30 days after reporting of the results; any samples requiring longer storage are archived. Longer retainage is available on a project-specific basis.

Archive samples are placed in boxes, labeled with the project numbers and disposal date, and retained in a sample archive area for any additional length of time specified by the client prior to disposal. The archive samples may be stored in the refrigerator or at ambient temperature, as specified by the client. Archived samples are disposed of at any time after the predetermined date in accordance with all applicable rules, regulations, and SOPs. Clients are informed of sample disposal procedures and given an opportunity to request exceptions to the routine practices as part of the contract or task order.

10.5 Sample Disposal

It is the policy of ASC to dispose of all samples and waste in accordance with all applicable rules and regulations. The following lists the primary regulations regarding disposal.

RCRA
Resource Conservation and Recovery Act

HMTA
Hazardous Materials Transportation Act

DOT
Department of Transportation

OSH
Occupational Safety and Health Act

NYCRR
New York Code of Rules and Regulations

Resource Conservation and Recovery Act (RCRA): Regulations pertaining to the Resource Conservation and Recovery Act (RCRA) and its amending legislation are published in 40 CFR 260-299. The regulations pertaining to satellite accumulation are found in 40 CFR 262. Regulations for generators are found in 40 CFR 265. Solid and hazardous waste definitions are located in 40 CFR 260 and 261.

Hazardous Materials Transportation Act (HMTA): The U.S. Department of Transportation (DOT) promulgates regulations under the HMTA in 49 CFR. HMTA regulations cover all hazardous materials (including both hazardous substances and hazardous wastes) and all aspects of their packaging, preparation, identification, and segregation during transportation.

Occupational Safety and Health Act: The Occupational Safety and Health Act (OSH) authorizes OSHA to provide and enforce regulations governing worker safety. OSHA regulations are found in 29 CFR, with

many of the sections pertaining to the laboratory found in part 1910 including 1910.120 *Hazardous Waste Operations and Emergency Response*, 1910.1200 *Hazard Communication*, and 1910.1450 *Occupational Exposure to Hazardous Chemicals in Laboratories.*

New York State Requirements: The State of New York has primacy in the area of environmental enforcement under RCRA. The regulations governing generators and storage of hazardous waste are found in Title 6, Conservation, of the State of New York Code of Rules and Regulations (NYCRR).

10.6 Sample Return to Clients

When a client has requested the return of samples, the sample custodian prepares and ships the samples according to the same custody procedures used to originally send the samples, and following any client-specified requirements. Special packaging procedures are implemented to protect the samples during delivery. Packages are delivered by a commercial carrier whose procedures for protecting the samples are beyond the control of ASC. Clients are informed that a commercial carrier will deliver their samples.

10.7 Sample Loss, Damage, or Unsuitability

Samples or sample containers can be lost, damaged, or deemed unsuitable, for whatever reason, after initial receipt at ASC. Whenever this happens, the event is recorded by the observer on the project manager notification form. The problem is reported to the project manager who informs the client. Plans for disposition of the affected sample(s) or containers are agreed upon with the client, carried out, and recorded on a corrective action form.

11 Document Control and Records

11.1 Document Control

ASC controls all documents that directly impact data quality as specified in the document control SOP A.27. This includes, but is not limited to, the QAM, SOPs, checklists, and forms. Electronic copies of controlled documents are available to all laboratory personnel. Also a hard copy of each document is stamped in green as the control copy and maintained by the Document Control Officer. The document control form (see Appendix B for example) provides a hard copy record of all review and approval signatures, and applicable training and implementation dates.

Uncontrolled copies may be made from the control copy for training and operational review or for external use. The procedure for version document control allows for the retention of a previous version for historical information purposes and includes the status of each active or archived document. Archived documents are procedures not currently requested that may be required in the future or are of old procedures that have been revised. Each document and any subsequent revisions must be reviewed and approved by authorized personnel prior to issue.

Every document is assigned a unique identification number that is present on each page of the document and entered on a master list of documents. The revision number, approval date, and page number are also listed on each control copy. All version-controlled documents are assigned an implementation date to clearly indicate when a new version is applicable.

11.2 Records

The laboratory maintains an extensive series of records related to sample tracking, analytical results and support information. Records management encompasses document control, software management, ana-

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lytical reports, and the LIMS. The types of records the laboratory manages include SOPs, forms and checklists, letters and memos, project files, analytical reports, electronic media, QA files, LIMS data, accounting data, instrument maintenance and other logbooks, programs, spreadsheets, and electronic data deliverables. The general procedures for managing records are outlined in the document control SOP A.27, but additional records management information is presented in individual administrative, QA, reports, and computer SOPs. The following sections summarize policy and procedures on some of the major records retained at the laboratory. All records are required to unequivocally reproduce the analytical results reported and provide evidentiary COC. Records are maintained in retrieval format at the laboratory for at least five years. Drinking water records are maintained for at least 10 years.

11.2.1 Standard Operating Procedures

SOPs are documents detailing every repetitive or standard operation performed by the laboratory. The author of each SOP is generally the person most familiar with the topic being addressed. Each SOP is peer reviewed and authorized by management and the QA area prior to distribution and implementation.

SOPs are evaluated by laboratory supervisors and key technical personnel. The content of each SOP conforms with applicable requirements of analytical methods and certification agencies and is consistent with the good laboratory practice standards. Within these constraints, the content of an SOP may be customized to meet the needs of a particular area of the laboratory. The performance of laboratory operations is subject to audit for compliance with written SOPs. If an SOP is impractical, difficult to follow, or no longer meets laboratory needs, it is modified or replaced.

The process for developing new or revising existing SOPs is documented in the SOP on SOP Administration (QA.18). At a minimum, SOPs are reviewed annually to ensure they reflect current procedures. Revisions are made to SOPs as necessary to reflect changes in procedures. Interim changes may be made to operations based on the implementation of a corrective action, provided that the corrective action is authorized. Finally, all personnel involved in the process affected by the change must be trained in the SOP change and the revised SOP.

The QA Coordinator administers the SOP program, and the Document Control Officer is responsible for clerical preparation and distribution of new or revised SOPs, record keeping, and archiving of replaced and retired SOPs under his/her direction.

11.2.2 Laboratory Notebooks

The Document Control Officer issues all logbooks and assigns and records logbook numbers in numerical order (SOP A.27). Completed logbooks are returned to the Document Control Officer within one year of completion of use of the logbooks. The logbooks are archived in locked cabinets in the warehouse. The Document Control Officer records the logbook's name, inclusive dates of use, disposition (to warehouse), and date of final disposition in the master logbooks. Logbooks may be destroyed after five years unless specifically required by the client.

The Document Control Officer maintains a logbook of all personnel with their signature and initials as a reference (traceability purposes) for other laboratory logbooks.

11.2.3 Laboratory Data

Laboratory report records are maintained in a file to prove that adequate QC procedures are being implemented, accountability of the data is maintained, and traceability of analytical results is completed. Accountability means that reported data reflect the sample as it was received, that sample mix-up was avoided, and that the sample was found properly preserved upon receipt. Traceability means that reported data may be reconstructed at a later date. Through proper documentation, the laboratory is able to demonstrate to clients or government agencies that the data quality is what the laboratory reports it to be. Records contain sufficient information to permit the reconstruction of calibrations, sample preparations, and sample analyses. Raw data information includes method identification, sample identification, instrument identification, and reference to operating conditions, date of analysis, example calculations, and analyst signature.

11.2.4 Quality Assurance Records

Quality records at ASC are maintained to prove that the QA system is being effectively applied. Specific procedures for the identification, collection, indexing, filing, storage, maintenance, and disposition of various quality records at ASC are described in the document control SOP

A.27. All quality records are recorded in permanent (indelible) ink, legible, attributable to those personnel who created them, and protected so they are not adversely affected by an unsuitable environment. They are stored and maintained in a manner that facilitates rapid retrieval for a period of at least five years after completion. Project-specific quality records are available for evaluation by the client or a client representative during the five-year archive period.

11.2.5 Project Files

Project managers maintain project files for all projects with specific requirements that deviate from routine SOPs. Project files may include an SOW, proposal or quotation, contract, client communications, or permit/regulatory requirements. The project documentation provided to specify analytical and QA requirements may be in the form of a QAPP.

QAPPs provide an overview of the way a project, group of projects, or specific analytical program is to be conducted. A copy of all client QAPPs to be used by the laboratory are available in the QA area. Historical copies of outdated QAPPs are retained in the archives by the Document Control Officer to aid historical interpretation of the data.

11.2.6 Archives

The archives are contained in a separate building, locked at all times, with access limited to the QA Coordinator, Document Control Officer, and their designees. The person retrieving the records must sign and date a logbook held by the Document Control Officer. All materials removed from and returned to the archive building must be signed, dated, and remain intact.

State and federal agencies and clients have access to applicable archived documentation.

All magnetic and hard copies of data, calibrations, equipment maintenance records, calculations, records of original observations, final test results, and any other miscellaneous quality records directly associated with sample analyses are stored in the archives for a minimum of five years after completion of a project.

11.3 Quality Assurance Manual

The QA Coordinator is responsible for the publication and distribution of the QAM. The QAM is submitted to management for review and

authorization annually. As major changes are implemented in the QA system, the QAM is revised and submitted to management for authorization. The QAM is published and revised with the assistance of laboratory management.

The authorization signatures found on the authorization page signify management review and approval of the QAM. The authorization section is kept current to reflect any organizational changes affecting the authorizing positions. The revision number and date of each section is always the same as the revision number and date for the authorization section, indicating that all major revisions have passed through management review. Minor revisions are authorized by the QA Coordinator as specified in the revision table. The revision table is updated to reflect major or minor revisions to sections.

Distribution of the QAM is regulated through document control procedures. Controlled copies of the manual may be distributed to an individual, client, or certifying agency. Uncontrolled copies distinctly marked as "Uncontrolled Copies" may be issued to persons or organizations outside ASC. A distribution list is maintained for all controlled copies of the manual. Updates are provided to all parties listed on the distribution list. All other copies marked as "Uncontrolled Copies" are not subject to updates upon revision of the manual.

Appendices of the QAM are controlled separately from the major text of the document and are not subject to the same training requirements as the policies in the QAM. Resumes are not part of the QAM proper since personnel requirements are often client dependent and the list of designated personnel will vary. Resumes are sent with the QAM to clients as designated in the document control SOP. A.27.

11.4 Software Management

The goals of the software development methodology, existing system validations, and the change control system are to ensure that the software systems perform the required functions accurately, that the users understand how to use the system, and that auditors can assure themselves of the validity of the analytical methods. This in turn insures the delivery of accurate, timely analyses to the client.

The computer systems used at ASC are leased from E & E or purchased for a specific instrument system. ASC has a formal validation

program for its computer systems that ensures that data transmitted, reported, or manipulated by electronic means are correct and free from errors. The validation and verification program is divided into three areas.

First, new software is developed according to the computer SOP Comp.9 for software validation. New software includes the development of macros for spreadsheets and other tools using commercial software packages.

Second, requests for changes to software are identified through flaws in existing documentation or the need to improve system processes. Final implementation of the change is documented with the software. This documentation provides a complete record of all software and electronic data reporting problems.

Finally, system integrity is verified through programs for routine maintenance, protection from unauthorized access, and electronic verification. Routine maintenance, including system backups, is performed on a scheduled basis. The backup process, password system, and access protection are defined in the computer area SOPs. Electronic verification programs may be used to assure that commercially purchased software is performing at its original specifications. This includes virus checking of all network operations at least once per day. Documentation of all verification and maintenance operations are retained in the equipment logbook for the computer network.

12 Reports

12.1 Data Collection and Reduction

Data collection is performed by the analyst, and data reduction is performed by the analyst and data reporting personnel. Data collection and reduction procedures for specific types of analytical methods are described in the Method SOPs. These procedures vary depending on whether data are acquired manually or electronically.

12.1.1 Manually Acquired Data

Manually acquired data may be recorded on data sheets, logbooks, or entered directly into the computer system. All data are immediately recorded in permanent ink by the analyst, and each page is signed and dated. Corrections are made by drawing one line through the error, dating, initialing, and, where appropriate, noting reasons for the correction in the margin. Unused portions of logbook page intentionally left blank are marked with a large "Z," then signed and dated on the "Z." All logbooks are signed by a peer or supervisory reviewer.

Data sheets are standardized, preprinted forms which are subject to document control and bound in a book. Notebooks are bound, consecutively numbered, and subject to document control.

Either the analyst or data entry personnel enter all manually acquired data directly into LIMS for reporting. Hand-entered data are checked for transcription errors as part of the data review process. Data processed in the LIMS may be corrected for dilution factor, percent solids, and the correct significant figures.

12.1.2 Electronically Produced Data

Electronically produced data may consist of chromatograms, spectra, data printouts, and raw quantitation reports. The analyst is identified on raw data with signature there or in a run sequence logbook. Any alterations to the raw data hard copies and computer files are fully docu-

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mented and clearly attributable to the person making such alterations. All manual integrations are hard-copied for inclusion in full data packages, with area changes fully documented on the data printouts. No ambiguity is allowed in data system printouts with regard to correspondence between peaks on a chromatogram and analytes of interest. Computer-collected data is reduced to hard copy as soon as possible. The electronic files are safeguarded by a system of backup media to protect against loss of data and programs. Software used for data acquisition and quantitation reports is tested according to written procedures to assure that no data anomalies are present.

Electronically produced data are transferred directly to the LIMS through an instrument interface program, or the data may be hand entered from the instrument printout. All data are checked for accuracy and transcription errors as part of the data review process. Electronically produced data may be corrected for percent solids, dilutions, and significant figures in the LIMS.

12.2 Data Review

Data review is performed by the analyst, the peer reviewer, project manager, and QA Coordinator as defined in the data review SOP A.25. At each stage of the analytical process, data are reviewed for completeness, adherence to protocol requirements and project-specific requirements, and documented on appropriate checklists. Results are fully validated; possible compromises of data quality are evaluated; and deviations from protocol requirements are documented. To the greatest extent possible, computer programs are utilized for data reduction. Where manual data manipulation procedures are required, data review includes additional steps to check for transcription errors. If any nonconformances are noted during data review, corrective actions are initiated at the earliest possible opportunity and documented according to the corrective action procedure.

12.2.1 QC Review

The main purpose of the QC review is to determine the extent to which the method and final data meet established requirements. Checklists for each test method and project requirements reference the QC criteria to be reviewed along with the acceptance criteria. The review is conducted by the analyst and peer review analyst. The data review includes a 100% review of all QC elements. All nonconformances are documented as defined in the corrective action procedure. After

analyst and peer review, the project manager reviews the reported information for consistency with project requirements. All exceptions are noted in the case narrative for client review. Checklists or benchsheets are signed and dated to indicate that the QC review is complete.

12.2.2 QA Review

Data reviews are performed by the QA Coordinator or designee on a random sampling of ASC data reports. The goal is to perform an evaluation of approximately 10% of sample reports. A data report is carefully evaluated for technical, clerical, and administrative accuracy. Primary emphasis is placed on the ability of the data report to meet current SOP requirements. Data audits are comprehensive assessments of the data review process as defined in Section 4.4. Data audits are conducted annually and allow upper level management to identify opportunities for process improvement, evaluate the efficiency of the system, detect inadequate execution of QC procedures, detect potential system deficiencies, and recommend corrective actions.

12.2.3 Data Qualification and Validation

As part of the data review process, ASC may qualify data prior to reporting. For CLP or CLP-equivalent reports, data qualifiers consistent with the CLP SOW are used. For routine reports, the laboratory uses the following qualifiers:

- "ND" – Not detected at the reporting limit.
- "J" – Analyte detected below the reporting limit.
- "B" – Analyte detected in the associated method blank.
- "H" – Value exceeds Maximum Contaminant Level.
- "M" – Matrix spike recovery outside limits.
- "E" – Value above quantitation limit.
- "N" – The compound was present, but no confirmation analysis using a second column was performed.
- "C" – See an additional comment.

- "*" – Recovery outside limits.
- "R" – RPD outside limits.

After the laboratory releases the data, an E & E corporate or client data validation chemist may review the data package. The data validation chemist will evaluate the package for completeness, and the results against the project-specific DQOs and determine the usability of the results. The data validation chemist may add or remove qualifiers from the data as required for the project. All potential problems will be reported to the laboratory on a corrective action form to ensure appropriate follow-up and tracking.

12.3 Data Reporting

Data are reported by analytical method, target analytes identified, and the quantities present. The content of the final report is dependent on the project-specific requirements. At a minimum, all reports include a coversheet, a case narrative (if applicable to client), analytical results, signature of the responsible laboratory party, the test methods used, dates of sampling and analysis, data qualifiers, and any QC results associated with the samples.

Full data packages may be presented in the latest CLP format or equivalent. CLP format includes a case narrative, standard report forms, and all associated raw data. Customized data packages following RCRA program QC requirements are also prepared. The project-specific checklists for reporting are developed at the start of the project for full data packages if customized packages are substantially different than standard report forms.

Clients requiring electronic data deliverables (EDD) receive data in the electronic format standard developed by the ASC. If client requirements differ from the ASC standard formats, EDDs may be compiled based on the criteria defined at the start of the project. The project-specific acceptance criteria are placed in the electronic file and used to check the final reported data. All flags or data anomalies are presented in the case narrative. The data are retained through the laboratory network back-up process. Details regarding EDD generation are presented in the electronic reporting SOPs.

12.3.1 Data Package Delivery

Data packages are prepared for delivery by each laboratory and compiled in the respective lab project managers area according to the reporting SOP Rep.20 and related electronic reporting SOPs. Unless otherwise requested by the client, a copy of the data is shipped while the original is retained in the laboratory archive facility to guard against possible loss of original data. Reports are fully paginated prior to copying. The total number of pages in a report is identified. The method of delivery of the data package is specified in the project or contract. Should the shipped data package be lost or damaged during delivery, a copy will be prepared from the original as a replacement. Results also may be faxed or e-mailed per client requirements.

12.4 Corrections and Additions to Documentation

Additions or corrections to reports already issued are handled according to the following policy. The project manager can request an addition or correction in writing using a "Report Correction Request" form (SOP A.33). The form is given to the appropriate data reporting personnel. The requested change is made in a timely manner and may need to be internally verified with the analyst and peer reviewer and must be approved by the laboratory director, general manager, or QA coordinator. After the change is reviewed by the responsible parties, the reporting area issues the new report to the customer. The case narrative is changed to record the amended report and the reason for the change from the original. The amended report is stored with the original data package for a minimum of five years.

13 Contracting

13.1 Contract Review

For all analytical services provided by ASC, contract review is accomplished through the generation of a written quote or contract. A written quote is utilized for short-term contracts, usually consisting of one analytical project. A written contract is utilized for long-term contracts consisting of multiple analytical projects.

Project managers are responsible for implementing and documenting contract review. Client requirements, including special needs that are not documented in the current SOPs, are defined and documented in the written quote or contract. Section supervisors having expertise in specific analytical services are consulted to ensure that the laboratory can meet special requirements. If it is decided that the special requirements cannot be met, this is discussed with the client, and alternatives may be offered. Information about ASC's capacity and availability is made available to project managers on a regular basis to enable them to make informed decisions regarding contracted delivery times.

Long-term project requirements are added to the affected SOPs in the section "Client Specific Requirements." Short-term project requirements such as one-time task order requirements or purchase orders are communicated by the project manager to the appropriate personnel by the use of work order comment or project comment in the LIMS, project memo, or a data review checklist. This documentation contains the project-specific requirements used by the analyst, peer reviewer, and project reviewer to ensure that all the requirements are met.

13.2 Subcontracted Analyses

ASC has established procedures for subcontracting any analyses they cannot perform. The client may be referred directly to another laboratory if no part of the work is to be performed by ASC, or work may be

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subcontracted. Subcontracting follows corporate procedures and the laboratory SOP A.32 on subcontracting. All subcontracted laboratories must be reviewed as outlined in SOP A.32. The review process ensures that the subcontracting laboratory meets all requirements of the contract including appropriate certifications. For areas that ASC routinely subcontracts, laboratories are audited either on-site or by a data audit at the beginning of the use of the laboratory. ASC subcontracts to laboratories that continue to meet quality objectives for the projects.

Subcontracting also may take place when ASC is able to perform only part of the requested analyses, testing capacity is exceeded, instrument failure occurs, analysis is not possible within the required hold times, or when a specific client request is made. Clients are notified in writing in advance of any work being subcontracted. Notification may be in the form of e-mail, fax memo, proposal, or contract. When the subcontracted analysis is one that ASC has been certified or approved to perform by the client or regulatory agency, the subcontracting lab must have an equivalent certification or approval such as NELAP, ACE, AFCEE, et al.

Incoming samples that will be subcontracted are subjected to normal sample receipt procedures. The samples are prepared and shipped to the subcontracting laboratory following the appropriate COC procedures. Results are received at ASC and reviewed before the report is sent to the client.

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Laboratory Procurement

14.1 Purchasing, Receiving, Inspection, Inventory, and Storage

The purchase, receipt, inspection, inventorying, and storage of laboratory materials are described in several SOPs. A completed purchase request form provides a clear description of the material to be ordered. The specification for the materials used in testing are listed in the test method SOP. This includes, where applicable, a precise identification and reference to any specifications that must be met. Purchase requisitions are prepared by section supervisors or designee and approved by the Business Manager. The corporate purchasing department orders the material from an approved supplier (see Section 14.3). Upon receipt of the goods, receiving personnel examine them for damage before signing the bill of lading. Materials and quantities in all shipments are compared with what was ordered, and this information is communicated to purchasing.

All stocked items are stored in the warehouse. Non-stocked inventory is forwarded to the requisitioning person. Reagent materials are recorded in a material receipt log, marked with the date received, and placed in storage so that the older materials will be used first.

14.2 Sample Container Purchase

Sample containers are purchased from vendors using generally accepted glass cleaning procedures. Vendors are qualified approximately every one to two years. The vendor selection process includes a preselection review of qualifications, preparation of a request for quotation, and receipt and review of proposals. The competitive bidders are required to send a series of bottles for testing. ASC tests a representative subset of containers to ensure product quality prior to awarding contracts. Once a vendor is awarded the contract, the vendor secures

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large lots of bottles required by the laboratory. Bottles are shipped to ASC or directly to sampling sites from the lots allocated to E & E.

14.3 Vendor Qualification

ASC personnel select vendors for all materials and supplies impacting data quality. The corporate purchasing department selects vendors for office items and non-critical supplies. The vendor selection process is similar to that used for sample containers. Vendors are qualified for approximately one to two years based on historical quality and service. ASC develops a list of materials as part of a request for quotation, and vendors prepare a proposal based on these materials. The competitive bidders are selected for a long-term contract. Critical reagents and standards are pre-tested prior to award. For high use solvents and reagents, vendors are required to maintain large lots allocated for ASC's use. ASC tests all new lots before the existing lot is expended.

The purchase of standards is somewhat different from the purchase of other supplies. In most cases, the laboratory has a reliable source of high quality standards and will continue to use that vendor for long periods. Many standards are ordered as custom mixes. All new lots of standards are checked during testing against the existing lot to ensure consistent quality. Where possible, standards are traceable to NIST or a NELAP-approved vendor, or other approved certification.

15 Client Service

The Project Management Unit is the primary group responsible for providing client service. Other laboratory personnel may handle projects when they are uniquely qualified or technical experts in the area the concern for the project. Project managers are independent of laboratory production and function as the on-site representative for the customer. Project managers are responsible for all client interactions from the quotation or proposal to the final report and invoice. The project manager will usually represent the laboratory in all face-to-face meetings with the client. All project managers are experienced in laboratory analyses and possess the technical expertise to review, interpret, and report client-specific chemistry requirements. Project managers work closely with the Business Manager to maintain schedules and forecast sales. As a group, they are responsible for ensuring that ASC has adequate capacity for each project they manage and the analytical results are reported in a timely manner.

15.1 Project Documentation

The documentation used to specify client requirements may include the initial contract or SOW; project requirements from manuals, meeting notes, or other sources; the original laboratory proposal; contract negotiation notes; a QAPP developed by either the client or the laboratory; or a client audit report. Contracts from the client are reviewed as described in Section 13.1. A process similar to the contract review process is used to review other project documentation. The project manager is responsible for communicating project requirements to the appropriate staff and reviewing all final reports to ensure that the reports meet the client requirements.

Client requirements are communicated to the laboratory staff in many ways, depending on the type of project. Examples of incorporating project-specific requirements into the laboratory process are as follows:

- A project-specific QAPP is developed as an addendum to the QAM or provided by the client. The QAPP is distributed and the staff trained in its use.

- Client-specific requirements are added to administrative or method SOPs.

- Client-specific requirements are incorporated into method data review checklists.

- The project manager notes project specific requirements in the LIMS.

- The project manager holds a kick-off meeting to review requirements of the project.

15.2 Client Concern Resolution

All problems or non-conformance with client requirements are handled through the corrective action process described in Section 4.5. The corrective action process requires the project manager to be notified immediately of any non-conformance with the samples or data. The project manager is responsible for informing the client. Depending on the situation and the client, the project manager will document the problem in the case narrative, or notify the client immediately and document the problem in the narrative.

If the client raises concerns after receiving the analytical report, those concerns are addressed as part of the corrective action process. All client inquiries should be addressed within 48 hours of receipt. If follow-up reports or analysis is required, the laboratory will proceed to complete the tasks with the highest possible priority. Long-term client concerns will be tracked as part of the corrective action process. The status of client concerns are reported to laboratory management at weekly project management meetings. A record of all inquiries and their resolution is maintained with the corrective action form in the QA files or in the project files.

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References

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International vocabulary of basic and general terms in metrology (VIM): 1984, issued by BIPM. IEC. ISO. and OIML.

ISO Guide 8402: 1986. Quality – Vocabulary.

ISO/IEC Guide 2: 1986. General terms and their definitions concerning standardization and related activities.

ISO/IEC Guide 17025: 1999 (E). General requirements for the competence of testing and calibration laboratories.

NELAC, July 1, 1999, *Quality Systems Manual*, Revision 12.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846 3rd ed., Update III, June 1997.

United States Environmental Protection Agency (EPA), August 1992. *Methods for the Determination of Organic Compounds in Drinking Water – Supplement II*, EPA-600/R-92/129.

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_____, revised March 1983 and 1979, *Methods for the Chemical Analysis of Water and Wastes*, EMSL, Cincinnati, EPA-600/4-79-020.

_____, *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, Appendix A to Part 136.

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Definitions

The following definitions are used in the QAM or related SOPs. The following references were used: ISO 8402, ANSI/ASQC E-4, EPA's Quality Assurance Division Glossary of Terms, EPA SW-46, New York State Department of Health Environmental Laboratory Approval Program, definitions developed by NELAC and/or the ASC QA Unit. The source of each definition is noted.

Acceptable Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: the process by which an agency or organization evaluates and recognizes a program of study or an institution as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. (NELAC)

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Batch: a group of samples which behave similarly with respect to the sampling or testing procedures being employed which are processed as a unit, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 of the same matrix which are prepared together. An **analytical batch** is composed of prepared samples (extracts, digestates or concentrates) which are analyzed together as a group. (EPA SW846, ASC)

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage, or analysis. The blank is subjected to the usual analytical and

measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC, Definitions of Environmental Quality Assurance Terms, 1996)

Blind Sample: a subsample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its concentration. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process. (NELAC)

Calibrate: to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration: the set of operations which establishes, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system, or values represented by a material measure, and the corresponding known values of a measurement. (VIM - 6.13)

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method: defined technical procedure for performing a calibration such as internal standard or external standard. Internal standard calibration is typically used for GCMS methods. External calibration is used for most other methods. (NELAC, ASC)

Calibration Standard: a solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The calibration solutions are used to calibrate the instrument response with respect to analyte concentration. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Certified Reference Material (CRM): a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30 - 2.2)

Chain of Custody: an unbroken trail of accountability that documents the physical security of samples, data, and records. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Confirmation: verification of the presence of a component through the use of an analytical technique that differs from the original method. These may include:
Second column confirmation,
Alternate wavelength,
Derivatization,
Mass spectral interpretation,
Alternative detectors, or
Additional cleanup procedures. (NELAC)

Continuing Calibration Verification: analysis of a calibration standard to verify the validity of the daily calibration during and at the end for each analytical batch or sequence. (ASC)

Corrective Action: action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria.) (NELAC)

Data Quality Objectives: A statement of the overall level of uncertainty that a decision-maker is willing to accept in results derived from environmental data. (EPA/QAMS)

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collating the data into a more useful form. (NELAC)

Data Validation: The process of evaluating the available data against the project DQOs to make sure that the objectives are met. (EPA SW-846)

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC, Definitions of Environmental Quality Assurance Terms, 1996)

Double Blind Sample: a sample submitted to evaluate performance with concentration and identity unknown to the analyst. (NELAC)

Holding Times (Maximum Allowable Holding Times): the maximum times that samples may be held prior to preparation and analysis and still be considered valid. (40 CFR Part 136).

Initial Calibration Verification: analysis of an independent calibration standard to verify the validity of the initial calibration curve for each analytical batch. (ASC)

Initial Demonstration of Analytical Capability: procedure to establish the ability of the laboratory to generate acceptable accuracy and precision which is included in many of EPA's analytical methods. In general, the procedure includes the addition of a specified concentration of each analyte into each of four separate aliquots of laboratory pure water or analyte-free solid matrix. These are carried through the entire analytical procedure, and the percentage recovery and the standard deviation are determined and compared to specified limits. (40 CFR Part 136, EPA SW-846).

Initial Precision and Recovery: same as Initial Demonstration of Analytical Capability. (ASC)

Instrument Blank: a clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Internal Standard: a known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical method. (NELAC)

Laboratory Control Sample: an uncontaminated sample matrix spiked with known amounts of analytes from a source independent of the calibration standards. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Matrix: the component or substrate that contains the analyte of interest. For purposes of batch determination, the following matrix types shall be used:

- Aqueous: Any aqueous sample excluded from the definition of a drinking water matrix or Saline/Estuarine source. Includes surface water, groundwater and effluents.
- Drinking water: Any aqueous sample that has been designated a potable or potential potable water source.
- Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
- Non-aqueous liquid: Any organic liquid with <15% settleable solids.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
- Chemical Waste: A product or by-product of a industrial process that results in a matrix not previously defined.
- Air Samples: Media used to retain the analyte of interest from an air sample such as sorbent tubes or summa canisters. Each medium shall be considered as a distinct matrix.
- Wipes: gauze. (Quality Systems)

Matrix Duplicate: an intralaboratory split sample which is used to document the precision of a method in a given sample matrix. (EPA SW-846)

Matrix Spike (spiked sample, fortified sample): prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the

effect of the matrix on a method's recovery efficiency. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Matrix Spike Duplicate (spiked sample, fortified sample duplicate): a second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

May: permitted, but not required. (TRADE)

Method Blank: a clean sample processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Method Detection Limit (Analytical Detection Limit): the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B).

Must: denotes a requirement that must be met. (Random House College Dictionary)

NELAC: National Environmental Laboratory Accreditation Conference. A voluntary organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

NELAP: the overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Performance Audit: the routine comparison of independently obtained quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS): a set of processes wherein the data quality needs, mandates, or limitations of a program or project are specified and serve as criteria for selecting appro-

priate methods to meet those needs in a cost-effective manner. (NELAC)

Practical Quantitation Limit (PQL): the limit typically reported for the method that is above the MDL. The ability to reach the PQL should be documented with each analytical batch, typically by using the lowest standard of a verified calibration curve at the PQL. (ASC)

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Test Sample (PT): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Proficiency Testing: Determination of the laboratory calibration or testing performance by means of interlaboratory comparisons. (ISO/IEC Guide 2 - 12.6, amended)

Pure Reagent Water: shall be ASTM Type I or Type II water in which no target analytes or interferences are detected as required by the analytical method.

Quality Assurance: an integrated system of activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

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Quality Control Sample: an uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Quality Manual: A document stating the quality policy, quality system and quality practices of an organization. This may also be called a Quality Assurance Plan or a Quality Plan. (NELAC)

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-41994)

Range: the difference between the minimum and the maximum of a set of values. (NELAC)

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media (including dictated observations), and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data, and verified accurate by signature), the exact copy or exact transcript may be submitted. (NELAC)

Reagent Blank (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Reagent Grade: Analytical reagent grade or related terms which conform to the specifications of the Committee on Analytical Reagents for the American Chemical Society. (EPA SW-846)

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or the assignment of values to materials. (ISO Guide 30 - 2.1)

Reference Sample: prepared by spiking a known amount of analyte into an appropriate solvent. The concentrate or quality control sample preferably should be obtained from an independent source. A sample prepared in-house may be used if it is prepared independently of the calibration standard. (NYSDOH ELAP)

Replicate Analyses: the measurements of the variable of interest performed identically on two or more subsamples of the same sample within a short time interval. (NELAC)

Sample Delivery Group (SDG): a group of samples received for the same project, site, and/or client within a maximum two-week period that are reported together in a single data package. An SDG typically consists of multiple work orders or sample log-ins and is often designated by the client. (ASC)

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method, including sampling and analysis. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Secure Storage Area: an area of the laboratory with access limited to essential personnel, trained in the function of retrieving samples or information from the area. (ASC)

Selectivity: the capability of a method or instrument to respond to a target substance or constituent in the presence of nontarget substances (Analytical chemistry)

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Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification as long as the requirement is fulfilled. (Style Manual for Preparation of Proposed American National Standards, American National Standards Institute, eighth edition, March 1991)

Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (Style Manual for Preparation of Proposed American National Standards, American National Standards Institute, eighth edition, March 1991)

Standard Additions: The practice of adding a known amount of an analyte to a sample immediately prior to analysis. It is typically used to evaluate interferences. (EPA SW-846)

Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Spike: a known mass of target analyte added to a blank sample or subsample; used to determine recovery efficiency or for other quality control purposes. (NELAC)

Split Sample: aliquots of sample taken from the same container and analyzed independently. In cases where aliquots of samples are impossible to obtain, field duplicates samples should be taken for the matrix duplicate analysis. These are usually taken after mixing or compositing and are used to document intra- or interlaboratory precision. (EPA SW-846)

Standard Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology

and characterized for absolute content, independent of analytical method. (NELAC)

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Systems Audit: a thorough, systematic, on-site qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (NELAC)

Temperature Blank: usually a 40-mL VOA vial filled with water sent with a shipment that is clearly marked "Temp Blank." The temperature blank is used to establish the temperature of the samples when the cooler is open. The packaging and placement of the vial should be representative of the samples in the container. (ASC)

Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure.

NOTE: The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2 - 12.1, amended)

Test Method: defined technical procedure for performing a test. (NELAC)

Testing Laboratory: laboratory that performs tests. (ISO/IEC Guide 2 - 12.4)

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards (generally international or national), through an unbroken chain of comparisons. (VIM - 6.12)

Verified Time of Sample Receipt (VSTR): the time the cooler is delivered to the laboratory by the client or independent courier as recorded in the package receipt log. (ASC from EPA CLP)

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Work Order: the number assigned to a shipment of samples delivered on the same day for the same project, site, or client. The job file contains all data associated with the samples in the work order. (ASC)

B ASC Commonly Used Forms

5514

ANALYTICAL SERVICES CENTER DOCUMENT CONTROL FORM

Document Title:	**SOP, Checklist, Form:**	
Assigned Reviewer:	**Method**	**Current Revision No.:**
Peer Reviewer:		**Final Revision No.:**
QA Review	**Notes:**	
Management Review		

REVIEW SCHEDULE

	Draft Reviewed	Date Reviewed	Final Approved	Date Final
Reviewer				
Peer Reviewer				
QA Review				
Management Review				

MINOR REVISION APPROVALS

Comment	QA Authorization/Date	Date Revision Issued	Comment	QA Authorization/Date	Date Revision Issued

PRODUCTION INFORMATION (To Be Completed by Document Control Officer)

Date Training Complete:
Assigned Implementation Date:
Date Archived:
Special Instructions:

Corrective Action Form

Tracking Number	Total Pages

SECTION 1 INITIATION

Originator	Start Date	SOP No.	Instrument
Work Order No.	Sample Nos.		

Source of Non-Conformance?

[ ] Data Review [ ] External Audit [ ] Client Review [ ] Analysis
[ ] Internal Audit [ ] PE [ ] SOP [ ] Other _____

Client	Section

Non-Conformance Description

Person to Complete Corrective Action: Person Assigned to Review Corrective Action:

Additional Routing: Projected Closure Date:

SECTION 2 CORRECTIVE ACTION

Corrective Action Description

Person Completing or Overseeing Corrective Action: Actual Closure Date:

SECTION 3 REVIEW

Additional Corrective Actions/Follow-up Comments

Person Reviewing & Approving:	Date:	New Form Tracking Numbers:

SECTION 4 QA

Additional Corrective Actions/Follow-up Comments

QA Approval: Date:

QA Classification

[ ] Hold Times	[ ] Surr. Recovery	[ ] Sample	[ ] Documentation	[ ] Technique/Process
[ ] Equip/Materials	[ ] Spike Recovery	[ ] Personnel	[ ] Calibration, initial	[ ] LIMS
[ ] Blank	[ ] LCS Recovery	[ ] Reporting	[ ] Calibration, continuing	[ ] Field

[ ] Other

_____

C

Laboratory Certifications and Accreditations

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Laboratory Certifications and Accreditations					
Source	**Issuing Agency**	**Description of License, Registration or Certification**	**License, Registration, Certification Number**	**Scope of Evaluation**	**Renewal Date**
United States Army Corps of Engineers	HTRW Center of Expertise, Omaha District	Solid and hazardous waste and water for organics and inorganics by EPA RCRA methods.	N/A	PE Sample Audit QA Manual	11/2/02
United States Army	Army Environmental Center	Soil and water for organics, and metals by validated methods.	Lab EL	PE Sample Audit QA Manual	Updated by project
United States Air Force	Air Force Center for Environmental Excellence (AFCEE)	Solid and hazardous waste for organics and inorganics based on AFCEE requirements.	N/A	Subcontract Audit	Updated by project
Alaska	Department of Environmental Conservation	Soil and water for organics, and inorganics for UST projects.	UST-024	PE Sample QA Manual	3/18/01
Arkansas	Department of Environmental Quality Laboratory Certification Program	Soils and waters for organics and inorganics.	N/A	NELAP	3/9/02
California	Department of Health Services	Solid and hazardous waste for organics and inorganics; waste extraction test.	1766	NELAP	6/30/02
Florida	Department of Health and Rehabilitative Services	Wastewater and solid and hazardous waste for organics and inorganics.	E87037	NELAP	6/30/01

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Laboratory Certifications and Accreditations					
Source	**Issuing Agency**	**Description of License, Registration or Certification**	**License, Registration, Certification Number**	**Scope of Evaluation**	**Renewal Date**
Florida	Department of Environmental Protection	Approved comprehensive QA plan pursuant to statewide environmental chemistry laboratory QA programs.	860165G	QA Manual	1/01
Illinois	Environmental Protection Agency	Hazardous wastes analysis for organics and inorganics.	000243	NELAP	12/31/01
Kansas	Department of Health and Environment	Solid and hazardous waste for organics and inorganics.	E-10157	NELAP	1/31/02
Kentucky	Natural Resources and Environmental Protection Cabinet	Drinking water for organics and inorganics.	90083	PE Sample	12/31/01
Maryland	Department of Health and Mental Hygiene	Drinking water for metals and volatile organics.	290	PE Sample	9/30/01
Massachusetts	Department of Environmental Protection	Potable water for select parameters and nonpotable water for organics and inorganics.	M-NY050	PE Sample	6/30/01
New Jersey	Department of Environmental Protection	Nonpotable water for organics and inorganics.	73792	NELAP	6/30/01

Laboratory Certifications and Accreditations					
Source	**Issuing Agency**	**Description of License, Registration or Certification**	**License, Registration, Certification Number**	**Scope of Evaluation**	**Renewal Date**
New York	State Department of Health Environmental Laboratory Approval Program	CLP for organics and inorganics by NYSDEC Analytical Services Protocol. Potable/nonpotable water, solid/hazardous waste, and air and emissions for organics and inorganics.	10486	NELAP Primary Accrediting Authority (PT Sample and Audit)	7/31/01
North Carolina	Department of the Environment and Natural Resources	Wastewater and groundwater for select parameters.	382	PE Sample Audit	12/31/03
Pennsylvania	Department of Environ-mental Protection	Drinking water for volatile organics.	68-519	PE Sample Audit QA Manual	7/11/01
South Carolina	Department of Health and Environmental Control	Wastewater and solid and hazardous waste for organics and inorganics.	91001	PE Sample	6/30/01

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Laboratory Certifications and Accreditations					
Source	**Issuing Agency**	**Description of License, Registration or Certification**	**License, Registration, Certification Number**	**Scope of Evaluation**	**Renewal Date**
Virginia	Department of General Services	Drinking water for organics and inorganics.	00124	PE Sample	6/30/01

Notes:

Audit = Successful completion of on-site audit and response to audit report.
PE Sample = Successful analysis of a performance evaluation sample.
QA Manual = Review of Quality Assurance Manual and related documentation by off-site agency.

Key:

CLP = Contract Laboratory Program (EPA).
HTRW = Hazardous, toxic, and radioactive waste.
N/A = Not applicable.
NYSDEC = New York State Department of Environmental Conservation.
PE = Performance evaluation.
QA = Quality assurance.
UST = Underground storage tank.

D

ASC Equipment List

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MAJOR LABORATORY EQUIPMENT FOR ANALYTICAL SERVICES CENTER			
Make/Model #	**Description**	**Year**	**Total**
Gas Chromatography Mass Spectrometry (GC/MS) Sections		**(All Instruments Purchased New)**	
Hewlett Packard (HP) 5972	GC/MS, Capillary Column	1994 [2]	2
HP 5971	GC/MS, Capillary Column	1993	1
HP 5970	GC/MS, Capillary Column	1985, 1986, 1987 [2] 1989	5
HP 5973	GC/MS, Capillary Column	1998, 2001	2
HP 5972	GC/MS, Capillary Column	1998	1
HP 9000/735	UNIX Operating Systems for GC/MS	1994	1
HP Vectra	Chemstation Operating Systems for GC/MS	1994, 1996, 1998	3
HP 1000 RTE-A	Mini-Computer Operating Systems for GC/MS	1987, 1990, 1991	3
Tekmar LSC-2000	Liquid Sample Concentrators for volatile organic compounds (VOCs)	1989, 1990, 1991, 1997	4
Tekmar ALS-2032	Sampler for VOCs in Water	1993	1
Tekmar ALS-2016	Samplers for VOCs in Water and Soil	1989, 1990, 1991	3
OI Analytical Model 4560/4551	Purge-and-Trap Unit with Closed System Vial Autosampler and SIM/Spiker for VOCs in Water	1994, 2001	2
OI Analytical Model 4560	Purge-and-Trap Unit with Closed System Vial Autosampler and SIM/Spiker for VOCs in Water	2000, 2001	2
OI Analytical Model 4552 Archon Autosampler	Purge-and-Trap Unit with Closed System Vial Autosampler and SIM/Spiker for VOCs in Water and Soil	1998[2], 2000, 2001	4
4 HP 7673A; 1 HP 7683	Single Tower Automatic Samplers	1987[2], 1993, 1994, 1998	5
Nutech	Autosampler for Air Canisters	1994	1
Nutech Model 3550A	Air System for Automated Method TO14 Analysis	1994	1
Dynatech Dynatrap	Automated Closed Loop Purge-and-Trap Unit for VOCs in Water and Soil	1993	1
Custom	SUMMA® Canister Cleaning Apparatus	1992	1
Dynatech Precision PTA 30	Purge-and-Trap Closed Loop Autosampler for VOCs in Water	1992	1

MAJOR LABORATORY EQUIPMENT FOR ANALYTICAL SERVICES CENTER			
Make/Model #	**Description**	**Year**	**Total**
Gas Chromatography (GC) Volatile and Semivolatile Sections		**All Instruments Purchased New**	
HP 6890	GC Capillary Column with Dual Electron Capture Detector (ECD) Detectors and HP7673B Twin Autoinjector Towers.	1997, 1997	2
HP 5890 II	GC Capillary Column Dual Flame Ionization Detectors (FID)	1994	1
HP 5890 II	GC Capillary Column with Dual ECD Detectors and HP7673A Single Autoinjector Tower	1991, 1993	2
Varian 3600	GC Capillary Column with Photoionization Detector (PID)/Hall Detectors	1994	1
Varian 3400	GC Capillary Column with Dual ECDs and LEAP A200SE Autosamplers	1989	3
Varian 3400	GC Capillary Column with FID	1990	1
Varian 3400	GC Capillary Column with FID/PID	1990	1
HP 5890 II	GC Capillary Column with Dual FID Detectors and HP7673A Twin Auto-injector Towers	1989	1
Perkin Elmer (PE) Turbochrom Client Server	Chromatographic Analysis Data System running on HP Netserver LH Pro 200 with seven HP Vectra Pentium 133 PC Stations	1997	1
Tekmar LSC-2000	Liquid Sample Concentrators for VOCs	1989, 1990, 1991	5
Tekmar ALS-2016	Samplers for VOCs in Water and Soil	1989, 1990, 1991	5
Varian 8200	Autosampler	1994	1
Whatman Model 75-34NA	Hydrogen Generators	1994, 1996 [2]	3
High Performance Liquid Chromatography (HPLC) Section		**All Instruments Purchased New**	
HP Series 1050	HPLCs with Variable Ultraviolet Detectors and one Fluorescence Detector	1990[2], 1992, 1995	4
HP Vectra	Chemstation Operating Systems	1990	2
Organic Sample Preparation Section		**All Instruments Purchased New**	
Organomation ROT-X-TRACT-HP	8-Position Extractor with Soxhlet Extractor Glassware	1994	1
National Model NLW-66	Glassware Washer	1994	1
ABC AS 2000	Gel Permeation/Autoevaporation Concentrator	1992	1

MAJOR LABORATORY EQUIPMENT FOR ANALYTICAL SERVICES CENTER			
Make/Model #	**Description**	**Year**	**Total**
Zymark BenchMate	Dual Column Injection/Filtration System for Gel Permeation	1995	2
ISCO Foxy 200	Fraction Collectors	1999	3
Metals Analysis Section		**All Instruments Purchased New**	
PE Optima 3000 XL	Inductively Coupled Argon Plasma (ICP) Spectrometer with Stand-Alone Data System	1994	1
PE 4100ZL	Zeeman Atomic Absorption Spectrophotometer (AAS) with Graphite Atomizer, Background Correction System, and Autosampler	1993	1
PE 5100	Zeeman AASs with Graphite Atomizers, Background Correction Systems, and Autosamplers	1991	2
Leeman Labs PS 200 II	Mercury Analyzer	1999	1
Accumet 925	PH Meter	1997	1
PE Optima 3380 DV	ICP Spectrometer with Dual View Graphics and Stand-Alone Data System	1999	1
General Analytical Chemistry (GAC) Section		**All Instruments Purchased New**	
Dionex 2000I	Ion Chromatograh with Anion Conductivity Detector	1987	1
Dionex ED-40	Electrochemical Anion Conductivity Detector	1996	1
Milton Roy Spectronic Geosys 5	Spectrophotometer	1994	1
Milton Roy Spectronic 601	Spectrophotometer	1990	1
PE Model 1650	FT-IR Spectrophotometer (helium laser)	1990	1
Lachat QuickChem FIA 8000 Series	Automated Analyzer with Flow Injection Random Sampler and Heat Block	1999	1
Shimadzu SSM 5000/5050A/ ASI-5000A	Total Organic Carbon Analyzer and Solid Sample Module/Autosampler	1997/2000	1
MCI (COSA) TOX-10	Total Organic Halogen Analyzer/Preparator	1985/1999 (Refurbished)	1
YSI Model 35	BOD Meter	1995	1
HF Scientific	Turbidity Meter	1992	1

MAJOR LABORATORY EQUIPMENT FOR ANALYTICAL SERVICES CENTER			
Make/Model #	**Description**	**Year**	**Total**
Westco Scientific	Cyanide Distillation System, Easy Dist Jr.	2000	1
Büchi 316	Distillation Unit	1994	1
Denver Instrument Model 225	pH meter	1999	1

E

ASC Tests List

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Test Name	Test No.	Matrix	Holding Time (d)	Bottle Type	Reference Source
Alkalinity, Total by Method 2320B	SM2320B	Water	14	1-L HDPE, Cool 4°C	Standard Methods, 18th ed., 1992
Ammonia, Nitrogen by Method 4500-NH3.B,E	SM4500-NH3 BE	Water	28	1-L HDPE, H2SO4 to pH <2, Cool 4°C	Standard Methods, 18th ed., 1992
Anions by Ion Chromatography by Method 300.0	EPA300.0	Water	2	1-L HDPE, Cool 4°C	EPA 600/R-93/100, August 1993
Anions by Ion Chromatography Method 9056	SW9056	Water	2	1-L HDPE, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Biochemical Oxygen Demand, 5-Day	SM5210B	Water	2	1-L HDPE, Cool 4°C	Standard Methods, 18th ed., 1992
Chemical Oxygen Demand by Method SM5220 C	SM5220 C	Water	28	1-L HDPE, H2SO4 to pH <2, Cool 4°C	Standard Methods, 18th ed., 1992
Chloride by Method 325.2	EPA325.2	Water	28	1-L HDPE, Cool 4°C	EPA 600/4-79-020, March 1993
Chlorinated Herbicides by Method 8151A	SW8151A	Water	7, 40	1-L Amber glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Chlorinated Herbicides by Method 8151A	SW8151A	Solid	14, 40	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Cyanide by SOW ILM04.1	ILM04.1	Soil	12	250-ml Clear widemouth glass, Cool 4°C	EPA SOW ILM04.1
Cyanide by SOW ILM04.1	ILM04.1	Water	12	1-L HDPE, NaOH to pH >12, Cool 4°C	EPA SOW ILM04.1
Cyanide, Total by Method 335.3	EPA335.3	Water	14	1-L HDPE, NaOH to pH >12, Cool 4°C	EPA 600/4-79-020, March 1993
Cyanide, Total by Method 335.4	EPA335.4	Drinking Water	14	1-L HDPE, NaOH to pH >12, Cool 4°C	EPA 600/R-93/100, August 1993
Cyanide, Total by Method 9012A	SW9012A	Solid	14	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Cyanide, Total by Method 9012A	SW9012A	Water	14	1-L HDPE, NaOH to pH >12, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Diesel Range Organics by Method 8015B	SW8015B	Organic	14, 40	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Diesel Range Organics by Method 8015B	SW8015B	Solid	14, 40	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Diesel Range Organics by Method 8015B	SW8015B	Water	7, 40	1-L Amber glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Diesel Range Organics by Method AK102	AK102	Solid	14, 40	250-ml Clear widemouth glass, Cool 4°C	ADEC UST Proc. Manual App. E 1/31/96
Diesel Range Organics by Method AK102	AK102	Water	7, 40	1-L Amber glass, H2SO4 to pH <2, Cool 4°C	ADEC UST Proc. Manual App. E 1/31/96
Gasoline Range Organics by Method 8015B	SW8015B	Solid	14	40-mL VOA vial, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Gasoline Range Organics by Method 8015B	SW8015B	Water	14	40-mL VOA vial, HCl to pH <2, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Gasoline Range Organics by Method AK101	AK101	Solid	28	40-mL VOA vial, Cool 4°C	ADEC UST Proc. Manual App. E 1/31/96
Gasoline Range Organics by Method AK101	AK101	Water	14	40-mL VOA vial, HCl to pH <2, Cool 4°C	ADEC UST Proc. Manual App. E 1/31/96
Hardness, Total by Method EPA 130.2	EPA130.2	Water	180	1-L HDPE, HNO3 to pH <2	EPA 600/4-79-020, March 1993
Hexavalent Chromium by 7196A/3060A	SW7196A	Soil	30, 7	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Hexavalent Chromium by Method 3500-Cr D	SM3500-Cr D	Water	1	125-mL HDPE, Cool 4°C	Standard Methods, 18th Edition

Test Name	Test No.	Matrix	Holding Time (d)	Bottle Type	Reference Source
Hexavalent Chromium by Method 7196A	SW7196A	Water	1	125-mL HDPE, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Ignitability (Flashpoint), Liquids by Method 1010	SW1010	Organic	40	250-ml Clear widemouth glass, Cool 4°C	SW-846
Ignitability, Solids by Method 1030	SW1030	Solid	40	250-ml Clear widemouth glass, Cool 4°C	SW-846
Mercury Analysis by Method ILM04.1	ILM04.1_HG	Solid	26	250-ml Clear widemouth glass, Cool 4°C	EPA CLP ILM04.1
Mercury Analysis by Method ILM04.1	ILM04.1_HG	Water	26	1-L HDPE, HNO3 to pH <2	EPA CLP ILM04.1
Mercury Analysis in Soil by Method 7471A	SW7471A	Soil	28	1-L HDPE, HNO3 to pH <2	SW-846 3rd Ed., Update III, June 1997
Mercury Analysis in Water by Method 7470A	SW7470	Water	28	1-L HDPE, HNO3 to pH <2	SW-846 3rd Ed., Update III, June 1997
Mercury by Method 245.1	EPA245.1	Water	28	1-L HDPE, HNO3 to pH <2	SW-846 3rd Ed., Update III, June 1997
Metals by ICP Method 200.7	EPA200.7	Drinking Water	180	1-L HDPE, HNO3 to pH <2	EPA- 600/R-94/111, May 1994
Metals by ICP Method 200.7	EPA200.7	Water	180	1-L HDPE, HNO3 to pH <2	EPA- 600/R-94/111, May 1994
Metals by ICP Method 6010B	SW6010B	Solid	180	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Metals by ICP Method 6010B	SW6010B	Water	180	1-L HDPE, HNO3 to pH <2	SW-846 3rd Ed., Update III, June 1997
Metals by ICP Method 7300	NIOSH7300	Air	180	37mm Air Cassette	NIOSH 4th Ed., August 1994
Nitrate/Nitrite by Method 353.2	EPA353.2	Water	2	1-L HDPE, Cool 4°C	EPA 600/4-79-020, March 1993
Nitrate/Nitrite by Method 353.2	EPA353.2	Water	28	1-L HDPE, Cool 4°C	EPA 600/4-79-020, March 1993
Nitroaromatics and Nitroamines by Method 8330	SW8330	Solid	14, 40	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Nitroaromatics and Nitroamines by Method 8330	SW8330	Water	7, 40	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Oil and Grease by Method 413.1	EPA413.1	Water	28	1-L Amber glass, H2SO4 to pH <2, Cool 4°C	Standard Methods, 18th ed., 1992
Orthophosphate by Method 365.2	EPA365.2	Water	2	1-L HDPE, Cool 4°C	EPA 600/4-79-020, March 1993
PAH by HPLC Method 610	EPA610	Water	7, 40	1-L Amber glass, Cool 4°C	40 CFR Part 136 Appendix A
PAH by Method 8310	SW8310	Solid	14, 40	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
PAH by Method 8310	SW8310	Water	7, 40	1-L Amber glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
PCB by Method 8082	SW8082	Solid	14, 40	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
PCB by Method 8082	SW8082	Water	7, 40	1-L Amber glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Perchlorate by Method 314.0	EPA 314.0	Water	28	1-L HDPE, Cool 4°C	EPA NERL ORD November 1999
Pesticide and PCB Analysis by Method 608	EPA608	Water	7, 40	1-L Amber glass, Cool 4°C	Method 608, 40 CFR, Part 136, Appendix A
Pesticide/PCB by Method OLM04.2	OLM04.2_PPCB	Solid	10, 40	250-ml Clear widemouth glass, Cool 4°C	EPA CLP OLM04.2
Pesticide/PCB by Method OLM04.2	OLM04.2_PPCB	Water	5, 40	1-L Amber glass, Cool 4°C	EPA CLP OLM04.2
Pesticides by Method 8081A	SW8081A	Solid	14, 40	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997

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Test Name	Test No.	Matrix	Holding Time (d)	Bottle Type	Reference Source
Pesticides by Method 8081A	SW8081A	Water	7, 40	1-L Amber glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Petroleum Products, Florida	FL-PRO	Solid	14, 40	1-L Amber glass, Cool 4°C	FDEP M for Det. of Pet. Range Organics Nov. 1995
Petroleum Products, Florida	FL-PRO	Water	7, 40	1-L Amber glass, Cool 4°C	FDEP M for Det. of Pet. Range Organics Nov. 1995
pH by Method 9040B	SW9040B	Water	immediately	125-mL HDPE, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
pH by method EPA 150.1	EPA150.1	Water	immediately	125-mL HDPE, Cool 4°C	EPA 600/4-79-020, March 1993
pH by Method EPA 9045C	SW9045C	Solid	ASAP	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Phenols by Method 420.1 (4AAP)	EPA420.1	Water	28	1-L Amber glass, H2SO4 to pH <2, Cool 4°C	EPA 600/4-79-020, March 1993
Phenols by Method 9065 (4AAP)	SW9065	Water	28	1-L Amber glass, H2SO4 to pH <2, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Phosphorus, Total by Method 365.1	EPA365.1	Water	28	1-L HDPE, H2SO4 to pH <2, Cool 4°C	EPA 600/4-79-020, March 1993
Phosphorus, Total by Method 365.2	EPA365.2	Water	28	1-L HDPE, H2SO4 to pH <2, Cool 4°C	EPA 600/4-79-020, March 1993
Reactive Cyanide by Method 9012A-7.3.3	SW7.3.3.2	Waste	28	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Reactive Sulfide by Method 9034-7.3.4	SW7.3.4.2	Waste	28	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Residual Range Organics by Method AK103	AK103	Solid	14, 40	250-ml Clear widemouth glass, Cool 4°C	ADEC UST Proc. Manual App. E 1/31/96
Semivolatile Organics by Method 1625	EPA1625	Water	40	1-L Amber glass, Cool 4°C	40 CFR Part 136 Appendix A
Semivolatile Organics by Method 625	EPA625	Water	40	2-L Amber glass, Cool 4°C	40 CFR Part 136 Appendix A
Semivolatile Organics by Method 8270C	SW8270C	Solid	14, 40	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Semivolatile Organics by Method 8270C	SW8270C	Water	7, 40	1-L Amber glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Semivolatile Organics by Method OLM04.2	OLM04.2_SVOA	Solid	10, 40	1-L Amber glass, Cool 4°C	EPA CLP OLM04.2
Semivolatile Organics by Method OLM04.2	OLM04.2_SVOA	Water	5, 40	1-L Amber glass, Cool 4°C	EPA CLP OLM04.2
Solids, Settleable by Method 160.5	EPA160.5	Water	2	1-L HDPE, Cool 4°C	EPA 600/4-79-020, March 1993
Solids, Total by Method 160.3	EPA160.3	Water	7	1-L HDPE, Cool 4°C	EPA 600/4-79-020, March 1993
Specific Conductance by Method 120.1	EPA120.1	Water	28	1-L HDPE, Cool 4°C	EPA 600/4-79-020, March 1993
SPLP Ext for Metals by M 1312	SW1312	Waste	28	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Sulfate by Method 375.4 (Turbidimetric)	EPA375.4	Water	28	1-L HDPE, Cool 4°C	EPA 600/4-79-020, March 1993
Sulfide by Method 376.1	EPA376.1	Water	7	1-L HDPE, 10N NaOH to pH 9 / ZnAc	EPA 600/4-79-020, March 1993
Sulfide by Method 9030A	SW9030A	Solid	7	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
TCLP Ext for Metals by Method 1311	SW1311	Waste	180(Hg 28)	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
TCLP Ext for Organics by Method 1311	SW1311	Waste	14	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997

Test Name	Test No.	Matrix	Holding Time (d)	Bottle Type	Reference Source
Total Dissolved Solids (TDS) by method EPA 160.1	EPA160.1	Water	7	1-L HDPE, Cool 4°C	EPA 600/4-79-020, March 1993
Total Kjeldahl Nitrogen (TKN) by Method 351.2	EPA351.2	Water	28	1-L HDPE, H2SO4 to pH <2, Cool 4°C	EPA 600/4-79-020, March 1993
Total Kjeldahl Nitrogen (TKN) by Method 351.3	EPA351.3	Water	28	1-L HDPE, H2SO4 to pH <2, Cool 4°C	EPA 600/4-79-020, March 1993
Total Organic Carbon by method 5310B	SM5310B	Water	28	125ml HDPE, H2SO4 to pH<2, Cool 4°C	Standard Methods, 18th ed., 1992
Total Organic Carbon by Method 9060	SW9060	Water	28	125ml HDPE, H2SO4 to pH<2, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Total Organic Carbon by Method Lloyd Kahn	Lloyd Kahn	Solid	14	250-ml Clear widemouth glass, Cool 4°C	EPA Attachment 6, July 1998
Total Organic Halogens by Method 9020B	SW9020B	Water	28	1-L Amber glass, H2SO4 to pH <2, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Total Suspended Solids, Non-filterable Residue	EPA160.2	Water	7	250-mL HDPE	EPA 600/4-79-020, March 1993
TRPH by Method 418.1	EPA418.1	Water	28	1-L Amber glass, H2SO4 to pH <2, Cool 4°C	EPA 600/4-79-020, March 1993
TRPH by Method 418.1M	EPA418.1	Solid	28	250-ml Clear widemouth glass, Cool 4°C	EPA 600/4-79-020, March 1993
TRPH, Soxhlet Extraction by Method 9071A	SW9071A	Solid	28	250-ml Clear widemouth glass, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
TRPH, Soxhlet Extraction by NJDEP Method	SW9071A	Solid	28	250-ml Clear widemouth glass, Cool 4°C	NJDEP Document 0QA-QAM-005-12/89
Turbidity (Nephelometric) by Method 180.1	EPA180.1	Water	2	125-mL HDPE, Cool 4°C	EPA 600/4-79-020, March 1993
VOC by GC Method 8021B	SW8021B	Solid	14	40-mL VOA vial, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
VOC by GC Method 8021B	SW8021B	Water	14	40-mL VOA vial, HCl to pH <2, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
VOC by GCMS Method 1624C	EPA1624	Water	14	40-mL VOA vial, HCl to pH <2, Cool 4°C	40 CFR Part 136 Appendix A
VOC by GCMS Method 524.2	EPA524.2	Drinking Water	14	40-mL VOA vial, HCl to pH <2, Cool 4°C	EPA 524.2, Rev. 4, August 1994
VOC by GCMS Method 624	EPA624	Water	14	40-mL VOA vial, HCl to pH <2, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
VOC by GCMS Method 8260B	SW8260B	Water	14	40-mL VOA vial, HCl to pH <2, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
VOC in Air by GCMS Method TO-1	EPATO1	Air	14	Sorbent Tube for VOC, Tennax	EPA-625/R-96/010B, January 1997
VOC in Air by GCMS Method TO-14A	EPATO14	Air	14	SUMMA Canister	EPA-625/R-96/010B, January 1997
VOC Low Level by GCMS Method 8260B	SW8260B	Water	14	40-mL VOA vial, HCl to pH <2, Cool 4°C	SW-846 3rd Ed., Update III, June 1997
Volatile Organic Aromatics by GC Method 602	EPA602	Water	14	40-mL VOA vial, HCl to pH <2, Cool 4°C	40 CFR Part 136 Appendix A
Volatile Organic Compounds by Method OLM04.2	OLM04.2_VOA	Solid	10	40-mL VOA vial, Cool 4°C	EPA CLP OLM04.2
Volatile Organic Compounds by Method OLM04.2	OLM04.2_VOA	Water	10	40-mL VOA vial, HCl to pH <2, Cool 4°C	EPA CLP OLM04.2
Volatile Organic Halocarbons by GC Method 601	EPA601	Water	14	40-mL VOA vial, Cool 4°C	40 CFR Part 136 Appendix A

Holding Time: values from collection (CLP VTSR) to extraction and from extraction to analysis

F

ASC SOP Inventory

Method	SOP Name	Status	Annual Review	Rev.	SOP No.
	Sample Container, Blank Preparation, and Shipment Procedures	F	1-Nov-01	3	A.4
	pH Control/Pump System Calibration Procedure	F	23-May-01	1	A.6
	Security for the ASC	F	5-Apr-02	1	A.7
	Purchasing and Storing Materials for the ASC	F	27-Sep-01	3	A.8
	Waste Disposal	F	8-Jun-02	4	A.10
	Quality Control Testing of Sample Containers	F	1-May-01	3	A.15
	Determination of Method Detection Limits	F	19-Sep-01	6	A.18
	Balance Check Procedure	F	27-Jul-01	5	A.19
	Laboratory Water Quality	F	2-Mar-02	5	A.21
	Batch Identification	F	11-Jul-01	2	A.22
	Initial Demonstration of Capability	F	12-Feb-02	5	A.23
	Micropipette Use	F	27-Sep-01	4	A.24
	Data Review Procedure	F	2-Aug-01	6	A.25
	Document Control	F	4-Dec-01	1	A.27
	Traceability of Reagents and Standards	F	9-Nov-01	3	A.28
	Continuing Demonstration of Staff Proficiency	F	15-Jan-02	2	A.29
	Oven Calibration and Use	F	6-Mar-02	0	A.30
	Glassware Cleaning	F	6-Mar-02	0	A.31
	Subcontracting Procedure	F	14-Aug-01	2	A.32
	Project Management	F	12-Jul-01	2	A.33
	Confidentiality/Proprietary/Ethics	F	31-Jul-01	1	A.34
	Electrical Power Outage	F	25-May-01	0	A.36
	Laboratory Notebooks	F	1-Nov-01	2	A.37
	Thermometer Calibration	F	1-Nov-01	6	A.38
	Sonic Disrupter Tuning	F	13-Feb-02	0	A.39
	Project Management, Hudson Chromate Project	F	13-Mar-02	0	A.40
	Sample and Laboratory Contamination Minimization Procedure	F	18-May-01	1	A.41
	Procedure for Requesting Permission to Dispose State Samples	F	1-May-01	1	A.42
	Refrigerator, Freezer, and Oven Unit Temperature Check	F	10-Jul-01	1	A.43
	Evidentiary Chain of Custody Procedures	F	5-Apr-02	0	A.44
	External Proficiency Test Program	F	27-Jun-01	0	A.45
	Manual Integration	F	30-Jun-01	0	A.46
	Laboratory Hood Use	F	12-Mar-02	0	A.47
	Internal Chain of Custody	F	18-Apr-02	0	A.48
3580A	Waste Dilution	N	New	0	A.49
	Case Narratives	F	17-Apr-02	0	A.51
	Data Management	F	14-Mar-01	5	Comp.5
	PC Backup, Security, and Recovery	F	14-Mar-01	4	Comp.6
	Electronic Disk Deliverable Generation	F	14-Sep-01	0	Comp.10
8081A/3510C/3520C	Extraction and Concentration of Pesticides and PCB from Water Samples	F	8-Jun-02	6	Ext.4
8151A	Extraction of Soil and Solid Waste for Chlorinated Phenoxy Acid Herbicides	F	19-Sep-01	3	Ext.7
8151A	Extraction of Chlorinated Phenoxy Acid Herbicides from Water Samples	F	14-Sep-01	2	Ext.8
8270C/3550B	High Level BNA Soil Extraction	F	18-Apr-02	2	Ext.12
8310/3510C/3520C	Extraction of PAH in Water for Subsequent Analysis by HPLC	F	29-Jan-02	2	Ext.13

	Security for the ASC	F	5-Apr-02	1	A.7
8081A/3550B	Pesticide/PCB Extraction and Concentration of Soil Samples by Method 3550B	F	12-Apr-02	5	Ext.19
8270C/3510C/3520C	Extraction and Concentration of Water Samples for Analysis of Semivolatile Compounds	F	23-Apr-02	8	Ext.20
8310/3550B	Extraction of PAH in Solids for Subsequent Analysis by HPLC	F	4-Jun-02	1	Ext.25
8015B/3510C/3550B	EPH Extraction and Concentration of Soil and Water Samples	F	25-Apr-02	2	Ext.30
1311/8270C/3510C	Extraction and Concentration of TCLP Extracts for Analysis of Semivolatile Compounds	F	20-Apr-02	2	Ext.31
OLM04.2	Continuous Liquid-Liquid Extraction of Semivolatile Compounds in Water; CLP	F	29-Jan-02	5	Ext.32
OLM04.2	Semivolatile (BNA) Low-Level Extraction of Soils, CLP	F	8-Dec-00	6	Ext.33
OLM04.2	Extraction of Pesticides/PCB from Water Samples; CLP	F	18-Jan-02	5	Ext.35
OLM04.2	Extraction of Pesticides/PCB from Soil Samples; CLP	F	13-Feb-02	5	Ext.36
8270C/3550B	Extraction and Concentration of Soil Samples for Analysis of Semivolatile Compounds	F	25-Apr-02	3	Ext.46
AK102	DRO Extraction/Concentration of Soil or Water Samples – AK102	F	14-Mar-01	2	Ext.48
1311/8081A	Pesticide Extraction and Concentration of TCLP Extracts by Method 3510C or 3520C	F	4-Jun-02	1	Ext.58
AK103	RRO Extraction and Concentration of Soil Samples	F	14-Mar-01	2	Ext.66
OLC03.2	Continuous Liquid-Liquid Extraction of Low Level Semivolatile Compounds in Water; CLP-LL	R	Reactivated	2	Ext.71
	Extraction Procedure and Special Cleanup for Buffalo Color Samples	F	23-Apr-02	2	Ext.70
OLM04.2	Gel Permeation Chromatography Cleanup for Pest/PCB Soils	F	14-Feb-02	2	Ext.72
OLM04.2	Gel Permeation Chromatography Cleanup for BNA Soils	F	14-Feb-02	2	Ext.73
1653	In-Situ Acetylation, Extraction, and Concentration of Water Samples for the Analysis of Chlorophenolics	F	2-Oct-01	0	Ext.78
1625	Extraction of Semivolatile Organics in Water by Method 1625 Isotope Dilution	F	11-Oct-00	0	Ext.80
OLM04.2	CLP Semivolatile Medium Level Extraction	F	24-May-01	0	Ext.81
3620B	Florisil Cleanup	F	18-Apr-02	0	Ext.85
	Isolation of DNA from Shrimp Tissue	F	19-Feb-02	0	Ext.87
OLC03.2	Extraction of Pesticide/PCB from Water Samples; Low Level CLP	N	NEW	0	Ext.90
	Analysis of PCR Products by Agarose Gel Electrophoresis	F	19-Feb-02	0	Ext.88
	Polymerase Chain Reaction (PCR) Assay for White Spot Virus in Shrimp	F	19-Feb-02	0	Ext.89
8015B/3520C	Extraction of DRO from Water Samples by Method 3520C	N	NEW	0	Ext.90
160.4	Solids, Volatile by Method 160.4	F	17-Apr-02	2	GAC.40
351.3	Total Kjeldahl Nitrogen (TKN)	F	16-Jan-02	2	GAC.4
SM4500 NH3 B E	Ammonia-Nitrogen by Method SM4500 NH3 B E	F	27-Jul-01	1	GAC.2
418.1	Preparation of Water Samples for 418.1 Total Recoverable Petroleum Hydrocarbons - Water	F	13-Jun-02	4	GAC.8
5210B	Biochemical Oxygen Demand (BOD5 and COD5)	F	6-Sep-01	3	GAC.11
5220C	Chemical Oxygen Demand (COD)	F	26-Mar-02	3	GAC.12
	Percent Solids	F	25-Jan-02	2	GAC.14
120.1	Specific Conductance	F	20-Apr-02	1	GAC.15
2320B	Alkalinity-Hydroxide, Carbonate, Bicarbonate, and Total By Method 2320B	F	26-Jul-01	2	GAC.17
5310B	Total Organic Carbon By Method 5310B	F	4-Jun-02	6	GAC.20
110.2	Color, Platinum-Cobalt	F	20-Mar-02	2	GAC.24
2340C	Total Hardness by EPA Method 2340C	F	1-Nov-01	3	GAC.27
9040B,150.1	pH of Water Samples	F	24-Jul-01	3	GAC.34
365.2	Orthophosphate by Method 365.2	F	7-Dec-01	2	GAC.35
365.2	Phosphorous, Total	F	13-Feb-02	4	GAC.36
160.1	Total Dissolved Solids (TDS), Filterable Residue	F	4-Jun-02	3	GAC.37
160.2	Total Suspended Solids, Nonfilterable Residue	F	4-Jun-02	2	GAC.38
160.5	Solids, Settleable by Method 160.5	F	1-Nov-01	1	GAC.39

	Security for the ASC	F	5-Apr-02	1	A.7
SM4500-S2-E, B	Sulfide by Method SM4500-S2-E, B	F	18-Jul-01	1	GAC.43
413.1	Oil and Grease; Partition-Gravimetric, Water	F	2-Mar-02	2	GAC.48
9020B	Total Organic Halogens	F	2-Nov-01	3	GAC.49
	Lachat General Startup Procedure	F	2-Nov-01	3	GAC.64
9012A, 335.3	Total Cyanide Analysis	F	20-Nov-01	11	GAC.65
353.2	Nitrate/Nitrite by Lachat	F	4-Dec-01	3	GAC.66
1010	Ignitability (Flashpoint, Liquids Method 1010)	F	26-Mar-02	2	GAC.72
325.2	Chloride by Water Flow Injection	F	7-Dec-01	2	GAC.75
Lloyd Kahn	TOC in Sediment and Soil - Lloyd Kahn	F	1-Jun-02	5	GAC.76
	PE-1600 - FTIR Operation for Total Recoverable Petroleum Hydrocarbons	F	2-Oct-01	2	GAC.79
SM3500-CR D	Hexavalent Chromium, Colormetric Analysis by Standard Methods SM3500-CR D	F	16-Mar-02	3	GAC.82
418.1 Mod	Total Recoverable Petroleum Hydrocarbons in Soil by Sonication	F	2-Oct-01	3	GAC.83
Ch 7 Sec 7.3.3.2	Reactive Cyanide	F	14-Sep-01	1	GAC.85
9071	Extraction of Total Recoverable Petroleum Hydrocarbons by Soxhlet in Sludge, Soil, and Sediment	F	2-Oct-01	3	GAC.87
3060A	Alkaline Digestion of Soils and Subsequent Hexavalent Chromium Colorimetric Analysis	F	27-Nov-01	3	GAC.91
9095A	Paint Filter Liquids Test (Method 9095A)	F	21-Feb-02	1	GAC.92
9045C	Soil pH	F	10-Jan-02	3	GAC.100
9012A, 335.3	Total and Amenable Cyanide in Water and Soil Preparation - MIDI	F	4-Dec-01	5	GAC.106
ILM04.1	Total Cyanide in Water Preparation, CLP	F	20-Nov-01	5	GAC.118
ILM04.1	Total Cyanide in Soil Preparation, CLP	F	27-Nov-01	4	GAC.119
7196A	Hexavalent Chromium in Water, Colormetric Analysis by 7196A	F	16-Mar-02	4	GAC.121
9013	Extraction of Cyanide Using Method 9013	F	2-Nov-01	1	GAC.122
375.4	Sulfate by Method 375.4 (Turbidimetric)	F	15-Nov-01	1	GAC.125
160.3	Total Solids, Dried at 103-105 C, Total Residue	F	21-Feb-02	0	GAC.126
180.1	Turbidity (Nephelometric by Method 180.1)	F	13-Feb-02	1	GAC.127
Ch 7 Sec 7.3.4.2	Reactivity, Sulfide	F	27-Nov-01	0	GAC.130
9030A	Acid Soluble and Acid Insoluble Sulfides, Samples Amendable to Distillation	F	19-Jul-01	1	GAC.131
1110	Corrosivity Toward Steel	F	26-Jul-01	1	GAC.133
OQAAM-005-12/89	TRPH Extraction from Soil by Shaker or Soxhlet by NJDEP Procedure	F	2-Oct-01	0	GAC.136
NJDEP Mod 3060A/7196A	NJ Alkaline Digestion of Soils; Hexavalent Chomium Colorimetric Analysis of Water and Digestates	F	21-Feb-02	1	GAC.139
1030	Ignitability of Solids	F	2-Mar-02	0	GAC.140
40 CFR 50 APP B	Suspended Particulate Matter - High Volume Method	F	12-Mar-02	0	GAC.142
NIOSH 7600	Hexavalent Chromium in Air	F	4-Apr-01	0	GAC.143
	Nitrocellulose	F	26-Mar-02	0	GAC.144
300.0,9056	Anion Analysis by Ion Chromatography	F	21-Dec-01	3	GAC.145
ASTM D2974	Percent Organic Matter	F	29-Sep-01	0	GAC.146
3500-Ca C	Calcium Hardness by Method 3500-Ca C	F	2-Nov-01	0	GAC.147
9065; 420.1	Phenols by 4-AAP (Medium Distillation, Autoanalyzer Analysis)	F	14-Mar-02	0	GAC.148
DOE/HWP-65/R2	Permanganate Water Test	F	24-Apr-02	0	GAC.149
314	Perchlorate Analysis by Ion Chromatography	F	25-May-02	0	GAC.150
351.1	Block Digestion and Automated Analysis for TKN	N	NEW	0	GAC.151
SM4500-NH3 B	Distillation for Ammonia-Nitrogen	N	NEW	0	GAC.152
SM4500-NH3 D	Analysis for Ammonia-Nitrogen by Automated Phenate	N	NEW	0	GAC.153
EPA 530-R-94-036	Neutralization Potential of Solids	F	29-May-02	0	GAC.154
601	Purgeable Halocarbon Analysis by Method 601	F	7-May-02	3	GAC.155

	Security for the ASC	F	5-Apr-02	1	A.7
1311/8151A	Analysis of TCLP Extracts for Chlorinated Herbicides by Method 8151A	F	8-Jun-02	2	GC.21
8082/3580A	Sample Preparation for PCB Oil Sample Analysis	F	19-Sep-01	1	GC.26
8015B	TPH-Gasoline 8015B and California LUFT	F	13-Jun-02	5	GC.27
OLM04.2	Pesticide/PCB by GC by Method OLM04.2	F	19-Sep-01	9	GC.31
8015B/AK102	Diesel Range Organics by Method 8015B or AK102	F	13-Mar-02	5	GC.37
602	Purgeable Aromatic Analysis by Method 602	F	7-May-02	3	GC.44
AK101	TPH-Gasoline Range Organics, AK 101 Determination	F	14-Mar-01	1	GC.46
504.1, 8011	Analysis of Water Samples for EDB and DBCP by 504.1,8011	F	22-Feb-02	2	GC.49
8021B	Volatile Organic Compound Analysis by Method 8021B	F	7-May-02	5	GC.59
8151A	Analysis of Soil or Water Samples for Chlorinated Herbicides by Method 8151A	F	2-Nov-01	4	GC.60
OLC03.2	Analytical Procedure for Low Concentration CLP Pesticides/PCBs by Gas Chromatography	R	Reactivated	1	GC.61
608	Pesticide, PCB or Pesticide and PCB Analysis by Method 608	F	18-Dec-01	1	GC.64
8081A	Pesticide Analysis by Method 8081A	F	3-Nov-01	2	GC.72
8082	PCB Analysis by Method 8082	F	13-Jun-02	1	GC.73
	Sample Preparation for PCB Wipe Sample Analysis	F	2-Mar-02	0	GC.74
FL-PRO	FL Petroleum Range Organics	F	5-Oct-01	0	GC.76
TX1005	TPH by TNRCC Method TX 1005	F	6-Nov-01	0	GC.78
524.2	EPA Method 524.2 for Volatile Analysis by Purge and Trap GC/MS	F	22-Dec-01	2	GCMS.8
624	Method 624 Volatile Analysis by Purge and Trap GCMS	F	12-Mar-02	1	GCMS.11
8260B	GCMS Analysis of Samples for Volatile Organic Compounds by Method 8260B	F	21-Dec-01	10	GCMS.17
8270C	GCMS Analysis of Extracts for Semivolatile Compounds by SW-846 Method 8270C	F	12-Mar-02	12	GCMS.19
625	Semivolatile Analysis of Water Extracts by GC/MS Method 625	F	25-Aug-99	0	GCMS.20
OLM04.2	CLP Analysis for Semivolatile Samples by Method OLM04.2	F	28-Feb-01	6	GCMS.23
OLM04.2	CLP Volatile Analysis by Purge and Trap by Method OLM04.2	F	29-Jan-02	7	GCMS.24
TO-14	Analysis of Volatile Organics in Air Using Summa Polished Canisters	F	21-Dec-01	2	GCMS.29
OLC03.2	Analysis of Low Concentration Water for Semivolatile Organic Compounds	R	Reactivated	2	GCMS.35
OLC03.2	CLP Volatile Analysis of Low Concentration Water by Purge and Trap by Method OLC03.2	R	Reactivated	2	GCMS.36
1624C	GC/MS Analysis of Samples for VOCs by Method 1624C	F	13-Feb-02	1	GCMS.38
1625	Method 1625 Analysis of Semivolatile Organics by GC/MS Isotope Dilution	F	4-Apr-01	0	GCMS.39
1311/8270C	GCMS Analysis of TCLP Extracts for Semivolatile Compounds Method 8270C	F	20-Mar-02	1	GCMS.42
	GC/MS Data Archive, Storage, and Security	F	13-Feb-02	0	GCMS.44
8310	Determination of PAH by HPLC	F	6-Oct-01	5	LC.7
NIOSH 7300,40 CFR 50 APPG	Digestion Process for Casette Air Filters	F	29-Sep-01	1	Metals.4
1311	Toxicity Characteristic Leaching Procedure	F	14-Sep-01	3	Metals.8
CA WET	California WET	F	27-Sep-01	0	Metals.15
ILM04.1	Preparation for Mercury in Water Samples by CLP Protocol	F	17-Feb-02	8	Metals.21
6010B	ICP Metals Analysis by Method 6010B	F	2-Nov-01	10	Metals.18
ILM04.1	ICP Metals by CLP Protocol	F	20-Nov-01	3	Metals.30
ILM04.1	GFAA and ICP Digestion of Solids by CLP Protocol	F	29-Sep-01	6	Metals.36
ILM04.1	GFAA and ICP Digestion of Waters by CLP Protocol	F	20-Nov-01	7	Metals.37
7000	Metals Analysis by GFAA SW-846	F	6-Mar-02	6	Metals.41
	Interelement Correction Factor Determination	F	19-Sep-01	2	Metals.59
ILM04.1	Preparation for Mercury in Soil Samples by CLP Protocol	F	13-Feb-02	7	Metals.60
200.9	GFAA for Potable Waters Low Level Method 200.9	R	Reactivated	1	Metals.63

	Security for the ASC	F	5-Apr-02	1	A.7
NIOSH 6009	Preparation for Mercury in Air from Solid Sorbent Tubes by NIOSH 6009--Block Digestion	F	14-Oct-00	0	Metals.66
7470A, 7471A	Mercury Analysis	F	4-Dec-01	0	Metals.67
9081	Cation Exchange Capacity of Soils (Sodium Acetate) by Method 9081	F	6-Mar-02	0	Metals.68
	Preparation for Mercury in Wipes	F	21-Feb-02	1	Metals.69
7470A	Preparation for Mercury in Water Samples b Method 7470A Using Block Digestor	F	15-Mar-02	1	Metals.70
7471A	Preparation for Mercury in Soil Samples by Method 7471A Using Block Digestor	F	15-Mar-02	1	Metals.71
3010A/8080A	ICP or GFAA Digestion of Waters by Methods 3010A Using Block Digestion	F	16-May-02	1	Metals.72
3050B	Digestion of Soil Samples By Method 3050B - Block Digestion	F	16-May-02	1	Metals.73
40 CFR Part 136 Method 200.7	Total Digestion of Wastewaters by 40 CFR Part 136 Method 200.7 Using Block Digestion	F	21-Feb-02	1	Metals.74
1312	Synthetic Precipitation Leaching Procedure	F	8-Mar-02	0	Metals.76
3005A	Method 3005A, SW-846 Total Recoverable Metals-Block Digestion	F	16-May-02	1	Metals.77
200.7, Rev.4.4	Analysis of Drinking Water by Method 200.7, Rev.4.4	N	New	0	Metals.78
3020A	Digestion of Waters by 3020A Using Block Digestion for GFAA Analysis	F	13-Mar-02	0	Metals.79
3050B	Digestion of Soils by 3050B Using Block Digestion for GFAA Analysis	F	19-Mar-02	0	Metals.80
40 CFR Part 136 Method 200.7	Total Recoverable Digestion of Wastewaters by 40 CFR Part 136 Method 200.7 Using Block Digestion	F	21-Feb-02	1	Metals.75
	Corrective Action Process	F	19-Feb-02	6	QA.1
	Training Program	F	2-Aug-01	9	QA.5
	Standard Operating Procedures, Use and Administration	F	9-Mar-02	11	QA.18
	Internal Audits	F	15-Dec-01	6	QA.19
	Control Charting	F	14-Feb-01	4	QA.20
	CLP Data Deliverables for Organics Analysis	F	26-Jan-02	5	Rep.6
	Job Case Filing	F	23-Apr-02	4	Rep.8
	CLP Complete SDG File (CSF) Assembly	F	4-Dec-01	4	Rep. 10
	Data Entry and Changes to CLP Data and Deliverables	F	2-Nov-01	3	Rep.12
	Standard Data Package Deliverables	F	13-Jun-02	0	Rep.20
	CLP Data Deliverables for Inorganics Analysis	F	7-Dec-01	4	Rep.7
	Sample Receipt	F	9-Mar-02	6	SM.1
	Sample Log In	F	4-May-02	9	SM.2
	Sample Custody Procedure	F	11-Jul-01	7	SM.4
	Sample Splitting	F	2-Mar-02	0	SM.10
	Completion of USEPA CLP Sample Log-in (Form DC-1)	F	2-Mar-02	1	SM.11
	New Jersey Chain of Custody Procedures	F	13-Jun-02	1	SM.12
	Radiation Monitoring of Coolers and Samples	F	30-May-02	0	SM.13

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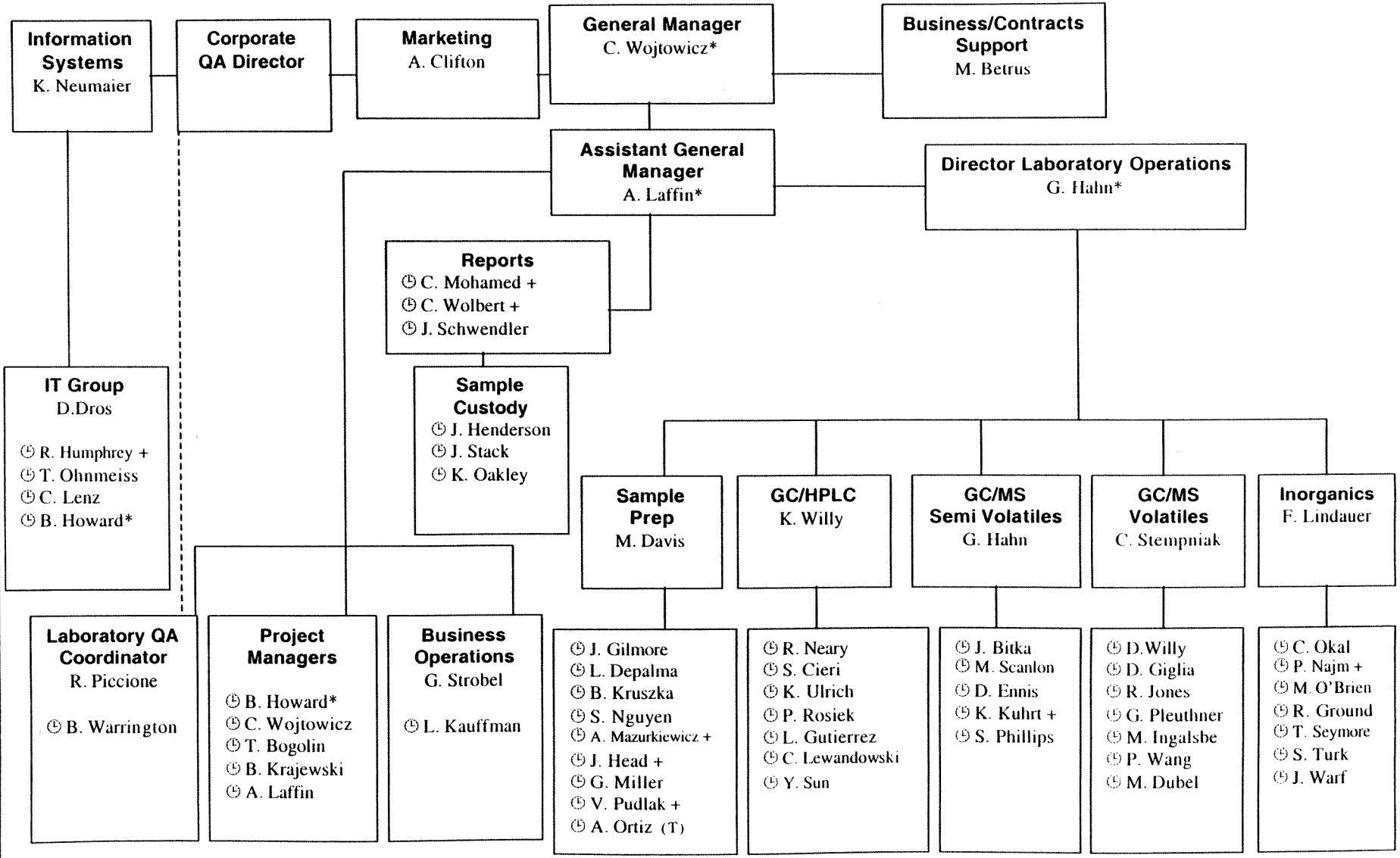
G

Organization Chart with Current Staffing



* Dual Assignment
\+ Part Time
T E & E Temporary

ASC ORGANIZATIONAL PLAN

Quality Assurance Project Plan
Attachment B
Ecology and Environment Inc., Analytical Services Center, Analytical Standard Operating Procedures for Analytical Methods

TITLE: Anion Analysis By Ion Chromatography			
Reviewer: P. Najm		Peer Reviewer: F. Lindauer	
Implementation Date: 12/21/00		Last Annual Review Date: 12/21/00	
Revision: 3	Status: Final	Method: SW-846, 9056; 300.0	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP details the ion chromatography analysis of samples for chloride, nitrate-N, nitrite-N, orthophosphate, fluoride, bromide, and sulfate. The QC criteria in Method 300.0 (as distinguished in this SOP) must be used for the analysis of SPDES, NPDES, or regulatory drinking water samples.

2.0 METHOD SUMMARY

2.1 Samples are analyzed using ion chromatography.

2.2 A 100-µL of sample is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center, Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Method 300.0, Rev. 2.1, Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.

4.2 Method 9056 (Rev. 0, 9/94), SW-846 Update III, June 1997.

4.3 Training SOP QA.5, A.23, and A.29.

4.4 Data Review SOP A.25.

4.5 Laboratory Quality Assurance Manual (L-001).

4.6 MDL SOP A.18.

4.7 Corrective Action Procedure, QA.1.

4.8 Chemical Hygiene Plan (L-009).

4.9 See SOP A.10, Waste Disposal Plan.

4.10 Use checklist number C-166 for initial and peer review.

5.0 DEFINITIONS/ACRONYMS

5.1 **MDL** - Method Detection Limit as determined by 40 CFR Part 136, Appendix B. Method detection limits must be determined yearly for both water and soil matrices. See SOP A.18 for procedure.

5.2 **PQL** - Practical Quantitation Limit is the concentration above the MDL that can be reasonably obtained and is used as the limit of reporting (Table 11-1).

5.3 **Batch** - A group of samples that behave similarly with respect to the sampling or testing procedures being employed which are processed as a unit. If the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

5.4 **Laboratory Reagent Blank (LRB) a.k.a. Method Blank (MB)** - An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is used to document contamination resulting from an analytical process.

5.5 **Matrix** - The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

5.6 **Matrix Spike (MS)** - An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix. Method 300.0 terminology is Laboratory Fortified Sample Matrix. (LFM).

5.7 **Laboratory Control Sample (LCS)** - A known matrix spiked with an analyte(s) representative of the target analytes. This is used to document laboratory performance. Method 300.0 terminology is Laboratory Fortified Blank (LFB).

5.8 **Instrument Performance Check Solution (IPC)** - A solution of method analytes used to evaluate performance of the instrument. These are designated as initial calibration verification (ICV) (a second source material) and continuing calibration verification (CCV) by the laboratory.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 High level organic or inorganic acids in industrial waste may interfere with inorganic anion analysis.

6.2 Bromide and nitrate may interfere with each other.

6.3 Reagent solutions or samples that contain visible particles require filtration. Filter solution or sample using a 10-cc syringe equipped with a 0.20-µm syringe filter.

6.4 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions.

6.5 The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

6.6 Compounds that are unretained by the column, such as low molecular weight organic acids (formate, acetate, propionate, etc.) are conductive and coelute with or near fluoride. The fluoride quantitation would be biased in some drinking and most wastewaters.

7.0 INSTRUMENTATION AND EQUIPMENT

Ion Chromatography System equipped with conductivity detector – Dionex ED40
Hewlett Packard 1050 Autosampler and pump
Hewlett Packard Vectra QS/165 data system
10-mL, 50-mL, 100-mL, 200-mL, 1000-mL volumetric flasks
1-mL, 2-mL Crimp-top autosampler vials
Columns: Anion Guard Column; Metachem ANI Guard
Anion separator column: Metachem ANI 250 x 4.6 mm
Ion suppresser: Dionex ASRS Ultra 4 mm

Volumetric pipettes
Graduated pipettes
Analytical balance readable to .0001 g
Vortex mixer
Shaker (for soils)
0.2-μm Filter

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard, Reagents, and Consumables

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
ASTM Type II Water (analyte-free standard matrix)	ASC Water Purification System	Neat
Sodium Carbonate (Na_2CO_3) ACS Grade	Fisher or equivalent	Dried at 105 ± 5 C for 30 minutes minimum
Sodium Bicarbonate ($NaHCO_3$) ACS Grade	Fisher or equivalent	Dried at 105 ± 5 C for 30 minutes minimum
1,000 mg/L Mixed Anion Standard	Accustandard IS 4957 or equivalent F, Cl, NO_2-N, Br, NO_3-N, PO_4, SO_4	1000 mg/L
Mixed Anion Standard (Second Source)	Metachem or equivalent F, Cl, NO_2-N, Br, NO_3-N, PO_4, SO_4	1000 mg/L

Revision: 3	Status: Final	Method: SW-846, 9056; 300.0	Minor Revision Date: None

8.2 Standard Solution Preparation

The shelf life of all stock standards and reagents is one year unless otherwise noted. Running standards are prepared fresh daily.

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
Eluent/Mobile phase	N/A	Dissolve 0.764 g Na_2CO_3 and 0.573 g of $NaHCO_3$ into 4L of ASTM Type II water. De-gas the eluent with helium for approximately 20 minutes.	1.8 mM Na_2CO_3/1.7 mM Na_2HCO_3
2nd Source Reference Stock Intermediate	Mixed Anion Standard, 2nd Source	10 mL of Mixed Anion Standard, 2nd Source, in 1 L ASTM Type II water	10 mg/L
Calibration Standard 5	1000 mg/L Mixed Anion Standard	10 mL of 1000 mg/L Mixed Anion Standard in 1 L ASTM Type II water	10 mg/L
Calibration Standard 4	Calibration Standard 5	200 μL of Calibration Standard 5 + 200 μL of ASTM Type II water	5.0 mg/L

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
Calibration Standard 3 and CCV	Calibration Standard 5	100 µL Calibration Standard 5 to 900 µL ASTM Type II water	1.0 mg/L
Calibration Standard 2	Calibration Standard 4	100 µL of Calibration Standard 4 + 900 µL of ASTM Type II water	0.5 mg/L
Calibration Standard 1	Calibration Standard 3	100 µL of Calibration Standard 3 + 900 µL of ASTM Type II water	0.1 mg/L
Preparation Blank	ASTM Type II Water	ASTM Type II water	0 mg/L
Initial Calibration Verification (ICV)	2nd Source Reference Stock Intermediate	100 µL of 2nd Source Reference Stock Intermediate + 900 µL ASTM Type II water	1.0 mg/L
LCS	2nd Source Reference Stock Intermediate	200 µL of 2nd Source Reference Stock Intermediate + 200 µL ASTM Type II water	5.0 mg/L
Matrix Spike (MS)	2nd Source Reference Stock Intermediate	200 µL of Anion 2nd Source Reference Stock Intermediate + 200 µL of sample or appropriate dilution to analyze species on scale.	5.0 mg/L added

Revision: 3	Status: Final	Method: SW-846, 9056; 300.0	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/ Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Solid	Standard	No method hold times exist. Laboratory imposed hold time is 28 days.	Extract for nitrate, nitrite and orthophosphate must be analyzed within 48 hours from extraction. Analyze extract for other species and combined (total) nitrate/nitrite within 28 days of extraction.	4 oz. glass jar 2-6 C
Water	Standard	N/A	Samples for anions must be analyzed within 28 days from the date of collection, except as noted below. • Nitrate, nitrite, and orthophosphate must be analyzed within 48 hours from collection.	Store at 2-6 C

Revision: 3	Status: Final	Method: SW-846, 9056; 300.0	Minor Revision Date: None

10.0 PROCEDURE

10.1 Apparatus Setup

10.1.1 The Ion Chromatograph (IC) is equipped with a guard column, separator column, suppresser, and a conductivity cell detector.

10.1.2 The eluent flow rate is 1.5 mL/min.

10.1.3 Nitrogen pressure is 100 psi to control injector valve and is nominally 7 psi (7-15 range) for regeneration solution.

10.1.4 Run time is approximately 15 min.

10.1.5 The autosampler injects 100 µL of sample.

10.2 Initial Calibration

10.2.1 Prepare the calibration standards as directed in Table 8-2.

10.2.2 Perform the initial calibration at the five levels found on Table 10-2, low to high by analyzing 100 µL of each standard.

10.2.3 Fit the response of the calibration standards to a linear regression curve. The correlation coefficient must be ≥ 0.995.

10.2.4 After completion of initial calibration, an initial calibration verification standard (ICV) must be analyzed. The ICV must contain all analytes of interest and be within the criteria specified in Table 12-1 for Method 9056 or 300.0. The ICV must be prepared from a source other than that used for initial calibration standards.

Table 10-2

INITIAL CALIBRATION CONCENTRATIONS (mg/L)

Analyte	1	2	3	4	5
Chloride	0.10	0.50	1.0	5.0	10.0
Nitrate-N	0.10	0.50	1.0	5.0	10.0
Sulfate	0.10	0.50	1.0	5.0	10.0
Nitrite-N	0.10	0.50	1.0	5.0	10.0
Fluoride	0.10	0.50	1.0	5.0	10.0
Orthophosphate	0.10	0.50	1.0	5.0	10.0
Bromide	0.10	0.50	1.0	5.0	10.0

Revision: 3	Status: Final	Method: SW-846, 9056; 300.0	Minor Revision Date: None

10.3 Retention Times

10.3.1 The daily retention time windows are established by using the retention time from the daily calibration standards as the mid-point. The daily retention time windows are defined as the mid-point ± three times the standard deviation (derived from a retention time study) from the retention time of the daily standard.

10.3.2 The width of the retention time window used to make identifications is based on measurements of actual retention time variations of standards over the course of a day. Instrument method settings are at 8%. The used windows are a sum of an absolute part in minutes and a relative part given in percent of the expected retention time. The percentage part compensates for variations in retention time that are proportional to the expected retention time. The absolute part compensates for variations that are independent from retention time. The absolute part specifies the complete RT variation both negative and positive shifts of peak elution.

10.4 Daily and Continuing Calibration Verification

10.4.1 At the beginning of each working day analyze an initial laboratory reagent blank to verify a clean system.

10.4.2 Analyze a Continuing Calibration Verification (CCV). The CCV must be within the criteria of Table 12-1 dependent upon the criteria specified for Method 9056 or 300.0.

10.4.3 If the CCV standard does not meet the above criteria, the CCV may be reanalyzed. Minor maintenance or standard repreparation may be performed between the first and second attempts, and MUST be documented. If the standard fails again, a new calibration curve must be analyzed for the failed analyte.

10.4.4 Analyze a continuing calibration verification (CCV) standard every 10 analyses or every 4 hours, whichever is more frequent, and at the end of the analytical sequence. CCV must be within the criteria of Table 12-1.

10.4.5 If the CCV fails for an analyte, analysis must cease for that analyte until the cause is determined and corrected. All samples analyzed since the last acceptable CCV must be reanalyzed.

10.5 SOIL EXTRACTION PROCEDURE

10.5.1 Weigh out 2 to 3 g to the nearest 0.01g of blended soil sample in a 40-mL VOA vial.

10.5.2 Add 25-mL of ASTM Type II water to the soil sample.

10.5.3 Vortex for 20 seconds and mix for 30 minutes using a mechanical shaker.

10.5.4 Centrifuge and filter using 0.2micron X 25mm Gelman 4496 Acrodisc or equivalent filter.

10.5.5 Follow analysis procedure Section 10.6.

10.6 Sample Analysis Procedure

10.6.1 A run sequence may be up to 24 hours in length.

10.6.2 The daily sequence is as follows:

- Laboratory Reagent Blank (aka method blank)
- CCV
- LCS (aka LFB)
- Method Blank for soils
- Analysis (not to exceed 10 samples; includes QC samples)
- CCV
- Analysis of 10 samples and CCV until completion of run
- Sample analysis procedure will end at 24 hours

10.6.3 Inject 100 µL of each sample extract and evaluate quantitation report.
10.6.4 Dilute and reanalyze any sample that has a target compound concentration in excess of the calibration curve.
10.6.5 If insufficient sample is available for MS/Duplicate or MS/MSD then analyze an LCS/LCSD instead.

Instrument Conditions and Maintenance

Table 10-3

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Preventative Maintenance or Repair	Frequency
HP Pump in-line frit	High Pressure	Replace in-line frit	1 per week or as needed
HP 1050 Injector	High Pressure	Replace needle seat capillary	As needed
HP 1050 Pump	High Pressure	Replace capillary tubing	As needed
Dionex-IC	High Pressure	Replace Guard Column frit	Bi-monthly dependent on amount of samples run
Suppressor		Regeneration is possible. See manual. Solution for this process is dependent upon suspected contaminant.	

Revision: 3	Status: Final	Method: SW-846, 9056; 300.0	Minor Revision Date: None

11.0 DATA REDUCTION/EVALUATION/REPORTING

Target Compounds and Reporting Limits
See Table 11-1, Target Compounds and Quantitation Limits.

11.1 Water Samples

Sample results are printed automatically in mg/L. The following equation is provided as a check.

$$mg/L = [(A_x - b)/m] \cdot df$$

where:

A_x = Area response of analyte in sample.
m = Slope of curve from regression analysis of standard curve (area/conc).
B = y-Intercept from regression analysis of standard curve (area).
Df = Dilution factor.

11.2 Soil Samples

Sample results are printed automatically in mg/kg using appropriate factors in the software. The following equation is provided as a check.

$$mg/kg = \left[(A_x - b)/m\right] \cdot \frac{V}{W} \cdot df$$

where:

V = Volume (mL) of aqueous extract (generally 25 mL).
W = Weight (g) of soil extracted (generally 2.5 g).
A_x = Area response of analyte in sample.
m = Slope of curve from regression analysis of standard curve (area/conc).
b = y-Intercept from regression analysis of standard curve (area).
df = Dilution factor.

11.3 Results as Phosphate-P

11.3.1 This SOP provides results as orthophosphate (PO_4^{3-}). For clients who request data as phosphate-P, multiply results by 0.3261. (Ratio AW P/MW PO_4^{3-})

11.3.2 This SOP provides results for nitrate as nitrogen (NO_3^--N) and nitrite as nitrogen (NO_2^--N). Add NO_3-N and NO_2^--N to report as NO_3^-/NO_2^- as nitrogen.

11.3.3 If a client requests nitrate in terms of the nitrate ion, multiply NO_3^--N result by 4.427.

11.3.4 If a client requests nitrite in terms of the nitrite ion, multiply NO_2^--N result by 3.284.

11.4 % Recovery

$$\%\,\text{Recovery} = \frac{\text{Amount Found (mg/L)}}{\text{Amount Added (mg/L)}} \cdot 100$$

Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL	
		Water mg/L	Soil mg/Kg
Chloride	T,S,L	0.10	1.0
Nitrate-N	T,S,L	0.10	1.0
Sulfate	T,S,L	0.10	1.0
Nitrite-N	T,S,L	0.10	1.0
Fluoride	T,S,L	0.10	1.0
Orthophosphate	T,S,L	0.10	1.0
Bromide	T,S,L	0.10	1.0
Revision: 3	Status: Final	Method: SW-846, 9056; 300.0	Minor Revision Date: None

Key Type:

T = Compound/analyte is target compound routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike.
L = Compound/analyte is routinely used as a LCS spike.
Q = Compound/analyte is used as a surrogate spike.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1

ACCEPTANCE CRITERIA FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
LCS (Method 9056) LFB (Method 300.0)	1 per 20 samples or less	Table 8-2	Method 9056: See Quality Control Limit Summary, Anions Method 300.0: 90-110% recovery	Reanalyze entire batch.
MS/MSD (Method 9056) LFM/LFM Duplicate (Method 300.0)	1 pair per 20 samples or less 1 pair per 20 samples or less	Table 8-2	Method 9056: Project requirements if specified. Method 300.0: 90-110% recovery Water: 20 RPD Soil: 35 RPD	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
ICV	After initial curve	Table 8-2	Method 9056: ± 10% of true value Method 300: ± 10% of true value	Repeat; recalibrate.
CCV	At beginning of run, after every 10 samples, and at end	Table 8-2	Method 9056: ± 15% of true value Method 300.0: ± 10% of true value	Repeat; reanalyze samples from last acceptable CCV.
Method Blank (Method 9056) LRB (Method 300.0)	Start of analytical sequence	Water: ASTM Type II water Soil: ASTM Type II water	< PQL	Inspect system and repeat. Reanalyze entire batch.

Revision: 3	Status: Final	Method: SW-846, 9056; 300.0	Minor Revision Date: None

Table 12-2

CONTROL CHARTED QUALITY CONTROL SAMPLES

QC Type		Analytes Charted	Matrix Charted
LCS		% Recovery	Water
Revision: 3	Status: Final	Method: SW-846, 9056; 300.0	Minor Revision Date: None

13.0 SPECIAL PROJECT REQUIREMENTS

13.1 Client requirements for Quantitation Limits vary therefore the calibration and reference concentrations are adjusted to meet client needs.

13.2 **Client: Jacobs Engineering**. Requires LCS.

13.3 **U.S. Army Corps of Engineers (ACE)**

13.3.1 Method Quantitation Limit (MQL) is between 3-10 times MDL. The low calibration standard concentration must be ≥3 times MDL. The Method Reporting Limit (MRL) may slide between two times the MDL and project-specific or regulatory requirements. Results below the MRL must be reported "ND." Results between the MQL and MRL must be reported as estimated (J flagged).

14.0 SAMPLE DISPOSAL

14.1 Dispose of all wastes per the Waste Disposal Plan, SOP A10.

14.2 In general, waste may be poured down drain with copious amounts of water.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: GCMS Analysis of Extracts for Semivolatile Compounds by SW-846 Method 8270C			
Reviewer: G. Hahn		Peer Reviewer: J. Bitka	
Implementation Date: 4/30/01		Last Annual Review Date: 3/9/01	
Revision: 12	Status: Final	Method: 8270C	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP details the capillary column GCMS analysis of extracted water and soil samples for Base/Neutral/Acid (BNA) or semivolatile extractable organic compounds using SW-846 procedures.

1.2 Method 8270C can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

1.3 In most cases, Method 8270C is not appropriate for the quantitation of multicomponent analytes, e.g., aroclors, toxaphene, chlordane, etc., because of limited sensitivity for those analytes.

1.4 Quantitation limits are presented in Table 10-4. Quantitation limits will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.5 The laboratory's usual reporting list is presented in Table 10-4. Other compounds may be added at the request of the client.

1.6 Details regarding method development and performance can be found in Method 8270C, Section 9.0.

2.0 METHOD SUMMARY

2.1 Semivolatile compounds are introduced from a sample extract (EXT.12, EXT.20, EXT.46) into the gas chromatograph by direct injection of 2 µL of the extract. Sample extracts are prepared for analysis by transferring a measured aliquot of extract to an autosampler vial and adding the appropriate amount of internal standard solution. Chromatographic conditions are such that the compounds are separated by the gas chromatograph. The compounds are detected using a mass spectrometer from which both qualitative and quantitative information is obtained. Quantitation is by the internal standard technique using relative response factors.

2.2 Method deviations are listed below.

2.3.1 Samples are not routinely reextracted for surrogate failure provided no more than one surrogate per fraction fails to meet criteria and recoveries are greater than 10%.

2.3.2 Samples are not routinely reextracted if levels of common phthalates are detected in the method blanks provided the levels detected are less than 5 times the quantitation limit and phthalates are not of critical concern for the project. A corrective action report must be generated and given to the project manager to make the determination. Application may be limited depending on the client (see Section 13).

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 SW-846 3rd Ed., Update III, June 1997, Method 8270C.
4.2 USACE, Shell for Analytical Chemistry Requirements, Version 1.0, November 2, 1998.
4.3 Training SOP QA.5, A.23, and A.29.
4.4 Data Review SOP A.25.
4.5 Laboratory Quality Assurance Manual (L-001).
4.6 MDL SOP A.18.
4.7 Corrective Action Procedure, SOP QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 **MDL** - Method Detection Limit as determined by 40 CFR Part 136, Appendix B. Method detection limits must be determined yearly for both water and soil matrices. See SOP A.18 for procedure.
5.2 **PQL** - Practical Quantitation Limit for reporting purposes; lowest calibrated concentration above the MDL.
5.3 **DFTPP** – Decafluorotriphenylphosphine

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 The sample matrix may cause chromatographic problems – High baseline, poor internal standards (IS) recovery, poor surrogate standard recovery, and carryover can often be directly attributed to the matrix of the sample. These problems can be confirmed by reanalysis and/or reextraction of the sample.
6.2 If sample matrix effects are expected for the project or site, a gel permeation chromatography (GPC) cleanup may be required to remove high-molecular weight compounds. Verify with project manager for project-specific requirements. Follow SOP EXT.73.
6.3 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.
6.4 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in solution, and photochemical decomposition.
6.5 N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.
6.6 N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.
6.7 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
6.8 Pyridine may perform poorly at the GC injection port temperatures listed in the method. Lowering the injection port temperature may reduce the amount of degradation. The analyst needs to use caution if modifying the injection port temperature as the performance of other analytes may be adversely affected.
6.9 When an interference is present on the primary quantitation ion, a secondary ion should be used to quantitate the target analyte. This will require establishing a 5-point calibration using the secondary ion and following the procedures in Sections 10.3 and 10.4.

7.0 INSTRUMENTATION AND EQUIPMENT

7.1 Hewlett-Packard 5970, 5972, and 5973 GC/MS equipped with HP Autosampler.
7.2 Hewlett-Packard HP1000 Series or Chemstation Data System/UNIX Series with "Target 3" software.
7.3 Restek 30-m, 0.25 mm fused silica column with 0.50-um RTX-5MS (or equivalent)
7.4 Varian Digital Gas Flow Meter.

8.0 REAGENTS AND MATERIALS

8.1 Summary of Standards, Reagent, and Consumables

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
DFTPP Std	Supelco or equivalent	50 ng/µL each of DFTPP, 4,4' – DDT, pentachlorophenol, and benzidine.
BN #1 Mix	Supelco or equivalent	2000 ng/µL each component
BN #2 Mix	Supelco or equivalent	2000 ng/µL each component
HSL #1 Mix	Supelco or equivalent	2000 ng/µL each component
HSL #2 Mix	Supelco or equivalent	2000 ng/µL each component
PAH Mix	Supelco or equivalent	2000 ng/µL each component
Phenols/Benzidine Mix	Supelco or equivalent	2000 ng/µL each component
BN Surrogate Mix	Supelco or equivalent	5000 ng/µL each component
AP Surrogate Mix	Supelco or equivalent	10,000 ng/µL each component
Pyridine	Accustandard or equivalent	200 ng/µL
SV Internal Standard	Restek or equivalent	2000 ng/µL each component
Methylene Chloride ($MeCl_2$)	Baker Ultra Resi or equivalent	Neat

Revision: 12	Status: Final	Method: 8270C	Minor Revision Date: None

8.2 Standard Solution Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
BNA 200 ng	BN#1 Mix BN#2 Mix HSL#1 Mix HSL#2 Mix Phenol/ Benzi-dine Mix PAH Mix AP Surrogate Mix Pyridine BN Surrogate Mix	200 µL 200 µL 200 µL 200 µL 200 µL 200 µL 40 µL 200 µL 80 µL All diluted to 2.0 mL with methylene chloride	200 ng/µL
SV Internal Standard (SVIS)	SV Internal Standard (SVIS)	NA	2000 ng/µL

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
SSTD120*	BNA 200 ng/µL	300 µL to 1 mL $MeCl_2$	60 ng/µL
SSTD100*	BNA 200 ng/µL	250 µL to 1 mL $MeCl_2$	50 ng/µL
SSTD080*	BNA 200 ng/µL	200 µL to 1 mL $MeCl_2$	40 ng/µL
SSTD060*	BNA 200 ng/µL	150 µL to 1 mL $MeCl_2$	30 ng/µL
SSTD050*	BNA 200 ng/µL	125 µL to 1 mL $MeCl_2$	25 ng/µL
SSTD 020*	BNA 200 ng/µL	50 µL to 1 mL $MeCl_2$	10 ng/µL
Revision: 12	Status: Final	Method: 8270C	Minor Revision Date: None

*To each standard add 10 µL of SVIS. The resulting concentration of each internal standard is 20 ng/µL.

8.3.1 Storage and Expiration

8.3.2 Store all stocks and standards in the GC/MS freezer. Store working standards at -10 C or less.

8.3.3 Stocks expire 3 months from date opened depending on stability.

8.3.4 Working Standard expires 2 weeks from date prepared.

8.3.5 No prepared standard may have an expiration date that is beyond the expiration date of its starting material(s).

8.3.6 Replace sooner if comparison versus independent check standards and/or versus previous standards indicates a discrepancy. Discard the questionable standard.

8.3.7 Before each use, inspect the expiration date on the vial label.

8.3.8 Discard expired standards.

Table 8.2.1

SUMMARY OF STOCK STANDARDS

Parent Stock	Components
Supelco BN#1 (2000 µg/mL each component in methylene chloride)	Bis(2-chloroethoxy)methane
	Bis(2-chloroethyl)ether
	Bis(2-ethylhexyl)phthalate
	Bis(2-chloroisopropyl)ether
	4-Bromophenylphenylether
	Butylbenzylphthalate
	4-Chlorophenylphenylether
	Diethylphthalate
	Dimethylphthalate
	Di-n-butylphthalate
	Di-n-octylphthalate
	N-nitrosodimethylamine
	N-nitrosodi-n-propylamine
	N-nitrosodiphenylamine

Table 8.2.1

SUMMARY OF STOCK STANDARDS

Parent Stock	Components
Supelco BN#2 (2000 µg/mL each component in methylene chloride)	Carbazole
	2-Chloronaphthalene
	1,2-Dichlorobenzene
	1,3-Dichlorobenzene
	1,4-Dichlorobenzene
	2,4-Dinitrotoluene
	2,6-Dinitrotoluene
	Hexachlorobenzene
	Hexachlorobutadiene
	Hexachlorocyclopentadiene
	Hexachloroethane
	Isophorone
	Nitrobenzene
	1,2,4-Trichlorobenzene
Supelco HSL#1 (2000 µg/mL each component in methylene chloride)	Benzoic acid
	2-Methylphenol
	4-Methylphenol
	2,4,5-Trichlorophenol
Supelco HSL#2 (2000 µg/mL each component in methylene chloride)	Aniline
	Benzyl alcohol
	4-Chloroaniline
	Dibenzofuran
	2-Methylnaphthalene
	2-Nitroaniline
	3-Nitroaniline
	4-Nitroaniline
Supelco PAH (2000 µg/mL each component in methylene chloride: benzene (50:50))	Acenaphthene
	Acenaphthylene
	Anthracene
	Benzo(a)anthracene
	Benzo(b)fluoranthene
	Benzo(k)fluoranthene
	Benzo(ghi)perylene
	Benzo(a)pyrene
	Chrysene
	Dibenzo(a,h)anthracene
	Fluoranthene
	Fluorene
	Indeno(1,2,3-cd)pyrene
	Naphthalene
	Phenanthrene
	Pyrene

Table 8.2.1

SUMMARY OF STOCK STANDARDS

Parent Stock	Components
Supelco Phenols/Benzidine Mix (2000 µg/mL each component in methylene chloride)	4-Chloro-3-methylphenol
	2-Chlorophenol
	2,4-Dichlorophenol
	2,4-Dimethylphenol
	2,4-Dinitrophenol
	2-Methyl-4,6-dinitrophenol
	2-Nitrophenol
	4-Nitrophenol
	Pentachlorophenol
	Phenol
	2,4,6-Trichlorophenol
	Benzidine
	3,3'-Dichlorobenzidine
Supelco BN Surrogate (5000 µg/mL each component in methylene chloride)	2-Fluorobiphenyl
	Nitrobenzene-d5
	4-Terphenyl-d14
Supelco AP Surrogate (10000 µg/mL each component in methanol)	2-Fluorophenol
	Phenol-d5
	2,4,6-Tribromophenol
Restek SV IS (2000 µg/mL each component in methylene chloride)	Acenaphthene-d10
	Chrysene-d12
	1,4-Dichlorobenzene-d4
	Naphthalene-d8
	Perylene-d12
	Phenanthrene-d10
Pyridine (2000 µg/mL each component in methylene chloride)	Pyridine

Revision: 12	Status: Final	Method: 8270C	Minor Revision Date: None

8.4 Materials

- 2-mL autosampler crimp-cap vials with PFTE-lined cap, 2-mL screw cap vials with PFTE-lined cap
- Syringes: 10-µL, 25-µL, 100-µL, 250-µL, 500-µL, and 1.0-mL
- Wiretrol Pipettes: 10-µL, 20-µL, 50-µL, 100-µL, and 200-µL
- Disposable Pasteur pipettes
- Volumetric flask: 1-mL and 2-mL
- Helium, Ultra High Purity

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/ Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
All	Standard	7 water, 14 soil	40 days from extraction	Store extracts in a secure area (GSP Storage) at <6 C. 2-mL screw-cap vial.
Revision: 12	Status: Final	Method: 8270C	Minor Revision Date: None	

10.0 PROCEDURE

10.1 Instrument Conditions and Settings (Settings may vary somewhat to optimize analytical separation).

10.1.1 The GC/MS is fitted with a split/splitless injection port. The port normally operates in split mode with a change to splitless mode during injection and for an additional 0.50 min. after injection.

10.1.2 Set instrument temperatures as follows:

Injector = 275 C Detector = 275 C

10.1.3 The 30-m column is attached at the injection port and directly into the GC/MS source. The injection port flow/pressure settings are as follows:

Head pressure = 5 psi
Split vent = 50-60 mL/min.
Purge vent = 1-2 mL/min.

10.1.4 Temperature ramp as follows: Initial temperature at 50 C hold 4 min.; ramp 8 C/min. to 300 C, hold for 13 minutes. Total run time is approximately 48 minutes.

10.1.5 Set autosampler to inject 2 µL of sample extract into the injection port of the GC.

10.1.6 Set the autosampler to rinse a minimum of 3 times with methylene chloride after sample injection.

10.1.7 The above values are guidelines and may be adjusted to optimize chromatography as required.

Instrument Maintenance

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Gas Chromatograph	Contamination Suspected	Change septum Clip column Change injection port liner and seal	Daily (or as needed)
GC column	Problems calibrating and/or severe contamination	Change column	As needed
Mass Spectrometer	High background	Bake source	As needed
Mass Spectrometer	Can not calibrate	Clean source	As needed
Revision: 12	Status: Final	Method: 8270C	Minor Revision Date: None

10.2 Mass Spectrometer Tuning Requirement

10.2.1 The scan range is 35-500 amu every 1 second or less, utilizing 70 eV nominal.

10.2.2 Inject one (1) µL of DFTPP (50 ng/µL) into the GC. This standard contains 50 ng/µL each of 4,4'-DDT, pentachlorophenol and benzidine to verify injection port inertness and column performance. The time of injection defines the beginning of a 12-hour clock.

10.2.3 Select the scan which corresponds to the top of the DFTPP peak, average it with the scan before and after, and subtract from it one scan from the background region before the peak. The resulting scan constitutes the DFTPP tune check scan.

10.2.4 Evaluate the DFTPP tune check scan against the criteria in Table 10-2.

10.2.5 If tuning criteria cannot be met, do not proceed until corrective action is taken. This may include retuning or cleaning the source. If necessary, manually tune instrument using tuning compound PFTBA. Repeat from Step 10.2.2

Table 10-2

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA FOR QUADRAPOLE MASS SPECTROMETERS

Mass	Ion Abundance Criteria
51	30 – 60% of mass 198
68	< 2 of mass 69
70	< 2 of mass 69
127	40 – 60% of mass 198
197	< 1% of mass 198
198	Base peak, 100 percent relative abundance
199	5 - 9% mass 198
275	10 – 30% mass 198
365	> 1% of mass 198
441	Present, but < mass 443
442	> 40% of mass 198
443	17 – 23% of mass 442

Revision: 12	Status: Final	Method: 8270C	Minor Revision Date: None

10.2.6 Verify and document degradation of DDT to DDE and DDD does not exceed 20%. Note on SOP checklist.

10.2.7 The analyst must verify the tailing factor is less than 3 for benzidine and less than 5 for pentachlorophenol using the tune check program.

10.3 Initial Calibration

10.3.1 See compound list in Table 10-3.

Table 10-3

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION*

1,4-Dichlorobenzene-d_4	**Naphthalene-d_8**	**Acenaphthene-d_{10}**
Benzyl alcohol	Benzoic acid	Acenaphthene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy) methane	Acenaphthylene
Bis(2-chloroisopropyl)ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3 Methylphenol	4-Chlorophenylphenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	1,2,4-Trichlorobenzene	Diethyl phthalate
Phenol	2,4-Dimethylphenol	2,4-Dinitrotoluene
1,2-Dichlorobenzene	Hexachlorobutadiene	2,4-Dinitrophenol
2-Fluorophenol(surr)	Naphthalene	2,6-Dinitrotoluene
Hexachloroethane	Nitrobenzene	Fluorene
Phenol-d_5 (surr). 2-Methylphenol 4-Methylphenol N-nitrosodimethylamine N-nitroso-di-n-propylamine Aniline*	Nitrobenzene-d_5 (surr.) 2-Nitrophenol Isophorone 2-Methylnaphthalene	2-Fluorobiphenyl (surr) Hexachlorocyclopentadiene 2,4,5-Trichlorophenol 2,4,6-Trichlorophenol 2-Nitroaniline 3-Nitroaniline 4-Nitroaniline 4-Nitrophenol 2,4,6-Tribromophenol (surr). Dimethylphthalate
Phenanthrene-d_{10}	**Chrysene-d_{12}**	**Perylene-d_{12}**
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenylphenylether	Bis(2-ethylhexyl) phthalate	Benzo(ghi)perylene
Di-n-butylphthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methyl-phenol	Chrysene	Benzo(b)fluoranthene
Fluoranthene	Pyrene	Dibenzo(a,h)anthracene
Hexachlorobenzene	Terphenyl-d_{14} (surr.)	
N-Nitrosodiphenylamine	Benzidine*	
Pentachlorophenol Phenanthrene Carbazole	3,3'-Dichlorobenzidine	
	Di-n-octylphthalate	
	Indeno (1,2,3-cd) pyrene	

Revision: 12	Status: Final	Method: 8270C	Minor Revision Date: None

(surr.) = Surrogate.

Note: *Not a routine target compound. Reported upon client request. Additional compounds can be analyzed upon client request.

10.3.2 Characteristic ions follow internal standards and target compounds (See Table 10-4).

Table 10-4

CHARACTERISTIC ION AND QUANTITATION LIMITS OF TARGET COMPOUNDS AND SURROGATES

Parameter	Primary Ion	Secondary Ion(s)	PQL (µg/kg)	PQL (µg/L)
N-Nitrosodimethylamine	74	42	330	10
Aniline**	93	65, 66	330	10
Phenol	94	65, 66	330	10
Bis(2-Chloroethyl)ether	93	63, 95	330	10
2-Chlorophenol	128	64, 130	330	10
1,3-Dichlorobenzene	146	148, 113	330	10
1,4-Dichlorobenzene	146	148, 113	330	10
1,2-Dichlorobenzene	146	148, 113	330	10
Benzyl alcohol	108	79, 77	330	10
2-Methylphenol	108	107	330	10
2,2'-oxybis(1-Chloropropane)	45	77, 79	330	10
4-Methylphenol	108	107	330	50
N-Nitroso-di-n-propylamine	70	42, 101, 130	330	10
Hexachloroethane	117	201, 199	330	10
Nitrobenzene	77	123, 65	330	10
Isophorone	82	95, 138	330	10
2-Nitrophenol	139	65, 109	330	10
2,4-Dimethylphenol	107	121, 122	330	50
Benzoic acid	122	105, 77	830	25
Bis(2-Chloroethoxy)methane	93	95, 123	330	10
2,4-Dichlorophenol	162	164, 98	330	10
1,2,4-Trichlorobenzene	180	182, 145	330	10
Naphthalene	128	129, 127	330	10
4-Chloroaniline	127	129	330	50
Hexachlorobutadiene	225	223, 227	330	10
4-Chloro-3-methylphenol	107	144, 142	330	10
2-Methylnaphthalene	141	142	330	10
Hexachlorocyclopentadiene	237	235, 272	830	25
2,4,6-Trichlorophenol	196	198, 200	330	10
2,4,5-Trichlorophenol	196	198, 200	830	25
2-Chloronaphthalene	162	164, 127	330	10
2-Nitroaniline	65	92, 138	830	25
Dimethylphthalate	163	194, 164	330	10
Acenaphthylene	152	151, 153	330	10
3-Nitroaniline	138	108, 92	830	25
Acenaphthene	153	152, 154	330	10
2,4-Dinitrophenol	184	63, 154	830	50
4-Nitrophenol	109	139, 65	830	25
Dibenzofuran	168	139	330	10
2,4-Dinitrotoluene	165	63, 182	330	10

Table 10-4

CHARACTERISTIC ION AND QUANTITATION LIMITS OF TARGET COMPOUNDS AND SURROGATES

Parameter	Primary Ion	Secondary Ion(s)	PQL (µg/kg)	PQL (µg/L)
2,6-Dinitrotoluene	165	89, 121	330	10
Diethylphthalate	149	177, 150	330	10
4-Chlorophenylphenylether	204	206, 141	330	10
Fluorene	166	165, 167	330	10
4-Nitroaniline	138	92, 108	830	25
4,6-Dinitro-2-methylphenol	198	51, 105	830	50
N-Nitrosodiphenylamine	169	168, 167	330	10
4-Bromophenylphenylether	248	250, 141	330	10
Hexachlorobenzene	284	142, 249	330	10
Pentachlorophenol	266	264, 268	830	50
Phenanthrene	178	179, 176	330	10
Anthracene	178	179, 176	330	10
Di-n-butylphthalate	149	150, 104	330	10
Carbazole	167	166, 139	330	10
Fluoranthene	202	101, 100	330	10
Benzidine**	184	92	830	50
Pyrene	202	101, 100	330	10
Butylbenzylphthalate	149	91, 206	330	20
3,3'-Dichlorobenzidine	252	254, 126	660	10
Benzo(a)anthracene	228	229, 226	330	10
Bis(2-Ethylhexyl)phthalate	149	167, 279	330	20
Chrysene	228	226, 229	330	10
Di-n-octylphthalate	149	-	330	20
Benzo(b)fluoranthene	252	253, 125	330	10
Benzo(k)fluoranthene	252	253, 125	330	10
Benzo(a)pyrene	252	253, 125	330	10
Indeno(1,2,3-cd)pyrene	276	138, 277	330	10
Dibenzo(a,h)anthracene	278	139, 279	330	10
Benzo(ghi)perylene	276	138, 277	330	10
Pyridine††	79	52	--	--
Surrogates				
Phenol-d_5*	99	42, 71	330	10
2-Fluorophenol	112	64	330	10
2,4,6-Tribromophenol	330	332, 141	330	10
Nitrobenzene-d_5	82	128, 54	330	10
2-Fluorobiphenyl	172	171	330	10
p-Terphenyl-d_{14}	244	122, 212	330	10

Table 10-4

CHARACTERISTIC ION AND QUANTITATION LIMITS OF TARGET COMPOUNDS AND SURROGATES

Parameter	Primary Ion	Secondary Ion(s)	PQL (µg/kg)	PQL (µg/L)
Internal Standards				
1,4-Dichlorobenzene-d_4	152	115	NA	NA
Naphthalene-d_8	136	68	NA	NA
Acenaphthene-d_{10}	164	162, 160	NA	NA
Phenanthrene-d_{10}	188	94, 80	NA	NA
Chrysene-d_{12}	240	120, 236	NA	NA
Perylene-d_{12}	264	260, 265	NA	NA
Revision: 12	Status: Final	Method: 8270C	Minor Revision Date: None	

QL = Quantitation Limit.
*Phenol-d6 becomes Phenol-d5 upon contact with water or methanol.
**Not a routine target compound. Reported upon client request.
†With 3-methylphenol.
††TCLP compound only.

10.3.3 Perform the initial calibration at the levels listed in Table 8-2: SSTD020, SSTD050, SSTD060, SSTD080, SSTD100, and SSTD120 levels (the calibration range may be extended with lower or higher standard concentrations).

10.3.4 Inject 2 µL of each calibration standard. Perform the initial calibration at the 6 standard levels for each analyte. Report the calibration in nanograms injected. Ten compounds (2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, 4,6-dinitro-2-methylphenol, benzoic acid, and pentachlorophenol and hexachlorocyclopentadiene) require only a 5-point initial calibration at 50, 60, 80, 100, and 120 total ng per 2 µL, since detection at less than 50 ng per injection is difficult.

10.3.5 Calculate relative response factors (RRF_x) for each compound and surrogate using the following equation:

$$RRF_x = (A_x/A_{is})(C_{is}/C_x)$$

where,

A_x = Area of the characteristic ion for the compound to be measured (Table 10-4).
A_{is} = Area of the characteristic ion for the specific internal standard (Table 10-3).
C_{is} = Amount of the internal standard (ng injected).
C_x = Amount of the compound to be measured (ng injected).

10.3.6 For each compound calculate the mean relative response factor and standard deviation (SD) using the following equation:

$$\text{Mean RRF} = \overline{RRF_x} = \frac{\sum_{i=1}^{n} RRF_i}{n}$$

$$SD = \sqrt{\sum_{i=1}^{n} \frac{\left(RRF_i - \overline{RRF_x}\right)^2}{n-1}}$$

10.3.7 Calculate the percent Relative Standard Deviation (%RSD) of the RRF_x values for the initial calibration using the following equation:

$$\%RSD = \frac{SD}{RRFx} \cdot 100$$

where:

RRF_x = Mean RRF for each compound (x) or surrogate.
RRF_I = Response factor for each calibration level for the compound/surrogate of interest.
n = Number of calibration levels.

10.3.8 The % RSD must be less than 30% for each Calibration Check Compound (CCC). System Performance Check Compounds (SPCC) must have a minimum average response factor of 0.050. The CCCs and SPCCs are listed in Table 10-5 and Table 10-6, respectively.
10.3.9 If the % RSD of any compound is 15% or less, the relative response factor is assumed to be constant over the calibration range, and the mean relative response factor may be used for quantitation.

Table 10-5

CALIBRATION CHECK COMPOUNDS (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

Revision: 12	Status: Final	Method: 8270C	Minor Revision Date: None

Table 10-6 8270C SYSTEM PERFORMANCE CHECK COMPOUNDS (SPCC)			
N-Nitroso-di-n-proplyamine			
Hexachlorocyclopentadiene			
2,4-Dinitrophenol			
4-Nitrophenol			
Revision: 12	Status: Final	Method: 8270C	Minor Revision Date: None

10.3.10 The initial calibration is valid as long as the mean of all the RSD values are less than or equal to 15%. The data user should review the associated quality control results carefully, with particular attention to the matrix spike and laboratory control sample results to determine if the calibration linearity poses a significant concern.

10.3.11 The mean RSD criterion applies to all analytes in the standards, regardless of whether or not they are of interest for a specific project.

10.3.12 The mean RSD value is documented and included with the initial calibration data.

10.3.13 If a % RSD greater than 30% is measured for any CCC, take corrective action before re-attempting calibration. If a % RSD for any other compound is greater than 30%, a regression fit may be used for the calibration curve for that compound. For a first order regression, r must be $\geq$0.990. For a second order regression, at least six calibration levels must be used and the coefficient of determination must be greater or equal to 0.990.

10.3.14 Complete runs must be used, and the entire 6-point calibration must be run under a single clock.

10.3.15 If SPCC mean response factors do not meet minimum high or criteria (see 10.3.8) the analyst must either:

- rerun the calibration points if they have anomalously high or low RRF; or
- take corrective action and perform a complete new calibration.

10.3.16 Initial Calibration Verification. The initial calibration curve shall be verified as accurate with a standard purchased or prepared from an independent source. This initial calibration verification (ICV) involves the analysis of a standard containing all of the target analytes, typically in the middle of the calibration range, each time the initial calibration is performed. The percent recovery of each target analyte in the ICV is determined from the initial calibration and should be between 70% and 130%. See Section 13 for individual client-specific quality control requirements for the ICV.

10.4 Calibration Verification Standard

10.4.1 Inject 2 µL of a BNA standard solution containing each compound at 25 ng/µL (50 ng) and internal standards at 20 ng/µL (40 ng). This corresponds to the SSTD050 standard of the initial calibration. The primary ion area counts of the internal standards should not vary by more than one half to double of the midlevel calibration run of most recent initial calibration. The absolute retention time of the internal standards should not vary by more than +/-30 seconds.

10.4.2 System Performance Check Compounds (SPCC) must have a minimum mean relative response factor of 0.050.

10.4.3 Quantitate each compound of the calibration verification standard against the mean relative response factor of the initial calibration using the following equation:

$$Cx = (A_x/A_{is})(C_{is}/\overline{RRF_x})$$

where:

A_x = Area of the primary ion for the compound to be measured (Table 10-4).
A_{is} = Area of the primary ion for the specific internal standard (Table 10-3).
C_{is} = Amount of the internal standard (ng injected).
C_x = Amount of the compound measured (ng injected).
RRF_x = Mean Average relative response factor of compound X.

10.4.4 Calculate the percent Difference (% D) value of each analyte according to the following equation:

$$\%\ D = \frac{RRF_x - \overline{RRF_x}}{\overline{RRF_x}} \cdot 100$$

where:

$\underline{RRF_x}$ = Response factor for compound x from analysis of calibration verification standard.
RRF_x = Average response factor of compound X from initial calibration.

10.4.5 If the percent difference (%D) is less than or equal to 20% for each Calibration Check Compound (CCC) and the response factors of the SPCC in the check standard are acceptable, then the initial calibration is still valid.
10.4.6 If no source of problem can be determined after corrective action has been taken, a new initial multi-point calibration must be generated.

10.5 Sample Preparation
10.5.1 Using a Wiretrol® calibrated disposable pipette, withdraw 300 µL of sample extract from the sample vial and place into an autosampler vial. Using a 10-µL Hamilton syringe, add 3 µL of SVIS solution. Load into autosampler tray for batch processing. Return the remaining original extract to GSP refrigerator.
10.5.2 Should additional analysis be required, a fresh volume of extract or diluted extract can be prepared just prior to reanalysis using the same formula of 1 µL SVIS per 100 µL sample or dilution.

10.6 Method Blank Analysis
10.6.1 Analyze the method blank as a sample.
10.6..2 After a run has ended, check the quantitation report to see that surrogate percent recovery values fall within the acceptable limits in Table 12-3.
10.6.3 The primary ion area counts of the internal standards should not vary by more than one-half to double of the most recent continuing calibration standard. The absolute retention time of the internal standard should not vary by more than ±30 seconds.
10.6.4 Check that no target compounds are found above the required quantitation limit. Note: (See Section 2.3.2). A method blank must contain less than five times the quantitation limit of the phthalates listed in Table 10-4. Refer to Table 12-1 for corrective action.

10.7 Sample Analysis
10.7.1 All analyses must be completed within 12 hours of the last DFTPP tune.
10.7.2 Examine the quantitation report for internal standard areas (as 10.6.3) and surrogate recoveries.

10.7.3 If all surrogate and internal standard criteria are met and no target compounds exceed the calibration range, file as an acceptable analysis.
10.7.4 If any criteria are not met, as an acceptable analysis, schedule reanalysis at appropriate dilution or reextraction.
10.7.5 Procedures when automatic quantitation algorithms are overridden: Do not change quantitation parameters without authorization from the supervisor or mass spectral specialist. When any changes are made, submit a hard copy of the changes with the data documenting the changes. For example, an alternate ion may be used for quantitation due to interference. Note the changes on the quantitation report. Submit hard copies of the integration of the alternate ion for both the standard and sample with the data. When a peak is poorly integrated using the automatic quantitation algorithms, perform a manual integration. An "M" flag is automatically appended to the quantitation report next to the compound that was integrated. Include original and manually integrated peaks, with explanation signed and dated. See SOP A.46 regarding documentation and approval for manual integrations.

10.8 LCS Analysis
10.8.1 Analyze an extracted Laboratory Control Sample (LCS) comprised of the spiking compounds with each extraction batch. Samples are spiked with either the full target list in Table 10-4 or the representative compound list in Table 12-3. For AFCEE projects, two LCSs, each consisting of a subset of compounds, are prepared and analyzed to avoid chemical interactions between compounds present in a single full list LCS. The two aqueous LCSs together must contain all target analytes unless noted differently under specific method requirements or a variance was obtained. LCSA shall contain all target analytes from SV Calibration Mixtures 1-3, 5, and 7 and LCSB shall contain all target analytes from SV Calibration Mixture 4 and 3,3-dichlorobenzidine. See preparation SOP EXT.20 and EXT.46. Nominal concentrations of component compounds in water is 30 µg/L, in soils is 1000 µg/kg. The choice of full/representative list is determined by project manager via the use of specific test codes.
10.8.2 Check quantitation report to make certain that percent recovery values for spiked compounds are within current E & E or client limits. Refer to Table 12-1 for corrective action.
10.8.3 Calculate the recovery in the LCS for those compounds that failed the matrix spike criteria. If the recovery of any such analyte is outside the current E & E limits, identify and correct the problem.

10.9 MS/MSD Analysis
10.9.1 Analyze with each batch of 20 or fewer samples.
10.9.2 Analyze at same dilution as the sample analysis.
10.9.3 Refer to Table 12-1 for corrective action.

10.10 Qualitative Analysis
10.10.1 Mass Spectral Identification Criteria.
10.10.1.1 For each individual instrument, a reference spectrum must be generated for each target compound of interest using the analytical conditions mentioned in Section 10. The spectrum is obtained at the peak apex and may include subtraction of interferences or background.
10.10.1.2 The characteristic ions present in the reference spectrum must be present in the sample spectrum. The characteristic ions are listed in Table 10-4.
10.10.1.3 The relative intensities of the characteristic ions must agree within ±30% (i.e., 30 absolute units relative to the normalized abundance of the ions of interest).
10.10.1.4 Ions in the spectra of interest which have a relative intensity >10% of the most abundant ion, but which are not found in the reference should be carefully considered to determine if they are a significant

component of the compound, or an interference. Seek guidance from the mass spectral specialist to determine the appropriate action in this case.

10.10.1.5 When a spectra does not meet all of the above criteria, but in the technical judgment of the analyst the compound may be present, seek guidance from the mass spectral specialist, who will make the final identification determination.

10.10.1.6 Consider each target compound found based on the mass spectra identification criteria of Section 10.1 and the retention time of that compound. The relative retention time should be within +/-0.06 units of the relative retention time determined in the daily calibration standard. If it does not agree, but in the technical judgment of the analyst the compound may be present, seek guidance from the mass spectral specialist. This case may arise in samples with excessive matrix effects.

10.11 Tentatively Identified Compounds (upon request)

10.11.1 Computer-generated TICs will be provided unless analyst interpretation is specifically requested.

10.11.2 Compare each tentatively identified compound with the top three library spectra generated by the automatic search routine. Major ions in the reference spectrum should be present in the sample spectrum.

10.11.3 The relative intensities of the major ions should agree within $\pm$20%.

10.11.4 Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.11.5 Ions present in the sample spectrum, but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

10.11.6 Ions present in the sample spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds.

10.11.7 If, after careful review and in the technical judgement of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound should be reported as follows:

10.11.7.1 If library search produces a match at or above 85%, report that compound.

10.11.7.2 If the library search produces more than one compound at or above 85%, report the first compound (highest).

10.11.7.3 If the library search produces no matches at or above 85%, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e., unknown phthalate, unknown hydrocarbon, unknown carboxylic acid, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

10.11.8 Perform mass spectral library searches on all additional peaks found in the sample run up to a maximum of 20 peaks. Exclude any peaks that are identified in the current run as target, internal, or surrogate compounds.

10.11.9 Quantitate the peak using the total ion area counts for the peak, and the total ion area counts of the nearest internal standard. Apply the following formula:

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 Soil Calculation

11.1.1 Instrument quantitation reports are in units of nanograms, uncorrected for any dilutions prior to analysis.

11.1.2 The sample concentrations are calculated as follows:

$$\mu g/kg = \left(\frac{m}{V_i}\right)\left(\frac{V_t}{M}\right)\cdot 1000\,\mu g/kg$$

where:

m = Nanograms determined (instrument quantitation report).
V_i = Volume extract injected, µL (2 µL).
V_t = Total final volume of extract, mL (normally 1 mL).
M = Sample mass extracted, g (normally 30 g).

11.2 Water Calculations

11.2.1 Instrument quantitation reports are in units of nanograms, uncorrected for any dilutions prior to analysis.

11.2.2 The sample concentrations are calculated as follows:

$$\mu g/L = \left(\frac{m}{V_i}\right)\left(\frac{V_t}{V}\right)$$

where:

M = Nanograms determined (instrument quantitation report).
V_t = Final volume of extracts, mL (1 mL).
V = Sample extracted (L).
V_i = Volume of combined extract injected, µL (2 µL).

11.3 Quantitation of Tentatively Identified Compounds

$$\text{Final Conc (TIC)} = \frac{\text{Mass(IS)} \cdot \text{Area Count(TIC)} \cdot \text{df}}{\text{Area Count (IS)} \cdot \text{S}}$$

where:

Final Conc (TIC) = Has the units µg/L or µg/Kg per matrix.
Mass (IS) = Internal standard mass (ng).
S = Sample weight or volume used (g or mL, per matrix).
Df = Dilution factor.
Area Count (TIC) = Area count found for the TIC.
Area Count (IS) = Area count found for the associated IS.

11.4 MS/MSD Percent Recovery and RPD Calculations

11.4.1 Calculate the recovery of each matrix spike compound in the matrix spike and matrix spike duplicate and report on the appropriate forms. Calculate the concentrations of the matrix spike compounds using the same equations as used for target compounds. Calculate the recovery of each matrix spike compound as follows:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \cdot 100$$

where:

SSR = Spike Sample Result.
SR = Sample Result.
SA = Spike Added.

11.4.2 Calculate the relative percent difference (RPD) of the recoveries of each compound in the matrix spike and matrix spike duplicate as follows:

$$RPD = \left| \frac{S-D}{1/2(S+D)} \right| \cdot 100$$

where:

RPD = Relative Percent Difference.
S = Matrix Spike Recovery.
D = Matrix Spike Duplicate Recovery.

The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

11.5 Surrogate Percent Recovery Calculation

11.5.1 Calculate the concentrations of the surrogate compounds using the same equations as used for the target compounds. Calculate the recovery of each surrogate using the following equation:

$$\%\ \text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \cdot 100$$

11.5.2 Calculate surrogate standard recovery on all samples, blanks, matrix spikes and matrix spike duplicates. Determine if recovery is within limits (see Table 12-3) and report on appropriate form.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
DFTPP Std (50 ng)	Every 12 hours	1 µL – Direct inject	See Section 10.2	Manual tune with PFTBA, Reanalyze
Initial Calibration	Prior to sample analysis	See Section 10.3	% RSD of CCC compounds ≤30%; min. RRF of SPCC ≥0.050 (see Section 10.3)	Correct problem; clean source, change column, etc., then repeat initial calibration.
ICV	After the initial calibration	See Section 10.3.16	% Recovery = 70-130%	-Reanalyze -Remake standard -Cut column -Perform routine maintenance. -Recalibrate
Calibration Verification Standard (50 ng) BNA	Every 12 hours	2 µL of SSTD050	% D of CCC compounds ≤ 20%; min. RRF of SPCC ≥0.050 (see Section 10.4)	-Reanalyze -Remake standard -Cut column -Perform routine maintenance. -Recalibrate
Method Blank	One per analytical extraction batch	Refer EXT.12, EXT.20 or EXT.46	Interference-free. Less than the QL of target compounds; 5 times QL of phthalate esters	Reextract and reanalyze associated samples.
Surrogate Recovery (SUR)	Every sample, blank, and QC.	Refer EXT.12, EXT.20 or EXT.46	All percent recovery values within E & E limits. One B/N and one A/P surrogate may exceed limits	-Check for errors in calculations, in surrogate solutions and internal standards. -Check instrument performance. -Reanalyze the sample. -Do not reanalyze if surrogates are outside limits on samples requiring five-fold or greater dilutions since surrogates will be diluted out or near QL. -It is not necessary to reanalyze a sample associated with a matrix spike if the MS/MSD surrogate

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
				recoveries show a similar pattern of surrogates out of limits. Document the similarity. -Check for integration errors and perform manual integration. -Reextract and reanalyze the sample.
Internal Standard (IS) responses and retention times	Calibrate Verification Standard	Refer to Section 10.4.1	Retention times for any (IS) must vary less than 30 seconds from most recent initial calibration. Calibration verification standard areas for any (IS) must vary less than a factor of 2 from most recent initial calibration mid level standard.	- Inspect for malfunction. If corrections are made, reanalyze standard.
Matrix spike (MS) and Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples	Refer EXT.12, EXT.20 or EXT.46	- Project requirements if specified. - Compare against LCS limits for bias.	Project requirements if any, otherwise evaluate results in conjunction with other QC information (e.g., LCS recoveries) to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Laboratory Control Sample (LCS)	One per analytical extraction batch.	Refer EXT.12, EXT.20 or EXT.46	Within E & E limits	-Identify and correct problem. Re-extract batch if within extraction hold times. Notify project manager if hold times will be exceeded. See Section 13.2.4 for additional guidance.

Revision: 12	Status: Final	Method: 8270C	Minor Revision Date: None

Table 12-2

CONTROL CHARTED QUALITY CONTROL SAMPLES

QC Type	Analytes Charted	Matrix Charted
Surrogate	Nitrobenzene-d5 2-Fluorobiphenyl Phenol-d5 Fluorophenol Terphenyl-d14 2,4,6-Tribromophenol	Soil/Water
Laboratory Control Sample	All targets	Soil/Water
Revision: 12 Status: Final	Method: 8270C	Minor Revision Date: None

Table 12-3

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES

QC Type	Matrix	Analyte	Nominal Spike Amount (ng)	Recovery (%)	RPD	Source
Surrogate	W	Nitrobenzene-d5	100	See Table 12-1	See Table 12-1	Lab
	W	2-Fluorobiphenyl	100	See Table 12-1	See Table 12-1	Lab
	W	Terphenyl-d14	100	See Table 12-1	See Table 12-1	Lab
	W	2-Fluorophenol	120	See Table 12-1	See Table 12-1	Lab
	W	Phenol-d5	120	See Table 12-1	See Table 12-1	Lab
	W	2,4,6-Tribromophenol	120	See Table 12-1	See Table 12-1	Lab
	S	Nitrobenzene d5	100	See Table 12-1	See Table 12-1	Lab
	S	2-Fluorobiphenyl	100	See Table 12-1	See Table 12-1	Lab
	S	Terphenyl-d14	100	See Table 12-1	See Table 12-1	Lab
	S	2-Fluorophenol	120	See Table 12-1	See Table 12-1	Lab
	S	Phenol-d5	120	See Table 12-1	See Table 12-1	Lab
	S	2,4,6-Tribromophenol	120	See Table 12-1	See Table 12-1	Lab
MS/MSD	W,S	1,4-Dichlorobenzene	60	See Table 12-1	See Table 12-1	Lab
	W	N-Nitroso-di-n-propylamine	60	See Table 12-1	See Table 12-1	Lab
	W	1,2,4-Trichlorobenzene	60	See Table 12-1	See Table 12-1	Lab
	W	Acenaphthene	60	See Table 12-1	See Table 12-1	Lab
	W	2,4-Dinitrotoluene	60	See Table 12-1	See Table 12-1	Lab
	W	Pyrene	60	See Table 12-1	See Table 12-1	Lab
	W	Phenol	60	See Table 12-1	See Table 12-1	Lab
	W	2-Chlorophenol	60	See Table 12-1	See Table 12-1	Lab
	W	4-Chloro-3-methylphenol	60	See Table 12-1	See Table 12-1	Lab
	W	4-Nitrophenol	60	See Table 12-1	See Table 12-1	Lab
	W	Pentachlorophenol	60	See Table 12-1	See Table 12-1	Lab

Table 12-3

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES

QC Type	Matrix	Analyte	Nominal Spike Amount (ng)	Recovery (%)	RPD	Source
LCS*	S, W	1,4-Dichlorobenzene	60	See Table 12-1	See Table 12-1	Lab
	S, W	N-Nitroso-di-n-propylamine	60	See Table 12-1	See Table 12-1	Lab
	S, W	1,2,4-Trichlorobenzene	60	See Table 12-1	See Table 12-1	Lab
	S, W	Acenaphthene	60	See Table 12-1	See Table 12-1	Lab
	S, W	2,4 Dinitrotoluene	60	See Table 12-1	See Table 12-1	Lab
	S, W	Pyrene	60	See Table 12-1	See Table 12-1	Lab
	S, W	Phenol	60	See Table 12-1	See Table 12-1	Lab
	S, W	2-Chlorophenol	60	See Table 12-1	See Table 12-1	Lab
	S, W	4-Chloro-3-methylphenol	60	See Table 12-1	See Table 12-1	Lab
	S, W	4-Nitrophenol	60	See Table 12-1	See Table 12-1	Lab
	S, W	Pentachlorophenol	60	See Table 12-1	See Table 12-1	Lab
Revision: 12		Status: Final	Method: 8270C		Minor Revision Date: None	

*Representative Compound List.

13.0 SPECIAL PROJECT REQUIREMENTS

Client-Specific Quality Control Requirements

13.1 Jacobs; Otis requires LCS/LCSD

13.2 U.S. Army Corps of Engineers (ACE)

13.2.1 Method Quantitation Limit (MQL) is between 3-10 times MDL. The low calibration standard concentration must be ≥3 times MDL. The Method Reporting Limit (MRL) may slide between two times the MDL and project-specific or regulatory requirements. Results below the MRL must be reported "ND." Results between the MQL and MRL must be reported as estimated (J flagged).

13.2.2 If using the grand mean approach for RSD (Section 10.3.9), no single analyte may be greater than 30% RSD.

13.2.3 Poor performers for Method 8270C are typically associated with the compounds that exhibit poor chromatographic behavior. Marginal failure for %RSD for these compounds shall not exceed 40%

13.2.3.1 Verify the mean response factors (RFs) for the SPCCs (system performance check compounds) satisfy the minimum RFs requirements specified in Method 8270C. If these criteria are not met, evaluate the system (e.g., for standard mix degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system). Take corrective action and recalibrate for all target analytes.

13.2.3.2 If the regression coefficient r <0.965 or RSD >30% for CCCs, this is indicative of system leak or column degradation. Take appropriate corrective action (e.g., instrument maintenance) and recalibrate for all target analytes and surrogates.

13.2.4 Initial Calibration Verification. The initial calibration curve shall be verified as accurate with a standard purchased or prepared from an independent source. This initial calibration verification (ICV) involves the analysis of a standard containing all of the target analytes, typically in the middle of the calibra-

tion range, each time the initial calibration is performed. The percent recovery of each target analyte in the ICV is determined from the initial calibration and should be between 70% and 130%.
Note: A small percentage of sporadic marginal failures (50-150% recovery) may be tolerated. The number of target analytes reported for the method will dictate the number of allowable QC failures as given below.

Number Target Analytes	Number Allowed Marginal Failures
05 – 15	1
16 – 30	2
31 – 45	3
46 – 60	4
61 – 75	5
76 – 90	6
91 – 105	7

The marginal failure allowance entails the application of an expanded acceptance criterion. If these QC criteria are not met, a new initial calibration must be performed.
13.2.5 For method blanks, check that no target compounds are found above one-half the required quantitation limit. A method blank must contain less than the quantitation limit of the phthalate listed in Table 10-4.
13.2.6 ACE criteria are summarized in Table 13-1.

Table 13-1

SUMMARY OF METHOD QUALITY OBJECTIVES FOR METHOD 8270 SEMIVOLATILES

(Source: USACE, Shell for Analytical Chemistry Requirements, Version 1.0, November 2, 1998)

QC Element	Target Analyte/Surrogate	Poor Performers/Sporadic Marginal Failures[1]
Initial Calibration (Shell, Section 9.2.2.5)	Instrument Evaluation: SPCCs: minimum RF values per method requirements CCCs: verify %RSD $\leq$ 30% Primary Evaluation (all target analytes) : $r \geq 0.995$, %RSD $\leq$ 15%, $r^2 \geq 0.990$ Alternative Evaluation: Mean %RSD for all target analytes $\leq$ 15%	No allowance Alternative Evaluation: Maximum allowable %RSD for each target analyte $\leq$ 40%
ICV (Shell, Section 9.3)	%Rec = 70% - 130%	Sporadic Marginal Failures[1]: %Rec = 50% - 150%

Table 13-1

SUMMARY OF METHOD QUALITY OBJECTIVES FOR METHOD 8270 SEMIVOLATILES

(Source: USACE, Shell for Analytical Chemistry Requirements, Version 1.0, November 2, 1998)

QC Element	Target Analyte/Surrogate	Poor Performers/Sporadic Marginal Failures[1]
CCV (Shell, Sections 9.5 / 9.5.2 / 9.5.2.4)	Instrument Evaluation: SPCCs: minimum RF values per method requirements CCCs: verify %D ≤ 30% Primary Evaluation (CCCs): %Drift ≤ 20%, %D ≤ 20%	 Primary Evaluation (remaining target analytes): Qualitative, see text
MB (Shell, Sections 10.2.1 / 11.4.1)	Target Analytes: Analytes < MDL Check Sample (~2X MDL)	Common Lab Contaminants: Analytes < MQLs
LCS (Shell, Sections 10.2.2 / 11.4.2)	Water: %Rec = 60% - 120% (~15 analytes) = 45% - 135% (~30 analytes) = 20% - 150% (~15 analytes) Solids: %Rec = 60% - 120% (~20 analytes) = 45% - 135% (~25 analytes) = 30% - 150% (~15 analytes)	Sporadic Marginal Failures[1]: Water: %Rec = 15% - 150% Solids: %Rec = 25% - 150%
MSD/MD (Shell, Sections 10.2.4 / 11.4.4)	Water: RPD ≤ 50% Solids: RPD ≤ 60%	Sporadic Marginal Failures[1]: Water: RPD ≤ 60% Solids: RPD ≤ 60%
Surrogates (Shell, Sections 10.2.5 / 11.4.5)	%Interference-Free Matrix: Water: %Rec = 60% - 120% B/N cmpds %Rec = 45% - 135% A cmpds Solids: %Rec = 60% - 120% B/N cmpds %Rec = 45% - 135% A cmpds Project Sample Matrix: Water: %Rec = 45% - 135% B/N cmpds %Rec = 35% - 140% A cmpds Solids: %Rec = 45% - 135% B/N cmpds %Rec = 35% - 140% A cmpds	Sporadic Marginal Failures[1]: Water: %Rec = 15% - 150% Solids: %Rec = 20% - 150%

[1]The number of Sporadic Marginal Failure (SMF) allowances depend upon the number of target analytes reported from the analysis. For instance, if the full list of target compounds as presented in Table 5A and 5B are reported, then five (5) SMFs are allowed to the expanded criteria presented for the ICV and LCS. If the MS includes only a subset of compounds and for surrogates, allow up to one (1) SMF for each B/N and A grouping. Refer to Section 9.3 for additional information on the application of sporadic marginal failures.

[2] B = Base, N = Neutral, and A = Acid compounds (cmpds).

Source: USACE, Shell for Analytical Chemistry Requirements, Version 1.0, November 2, 1998.

13.3 AFCEE - General Requirements
13.3.1 All AFCEE projects are subject to project-specific variances.
13.3.2 Reporting
13.3.2.1 MDL may not be > 1/2 reporting limit (RL) unless a variance was obtained.
13.3.2.2 A calibration standard at or below the RL must be included.
13.3.2.3 Apply a "U" flag if analyte result <MDL; an "F" flag if MDL ≤ analyte result ≤ RL.
13.3.3 Calibration
13.3.3.1 All initial calibration curves, must be **verified with an independent source (vendor) standard** prior to sample analysis unless an independent vendor is unavailable and a variance was obtained.
13.3.3.2 The daily and continuing calibration verifications are not required to be independent, however, the continuing calibration verification may not be used as the LCS.
13.3.4 Batching
13.3.4.1 An AFCEE analytical batch is the environmental samples that are similar in composition (matrix) and are prepared at the same time and with the same **lot of reagents.**
13.3.4.2 A batch is not to exceed 20 samples. **The sample duplicate, MS, MSD are considered environmental samples; therefore, the actual number of unique samples is less than 20.**
13.3.5 Laboratory Control Sample
13.3.5.1 Laboratory Control Sample for solid must be prepared using **Ottawa sand** unless a variance was obtained
13.3.5.2 Two LCS, each consisting of a subset of compounds, are prepared and analyzed to avoid chemical interactions between compounds present in a single full list LCS. The two aqueous LCS together must contain all target analytes unless noted differently under specific method requirements or a variance was obtained. LCSA shall contain all target analytes from SV Calibration Mixtures 1-3, 5, and 7 and LCSB shall contain all target analytes from SV Calibration Mixture 4 and 3,3-dichlorobenzidine. See preparation SOP EXT.20 and EXT.46. Nominal concentrations of component compounds in water is 30 µg/L, in soils is 1000 µg/kg.
13.3.5.4 The LCSs must be spiked at a concentration ≤ the midpoint of the calibration curve for each analyte unless a variance was obtained.
13.3.6 Matrix Spike/Matrix Spike Duplicate
13.3.6.1 The MS and MSD must be spiked at a concentration ≤ midpoint of the calibration curve for each analyte unless a variance was obtained.
13.3.6.2 AFCEE samples must be used for spiking.
13.3.7 Surrogates
13.3.7.1 Whenever one surrogate is outside limits, corrective action must be performed.
13.3.8 When **retention time** is outside windows, perform corrective action.
13.3.9 Method blank must be less than RL unless a variance was obtained.
13.3.10 Holding times for analysis is calculated from the time of extraction completion to the time of completion of all **analytical runs including dilutions, confirmation, reanalyses.**
13.3.11 AFCEE Project-Specific Method Requirements
13.3.11.1 Retention times must be updated at each initial calibration and each calibration verification unless a variance was obtained.
13.3.11.2 The AFCEE term second source calibration verification is the laboratory term initial calibration verification (ICV), run once after a curve is analyzed. Do not confuse with the AFCEE term ICV (see below). All analytes must be ± 25% of expected value for GCMS.
13.3.11.3 The AFCEE term of initial calibration verification is the same as the laboratory term daily calibration check. It may not be used for the LCS. Use calibration material for the AFCEE term ICV and continuing calibration verifications.

13.3.11.4 Percent RSD for CCC must be < 30, %RSD for all other calibration analytes must be ≤ 15%.
13.3.11.5 Second source calibration must be performed after a 5-point curve and must be ± 25% of expected.
13.3.11.7 Calibration check standard (IS) retention time must be ± 30 seconds and (IS) area within 1/2 to 2 times that of last calibration verification (12 h).

13.4 DEC – Tentatively Identified Compounds (TICs)
13.4.1 Perform mass spectral library searches on all additional peaks found in the sample run up to a maximum of 20 peaks. Exclude any peaks with area counts less than 10% of the area count of the nearest internal standard. Exclude any peaks that are identified in the current run as target, internal, or surrogate compounds.
13.4.2 Compare each TIC with the top three library spectra generated by the automatic search routine. Use as a guide to assign the identification name to the TIC if the match of the best fit is >85%. If the compound of interest has multiple isomers of identical spectra, assign a classification name to the compound. If in the technical judgement of the analyst, the spectra represents a member of a class of compounds, that classification should be assigned to the compound (e.g., unknown hydrocarbon). If no identification can be assigned to the compound, assign the name "Unknown."

13.5 Illinois EPA
13.5.1 An ICV must be analyzed with the mean %D <15%.

14.0 SAMPLE DISPOSAL
14.1 See SOP A.10, Waste Disposal, for information.

15.0 EXAMPLE FORMS
NA

END OF SOP

TITLE: GCMS Analysis of Samples for Volatile Organic Compounds by Method 8260B			
Reviewer: M. Dubel		Peer Reviewer: C. Stempniak	
Implementation Date: 12/21/00		Last Annual Review Date: 12/21/00	
Revision: 10	Status: Final	Method: 8260B	Minor Revision Date: 5/24/01

1.0 SCOPE AND APPLICATION

1.1 This SOP details the GC/MS analysis of water and soil samples for volatile organic compounds using Method 8260B. Sensitivity is generally available to 1μg/L or 1μg/kg except for the ketones and other water soluble compounds.

1.2 A 25-mL sample volume can be used to achieve lower detection limits.

1.3 The laboratory's usual reporting list is presented in Table 10-6. The target compound list can be expanded to include other compounds found in Method 8260B.

1.4 Details regarding method development and performance can be found in Method 8260B, Section 9.0.

2.0 METHOD SUMMARY

2.1 Volatile organic compounds (VOCs) are introduced into the gas chromatograph by the purge and trap technique using a 5 g soil or 5 mL water sample with added surrogates (SS) and internal standards (IS). Chromatographic conditions are such that the compounds are separated by the gas chromatograph. The compounds are detected using a mass spectrometer from which both qualitative and quantitative information is obtained. Quantitation is by the internal standard technique using relative response factors.

2.2 Utilize methanol extraction for high level soil and waste samples (see Section 10.12.6) where any expected target analyte concentration is >1000 µg/kg. This procedure is utilized for waste oil samples instead of waste dilution for volatile organics by Method 3585 (direct injection).

2.3 The following deviations from the method are noted.

2.3.1 Injection of 1 µL of BFB (50 ng/µL) rather than 2 µL of 25 ng/µL for tuning of instrument.

2.3.2 Method blanks containing common laboratory solvents (e.g., methylene chloride, acetone) below five times the quantitation limit (QL) will be considered acceptable. See Section 13 for specific client exceptions.

2.3.3 Use of 43 amu for quantitation of 4-methyl-2-pentanone and 2-butanone rather than the method suggested primary ion at 100 and 72. (Surrogate compound toluene-d8 interferes at 100 amu.)

2.3.4 Acetone is quantitated using the more responsive 43 amu peak.

2.3.5 1,2-Dichloroethane-d4 is quantitated using the more responsive 65 amu peak.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Method 8260B, (Rev 2, 12/96), SW-846 3rd Ed., Update III, June 1997.
4.2 Methods 8000B, 5030B, 5035.
4.3 Training SOP QA.5, A.23, and A.29.
4.4 Data Review SOP A.25.
4.5 Laboratory Quality Assurance Manual (L-001).
4.6 MDL SOP A.18.
4.7 Corrective Action Procedure, QA.1

5.0 DEFINITIONS/ACRONYMS

5.1 **Purged Water, Purged ASTM Type II Water, Purged DI Water** – ASTM Type II water, under constant N_2 flow, maintained in large glass bottle in the VOA laboratory.
5.2 **MDL** - Method Detection Limit as determined by 40 CFR Part 136, Appendix B.
5.3 **QL** - Quantitation limit for reporting purposes.
5.4 **Primary Ion** - Ion used for quantitation.
5.5 **Analytical Batch** - Includes a system performance check, calibration verification standard, method blank, samples, and associated QC in a 12-hour clock.
5.6 **VOA** - Volatile Organic Analysis (lab or analyst).
5.7 **VTSR** - Verified Time of Sample Receipt at laboratory.
5.8 **VOC** - Volatile Organic Compound.
5.9 **TCLP** - Toxicity Characteristic Leaching Procedure.
5.10 **CCC** - Calibration Check Compound.
5.11 **SPCC** - System Performance Check Compound.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Special care should be taken when analyzing high and low concentration VOC samples within the same analytical batch. Blanks should be analyzed to check for cross-contamination. If cross-contamination is suspected, all affected samples should be reanalyzed.
6.2 VOA analyst should avoid entering the organic sample preparation lab as methylene chloride, acetone, etc., may be introduced into the VOA lab via lab coats and clothing.
6.3 Storage and trip blanks should be analyzed to check for VOCs that may have diffused through the septum seal of sample containers.

7.0 INSTRUMENTATION AND EQUIPMENT

7.1 Tekmar 2000/2016 purge and trap unit equipped with individual heating pockets, O.I. Analytical 4560/4552 purge and trap unit, ~~Dynatech Precision Dynatrap,~~ Dynatech Precision PTA – 30, EST Archon/O.I. Analytical 4560.
7.3 Hewlett Packard HP1000 Series or Chemstation Data Acquisition System.
7.4 HP-Unix Data System with "Target 3" software.
7.5 Gas Flow Meter.
7.6 Fisher Scientific S-400 Analytical Balance (or equivalent).
7.7 Current configurations – systems may be reconfigured, substituting one purge and trap unit for another.

UNIX System Names

~~**Clyde-II**~~
~~HP5995A GC/MS~~
~~HP1000/Unix DS~~
~~Dynatech Precision Dynatrap P&T (alternative: OI Analytical 4560/4552 P&T)~~

7001D – Duke
HP5970 MS
HP5890 GC
HP1000/Unix DS
Tekmar LSC 2000 P&T
O.I. Analytical 4552 Autosampler

7001F – Fred
HP5970 MS
HP5890 GC
HP1000/Unix DS
O.I. Analytical 4560 P&T with IR Heater
O.I. Analytical 4552 Autosampler

Hank
HP5971 MS
HP5890 Series II GC
Chemstation/Unix DS
Tekmar LSC 2000
Dynatech Precision PTA-30 Autosampler
Tekmar 2032ALS (alternate)

Jake
HP5972 MS
HP5890 Series II Plus
Chemstation/Unix DS
Tekmar LSC 2000 P&T
Tekmar 2016 ALS Autosampler
Tekmar Heater Pocket Units

Linus
HP5973 MS
HP6890 GC
O.I. Analytical 4560 P&T
EST Archon Autosampler
ChemStation/Unix DS

Niles
Agilent 5973 MS
Agilent 6890 Series GC
O.I. Analytical 4560 P&T with Infrared Heater
O.I. Analytical 4552 Autosampler
ChemStation/Unix DS

Additional Equipment	Source
30-m x 0.53-mm ID DB-624 fused silica wide-bore column with 3 um film thickness	J&W Scientific, Inc., or equivalent
25-m x 0.20-mm ID HP624 fused silica column with 1.12 um film thickness	Hewlett Packard or equivalent
2-mL Amber screw cap vials with PTFE-lined septa	Fisher or equivalent
10-µL, 25-µL, 100-µL, 250-µL, 500-µL, 1-mL Syringes	Hewlett Packard or equivalent
1-mL, 2-mL, 5-mL, 10-mL, 100-mL, 1-L Volumetric flasks with ground glass stoppers	Fisher or equivalent
5-mL, 10-mL, 25-mL and 50-mL Gastight syringes	Fisher or equivalent
Liquid N_2 (for cooling GC oven)	Praxair
Disposable Pasteur pipettes	Fisher or equivalent
Ultra high purity helium	Rodgers Welding or equivalent
¼" x 8" Purge Rods 3/4" x 6" Purge tubes	Tekmar or equivalent Tekmar or equivalent
1/8" x 12" Tekmar #6 trap (or equivalent)	Tekmar or equivalent
1/8" x 12" Tekmar # 5 trap (or equivalent)	Tekmar or equivalent
Magnetic stir bars – PTFE or glass-coated	Fisher or equivalent
Stainless steel spatula	Fisher or equivalent
43-mL glass vial with Teflon-backed septa	Fisher or equivalent
Narrow-range pH paper (≤ 4 pH units)	Fisher or equivalent

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard, Reagents, and Consumables

Standard preparation, storage, and expiration are discussed in Section 10.4 of this SOP.

Note: Vendor substitutions/equivalent mixes may be used.

Table 8-1

SUMMARY OF STANDARDS AND REAGENTS

Source/Stock	Component	Concentration
Supelco TCL Volatile Mix No. 1 Catalog No. 48949	Acetone 2-Butanone 2-Hexanone 4-methyl-2-Pentanone	2000 ng/μL each
Supelco VOC Mix No. 6 Catalog No. 48799U	Bromomethane (methyl bromide) Chloroethane Chloromethane Dichlorodifluoromethane Trichlorofluoromethane Vinyl chloride (chloroethylene)	2000 ng/μL each
Supelco Custom Mix (20032291) Catalog No. 20032291	Carbon disulfide Methyl tert-butyl ether 2-Chloroethyl vinyl ether	2000 ng/μL each
Supelco Custom Mix (~~20032289~~) (20178498) Catalog No. ~~20032289~~ 20178498	1-Chlorohexane	2000 ng/μL
Restek Vinyl Acetate Catalog No. 30216	Vinyl acetate	2000 ng/μL
Supelco Custom Mix (20030968) Catalog No. 20030968	Chlorobenzene-d5 Fluorobenzene 1,4-Dichlorobenzene-d4	5000 ng/μL each
Restek Catalog No. 30240	Dibromofluoromethane Toluene-d8 1,2-Dichloroethane-D4 4-Bromofluorobenzene	2500 ng/μL each
Supelo 502/524 VOC Mix Catalog No. 502111	Benzene Bromobenzene Bromodichloromethane Bromoform Carbon tetrachloride Chlorbenzene Chloroform cis-1,3-Dichlopropene cis-1,2-Dichloroethylene	2000 ng/μL each

Table 8-1

SUMMARY OF STANDARDS AND REAGENTS

Source/Stock	Component	Concentration
	Dibromochloromethane	
	Dibromomethane	
	Ethylbenzene	
	Hexachlorobutadiene	
	Isopropylbenzene (cumene)	
	m-xylene	
	Methylene chloride	
	n-Butylbenzene	
	n-propylbenzene	
	Naphthalene	
	o-Xylene	
	p-Isopropyltolune	
	p-Xylene	
	sec-Butylbenzene	
	Styrene	
	Tert-Butylbenzene	
	Tetrachloroethene	
	Toluene	
	Trans-1,3-Dichloropropene	
	Trans-1,2-Dichloroethylene	
	Trichloroethylene	
	1,1-Dichloroethane	
	1,1-Dichloroethylene	
	1,1-Dichloropropene	
	1,1,1-Trichloroethane	
	1,1,1,2-Tetrachloroethane	
	1,1,2-Trichloroethane	
	1,1,2,2-Tetrachloroethane	
	1,2-Dibromo-3-chloropropane	
	1,2-Dibromoethane	
	1,2-Dichlorobenzene	
	1,2-Dichloroethane	
	1,2-Dichloropropane	
	1,2,3-Trichlorobenzene	
	1,2,3-Trichloropropane	
	1,2,4-Trichlorobenzene	
	1,2,4-Trimethylbenzene	
	1,3-Dichlorobenzene	
	1,3-Dichloropropane	
	1,3,5-Trimethylbenzene	
	1,4-Dichlorobenzene	

Table 8-1

SUMMARY OF STANDARDS AND REAGENTS

Source/Stock	Component	Concentration
	2-Chlorotoluene 2,2-Dichloropropane 4-Chlorotoluene	
Supelo Custom Mix (2S*) Catalog No. 20099962	Benzene Bromobenzene Bromodichloromethane Bromoform Carbon tetrachloride Chlorbenzene Chloroform cis-1,3-Dichlopropene cis-1,2-Dichloroethylene Dibromochloromethane Dibromomethane Ethylbenzene Hexachlorobutadiene Isopropylbenzene (cumene) m-xylene Methylene chloride n-Butylbenzene n-propylbenzene Naphthalene o-Xylene p-Isopropyltolune p-Xylene sec-Butylbenzene Styrene Tert-Butylbenzene Tetrachloroethene Toluene Trans-1,3-Dichloropropene Trans-1,2-Dichloroethylene Trichloroethylene 1,1-Dichloroethane 1,1-Dichloroethylene 1,1-Dichloropropene 1,1,1-Trichloroethane 1,1,1,2-Tetrachloroethane 1,1,2-Trichloroethane 1,1,2,2-Tetrachloroethane	2000 ng/μL each

Table 8-1

SUMMARY OF STANDARDS AND REAGENTS

Source/Stock	Component	Concentration
	1,2-Dibromo-3-chloropropane 1,2-Dibromoethane 1,2-Dichlorobenzene 1,2-Dichloroethane 1,2-Dichloropropane 1,2,3-Trichlorobenzene 1,2,3-Trichloropropane 1,2,4-Trichlorobenzene 1,2,4-Trimethylbenzene 1,3-Dichlorobenzene 1,3-Dichloropropane 1,3,5-Trimethylbenzene 1,4-Dichlorobenzene 2-Chlorotoluene 2,2-Dichloropropane 4-Chlorotoluene	
Supelco TCL Volatile Mix No. 1 (2S)* Catalog No. 4S8949	Acetone 2-Butanone 2-Hexanone 4-methyl-2-Pentanone	2000 ng/µL each
Supelco VOC Mix No. 6 (2S)* Catalog No. 4S8799	Bromomethane Chloroethane Chloromethane Dichlorodifluoromethane Trichlorofluoromethane Vinyl chloride	2000 ng/µL each
Supelco Custom Mix (20032292) Catalog No. 20032292	Carbon disulfide Methyl-tert-butyl-ether 2-chloroethylvinyl ether	2000 ng/µL
UltraSci 1-Chlorohexane (2S)* Catalog No. EPA 1208	1-Chlorohexane	1000 ng/µL
Supelco Custom Mix (20032293) (2S)* Catalog No. 20032293	Vinyl acetate	2000 ng/µL
Supelco BFB Catalog No. 48083	Bromofluorobenzene	2000 ng/µL
Baker Sodium Bisulfate Catalog No. 353401	Sodium bisulfate	Neat
Burdick & Jackson Methanol Catalog No. 232235	Methanol (purge and trap grade)	Neat

Table 8-1

SUMMARY OF STANDARDS AND REAGENTS

Source/Stock	Component	Concentration
Fisher Ottawa Sand Catalog No. S233	Ottawa sand	Neat
Supelco Custom Mix (20182464) Catalog No. 20182464	Cyclohexane 1,1,2-Trichlor-1,2,2-Trifluoromethane Methyl acetate Methyl cyclohexane	5,000 ng/µL
Supelco Custom Mix (20182466) (2S)* Catalog No. 20182466	Cyclohexane 1,1,2-Trichlor-1,2,2-Trifluoromethane Methyl acetate Methyl cyclohexane	5,000 ng/µL
Purged ASTM Type II water	In-lab system	(Charcoal scrubbed)

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*2S = Second source.

8.2 Standard Solution Preparation (SS = Surrogate Standard)

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
BFB	Supelco BFB	25 µL to 1 mL with methanol	50 ng/µL
VSTD 250	Supelco 502/524 VOC Mix Supelco TCL Volatile Mix No. 1 Supelco VOC Mix No. 6 Supelco Custom Mix (20032291) Supelco Custom Mix ~~(20032289)~~ (20182464) Restek Vinyl Acetate Supelco Custom Mix (20182464)	250 µL 250 µL 250 µL 250 µL 250 µL 250 µL 100 µL to 2 mL with methanol	250 ng/µL 100 µL
VSTD 050	E & E VSTD 250	400 µL to2 mL with methanol	50 ng/µL
8260 TK IS	Supelco Custom Mix (20030968)	10 µL to 2 mL with methanol	25 ng/µL
~~8260 DY IS~~ 4552 'N' SS	Supelco Custom Mix (20030968)	~~75.0~~ 115 µL to 2 mL with methanol	~~188~~ 287.5 ng/µL
8260 PTA IS	Supelco Custom Mix (20030968)	47 µL to 2 mL with methanol	118 ng/µL
8260 Archon IS (Soil, Low-Level Water)	Supelco Custom Mix (20030968)	120 µL to 2 mL with methanol	300 ng/µL
8260 4552 "D" IS	Supelco Custom Mix (20030968)	125 µL to 2 mL with methanol	312 ng/µL
8260 4552 "F" IS	Supelco Custom Mix (20030968)	95 µL to 2 mL with methanol	237.5 ng/µL
8260 SSS Mix	Restek 8260A SS Mix	~~125~~ 200 µL to ~~1~~ 2 mL with methanol	250 ng/µL
8260 SS Mix	Restek 8260A SS Mix	~~12.5~~ 20 µL to ~~1~~ 2 mL with methanol	25 ng/µL
8260 TK IS/SS	Supelco Custom Mix (20030968) Restek 8260A SS Mix	10 µL ~~25~~ 20 µL to 2 mL with methanol	25 ng/µL 25 ng/µL
8260 ~~DY~~ 4552 'N' IS/SS	Supelco Custom Mix (20030968) Restek 8260A SS Mix	~~75.0~~ 115 µL ~~188~~ 230 µL to 2 mL with methanol	~~188~~ 287.5 ng/µL

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
8260 PTA IS/SS	Supelco Custom Mix (20030968) Restek 8260A SS Mix	47 µL 95 µL } to 2 mL with methanol	118 ng/µL
8260 Archon IS/SS (Soil, 25-mL Water)	Supelco Custom Mix (20030968) Restek 8260A SS Mix	120 µL ~~300~~ 240 µL } to 2 mL with methanol	300 ng/µL
8260 4552 "D" IS/SS	Supelco Custom Mix (20030968) Restek 8260A SS Mix	125 µL 250 µL } to 2 mL with methanol	312 ng/µL
8260 4552 "F" IS/SS	Supelco Custom Mix (20030968) Restek 8260A SS Mix	95 µL 190 µL } to 2 mL with methanol	237.5 ng/µL
Archon 5 mL Water 8260 IS	Supelco Custom Mix (20030968) ~~Restek 8260A SS Mix~~	~~95~~ 88 µL ~~190 µL~~ } to 2 mL with methanol	220 ng/µL
Archon 5 mL Water 8260 IS/SS	Supelco Custom Mix (20030968) Restek 8260A SS Mix	88 µL ~~167~~ 176 µL } to 2 mL with methanol	220 ng/µL
VOA MS 250	Supelco Custom Mix (20099962) Supelco TCL Volatile Mix No.1 (2S) Supelco VOC Mix No. 6 ~~(SS)~~ (2S) Supelco Custom Mix (20032292) UltraSci 1-Chlorohexane Supelco Custom Mix ~~(2003293)~~ (20032293)	250 µL 250 µL 250 µL 250 µL 250 µL 250 µL } to 2 mL with methanol	250 ng/µL
	Supelco Custom Mix (20182466)	100 µL	100 µL
VOA MS 050	E & E VOA MS 250	400 µL to2 mL with methanol	50 ng/µL

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Key:

Archon = Made for EST P&T Unit.
~~DY = Made for Dynatrap P&T Unit.~~
PTA = Made for Dynatech PTA30 P&T Unit.
SS = Surrogate standard.
2S = Second source.
TK = Made for Tekmar P&T Unit.
4552 'D' = Made for 4552 O.I. Analytical P&T Unit on Duke.
4552 'F' = Made for 4552 O.I. Analytical P&T Unit on Fred.
4552 'N' = Made for 4552 O.I. Analytical P&T Unit on Niles.

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Solid	Standard	N/A	14 Days from collection	4-oz. Septum jar or VOA vial, 2-6 C
Water	Standard	N/A	14 Days from collection	43-mL VOA vial pH <2 with HCl 2-6 C
Water	NYSDEC	N/A	7 Days VTSR	43-mL VOA vial
Solid	TCLP	14	14 Days from ZHE Extraction	4-oz. Septum jar 2-6 C if applicable to waste.
Solid	Method 5035	NA	48 Hours from collection to preservation	EnCore™ or equivalent sample. Sodium bisulfate or methanol added at laboratory.
Solid	Method 5035	NA	14 Days from collection	VOA vial pH<2 with $NaHSO_4$ solution, 2-6 C.
Solid	NYSDEC	NA	7 days VTSR	4-oz Septum jar, 2-6 C
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9.1 Method 5035 Sample Vial Preparation

9.1 Preparation of sample vial for Method 5035.

9.1.1 Sample vials are to be prepared in a fixed laboratory environment, sealed and shipped to the field location.

9.1.1.1 Vials used to collect low concentration soil samples to be analyzed by the closed system purge-and-trap equipment will have:

- A clean magnetic stirring bar;
- Approximately 1g of sodium bisulfate (adjust when needed to 0.2 g $NaHSO_4$/1 g sample); and
- 5 mL organic-free reagent ASTM Type II purged water added to each clean vial. The vial is then sealed with a screw-cap and septum seal and a label is affixed. The vials are weighed to the nearest 0.01g. The weight is recorded in EnCore™ logbook and written on the label.

9.1.1.2 Vials used to collect high concentration soils will have 10 mL methanol added and be sealed with the screw-cap and septum seal. A label will be affixed and the weight (to the nearest 0.01g) will be recorded in EnCore™ logbook and written on the label.

10.0 PROCEDURE

10.1 Apparatus Setup

10.1.1 The column is attached from the injection port to a jet separator. A transfer line connects the jet separator to the GC/MS source. The column flow is set at 5-10 mL/min. This results in a pressure of 0.2-0.5 torr at the jet separator pump. A make-up gas adapter is used if the particular jet separator requires it. For Jake, ~~and~~ Linus, and Niles - GC column connected to MS source via transfer line only. No jet separator used on these instruments.

10.1.2 The temperature ramp for sample analysis is adjusted to adequately separate the volatile organic target compounds. Start the run at 5 C and hold for 5 minutes. Ramp at 5 C per minute to 90 C. Then ramp at 10 C/min. to 170 C., at which point all Table 10-6 target analytes are eluted. For Jake, ~~and~~ Linus, and Niles – Initial temperature is ~~0~~ 40 C, initial time – 4.0 mins. Then ramp at 8 C/min. to 180 C. Bake column for 2.5 mins. at 180 C.

10.1.3 The injection/detector temperatures are as follows.

~~Clyde,~~ Duke, Fred, Hank:
Injector = 150 C
Detector = 200 C
Linus, Jake, Niles:
Injector = 220 C
Detector = ~~230~~ 250 C (Linus and Niles); 280 C (Jake)

10.1.4 The Tekmar purge and trap, autosampler, and sample heater is set up with the following conditions:
Jake, Hank, and Duke (P&T only):

- ALS 2016 Valve = 110 C
- ALS 2016 Lines = 110 C
- LSC 2000 Valve = 110 C
- LSC 2000 Lines = 110 C
- Preheat Sample = 1 min. at 40 C (soil samples only)
- Purge Time = 11 min. (Heat at 40 C while purging for soil samples only)
- Desorb Time = 2 min. at 180 C
- Bake Time = 10-13 min.
- Bake Temperature = 225 C
- Purge Pressure = 20 psi
- Purge Flow = 25-40 mL/min.
- Trap Type = Tekmar #5 or equivalent
- Purge Volume = 5 g soil sample + 5 mL purged ASTM Type II water, or 5 mL water sample.

~~The Dynatech Dynatrap purge and trap unit settings are as follows:~~
~~Clyde:~~

- ~~Purge time = 11 min~~
- ~~Desorb time = 1.5 min~~
- ~~Bake time = 11-15 min~~
- ~~Desorb Temp. = 180 C~~

- ~~Bake Temp. = 225 C~~
- ~~Valve Temp. = 110 C~~
- ~~Line Temp. = 110 C~~
- ~~Purge pressure = 20 psi~~
- ~~Purge flow = 25-40 mL/min.~~
- ~~Trap type = OI #8 Trap or equivalent, Dynatech "F" trap~~
- ~~The mass spectrometer is set up per manufacturer's instructions. For this analysis, the scan range is set to 35-300 amu, a two second or better scan speed, at 70 eV nominal.~~

10.1.5 The EST Archon, O.I. Analytical 4552 ALS and 4560 purge and trap unit settings are as follows: Linus, Fred, ½ Duke, Niles
(IR heated purge for 25 mL water = 40 C. Preheat sample (soils) 1.0 min at 40 C):

- Purge time = 11.0 min
- Desorb time = 2.0 min
- Bake time = 9-12 min.
- Desorb Temp. = 180 C
- Bake Temp. = 225 C
- Valve Temp. = 100 C
- Line Temp. = 100 C
- Purge pressure = 20 psi
- Purge flow = 25-40 mL/min.
- Trap type = O.I. Analytical #8 trap or equivalent, or Tekmar #5 trap.

10.1.6 Note: Instrument settings for any batch that varies from those listed are recorded in the injection logbook.

10.2 Instrument Maintenance

10.2.1 Nonroutine Maintenance - Each instrument is under a Basic Hardware Maintenance Service Agreement provided by the manufacturer which includes both preventative and emergency service. Preventative maintenance is carried out by a qualified service engineer biannually. Document maintenance and service in a logbook including the following information:

- Date of repair or maintenance
- Person repairing instrument
- Summary of problems instrument was experiencing before repair
- Detailed description of repairs completed
- Parts replaced during repair

10.2.2 Routine Maintenance - perform routine instrument maintenance procedures as noted in Table 10-1.

10.2.3 The section supervisor reviews and signs the maintenance log as necessary.

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Gas Chromatograph	Contamination suspected	Change septum	As needed
		Clip column	As needed
		Change injection port liner	As needed
	Problems calibrating and/or severe contamination	Change column	As needed
Mass Spectrometer	High background	Bake source	As needed
	Cannot calibrate	Clean source	As needed
Purge and Trap Unit	Contamination suspected	Change trap	As needed
		Bake lines	As needed
		Methanol rinse system	As needed
	Problems calibrating	Change trap	As needed
		Refurbish instrument Adjust purge flow	As needed

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Table 10-4.1 (Jake)

STANDARD CALIBRATION PREPARATION FOR TEKMAR P+T UNITS

Standard	Amt. TK IS	Amt. TK SS	Amt. SSS	Amt. VOA STD 050	Amt. VOA STD 250	Equivalent Concentration (µg/kg)	Equivalent Concentration (µg/L)
VSTD005	10 µL	1 µL	–	0.5 µL/5 mL DI	–	5	5
VSTD010	10 µL	2 µL	–	1 µL/5 mL DI	–	10	10
VSTD020	10 µL	4 µL	–	2 µL/5 mL DI	–	20	20
VSTD050	10 µL	10 µL	–	5 µL/5 mL DI	–	50	50
VSTD100	10 µL	–	2 µL	–	2 µL/5 mL DI	100	100
VSTD200	10 µL	–	4 µL	–	4 µL/5 mL DI	200	200

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Note: DI = Purged ASTM Type II water for Tables 10-4.1 through 10-4.8.

Table 10-4.2

STANDARD CALIBRATION PREPARATION FOR O.I. ANALYTICAL P+T UNIT

Standard	Amt. PTA IS	Amt. SS	Amt. SSS	Amt. VOA STD 050	Amt. VOA STD 250	Equivalent Concentration (µg/L)
VSTD0005	2.19 µL	2 µL	—	1 µL/100 mL DI	—	.5
VSTD001	2.19 µL	2 µL	—	1 µL/50 mL DI	—	1
VSTD005	2.19 µL	—	1 µL	—	1 µL/50 mL DI	5
VSTD010	2.19 µL	—	2 µL	—	2 µL/50 mL DI	10
VSTD020	2.19 µL	—	4 µL	—	4 µL/50 mL DI	20
VSTD040	2.19 µL	—	8 µL	—	8 µL/50 mL DI	40
VSTD050	2.19 µL	—	10 µL	—	10 µL/50 mL DI	50
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Table 10-4.3 (Hank)

STANDARD CALIBRATION PREPARATION FOR DYNATECH PTA 30 FOR ALS/LSC 2000 P+T UNIT

Standard	Amt. PTA IS	Amt. SSS	Amt. VOA STD 250	Equivalent Concentration (µg/L)
VSTD005	2.19 µL	2 µL	2 µL/100 mL DI	5
VSTD010	2.19 µL	2 µL	2 µL/50 mL DI	10
VSTD020	2.19 µL	4 µL	4 µL/50 mL DI	20
VSTD050	2.19 µL	10 µL	10 µL/50 mL DI	50
VSTD100	2.19 µL	20 µL	20 µL/50 mL DI	100
VSTD200	2.19 µL	40 µL	40 µL/50 mL DI	200
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~~**Table 10-4.4 (Clyde)**~~

~~**STANDARD CALIBRATION PREPARATION FOR DYNATECH DYNATRAP P+T UNIT**~~

~~Standard~~	~~Amt. DY IS~~	~~Amt. SSS~~	~~Amt. VOA STD 250~~	~~Equivalent Concentration (µg/L)~~
~~VSTD005~~	~~1.33 µL~~	~~2 µL~~	~~2 µL/100 mL DI~~	~~5~~
~~VSTD010~~	~~1.33 µL~~	~~2 µL~~	~~2 µL/50 mL DI~~	~~10~~
~~VSTD020~~	~~1.33 µL~~	~~4 µL~~	~~4 µL/50 mL DI~~	~~20~~
~~VSTD050~~	~~1.33 µL~~	~~10 µL~~	~~10 µL/50 mL DI~~	~~50~~
~~VSTD100~~	~~1.33 µL~~	~~20 µL~~	~~20 µL/50 mL DI~~	~~100~~
~~VSTD200~~	~~1.33 µL~~	~~40 µL~~	~~40 µL/50 mL DI~~	~~200~~
~~Revision: 10~~	~~Status: Final~~	~~Method: 8260B~~	~~Minor Revision Date: 5/24/01~~	

Table 10-4.4 (Linus)

**STANDARD CALIBRATION PREPARATION FOR EST ARCHO
O.I. ANALYTICAL 4560 P+T UNIT
(WATERS)**

Standard	Amt. Archon 5 mL Water IS*	Amt. SSS	Amt. VOA STD 250	Equivalent Concentration (µg/L)
VSTD005	0.83 µL	2 µL	2 µL/100 mL DI	5
VSTD010	0.83 µL	2 µL	2 µL/50 mL DI	10
VSTD020	0.83 µL	4 µL	4 µL/50 mL DI	20
VSTD050	0.83 µL	10 µL	10 µL/50 mL DI	50
VSTD100	0.83 µL	20 µL	20 µL/50 mL DI	100
VSTD200	0.83 µL	40 µL	40 µL/50 mL DI	200

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Table 10-4.5 (Linus)

**STANDARD CALIBRATION PREPARATION FOR EST ARCHON
O.I. ANALYTICAL 4560 P+T UNITS
(SOILS)**

Standard	Amt. Archon Soil/25 mL Water IS	Amt. SS	Amt. SSS	Amt. VOA STD 050	Amt. VOA STD 250	Equivalent Concentration (µg/kg)
VSTD005	0.83 µL	1 µL	—	0.5 µL/5 mL DI	—	5
VSTD010	0.83 µL	2 µL	—	1 µL/5 mL DI	—	10
VSTD020	0.83 µL	4 µL	—	2 µL/5 mL DI	—	20
VSTD050	0.83 µL	10 µL	—	5 µL/5 mL DI	—	50
VSTD100	0.83 µL	—	2 µL	—	2 µL/5 mL DI	100
VSTD200	0.83 µL	—	4 µL	—	4 µL/5 mL DI	200

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Table 10-4.6 (Linus)

STANDARD CALIBRATION PREPARATION FOR EST ARCHON O.I. ANALYTICAL 4560 P+T UNITS (LOW LEVEL WATERS)

Standard	Amt. Archon Soil/25 mL Water IS	Amt. SS	Amt. SSS	Amt. VOA STD 050	Amt. VOA STD 250	Equivalent Concentration (µg/L)
VSTD0005	0.83 µL	2 µL	—	1 µL/100 mL DI	—	.5
VSTD001	0.83 µL	2 µL	—	1 µL/50 mL DI	—	1
VSTD005	0.83 µL	—	1 µL	—	1 µL/50 mL DI	5
VSTD010	0.83 µL	—	2 µL	—	2 µL/50 mL DI	10
VSTD020	0.83 µL	—	4 µL	—	4 µL/50 mL DI	20
VSTD040	0.83 µL	—	8 µL	—	8 µL/50 mL DI	40
VSTD050	0.83 µL	—	10 µL	—	10 µL/50 mL DI	50

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Table 10-4.7 (Duke, ~~and~~ Fred, and Niles)

STANDARD CALIBRATION PREPARATION FOR O.I. ANALYTICAL 4552/4560 P+T UNIT (WATERS)

Standard	Amt. 4552 "D"/ Amt. 4552 "F"/ Amt. 4552 "N" IS	Amt. SSS	Amt. VOA STD 250	Equivalent Concentration (µg/L)
VSTD005	0.80 µL/1.05 µL/0.87 µL	2 µL	2 µL/100 mL DI	5
VSTD010	0.80 µL/1.05 µL/0.87 µL	2 µL	2 µL/50 mL DI	10
VSTD020	0.80 µL/1.05 µL/0.87 µL	4 µL	4 µL/50 mL DI	20
VSTD050	0.80 µL/1.05 µL/0.87 µL	10 µL	10 µL/50 mL DI	50
VSTD100	0.80 µL/1.05 µL/0.87 µL	20 µL	20 µL/50 mL DI	100
VSTD200	0.80 µL/1.05 µL/0.87 µL	40 µL	40 µL/50 mL DI	200

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Table 10-4.8 (Duke, ~~and~~ Fred, and Niles)

STANDARD CALIBRATION PREPARATION FOR O.I. ANALYTICAL 4552/4560 P+T UNITS (SOILS)

Standard	Amt. 4552 "D"/ Amt. 4552 "F"/ Amt. 4552 "N" IS	Amt. SS	Amt. SSS	Amt. VOA STD 050	Amt. VOA STD 250	Equivalent Concentration (µg/kg)
VSTD005	0.80 µL/1.05 µL/0.87 µL	1 µL	—	0.5 µL/5 mL DI	—	5
VSTD010	0.80 µL/1.05 µL/0.87 µL	2 µL	—	1 µL/5 mL DI	—	10
VSTD020	0.80 µL/1.05 µL/0.87 µL	4 µL	—	2 µL/5 mL DI	—	20
VSTD050	0.80 µL/1.05 µL/0.87 µL	10 µL	—	5 µL/5 mL DI	—	50
VSTD100	0.80 µL/1.05 µL/0.87 µL	—	2 µL	—	2 µL/5 mL DI	100
VSTD200	0.80 µL/1.05 µL/0.87 µL	—	4 µL	—	4 µL/5 mL DI	200

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Table 10-5

BFB KEY IONS AND ION ABUNDANCE CRITERIA FOR QUADRAPOLE MASS SPECTROMETERS

Mass	Ion Abundance Criteria
50	15.0 – 40.0% of mass 95
75	30.0 – 60.0% of mass 95
95	Base peak, 100% relative abundance[1]
96	5.0 – 9.0% of mass 95
173	Less than 2.0% of mass 174
174	>50% of mass 95
175	5.0 – 9.0 % of mass 174
176	Greater than 95.0 but less than 101.0% of mass 174
177	5.0 – 9.0% of mass 176

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[1]All ion abundances MUST be normalized to m/z 95.
[2]Alternate tuning criteria may be used if requested by the client.

Table 10-6

CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS AND QUANTITATION LIMITS

Analyte	Primary Ion	Secondary Ion(s)	PQL Soils (µg/kg)	PQL 5-mL Water (µg/L)	PQL 25-mL Water (µg/L)
Chloromethane	50	52	10	10	.5
Bromomethane	94	96	10	10	.5
Vinyl chloride	62	64	10	10	.5
Chloroethane	64	66	10	10	.5
Methylene chloride	84	49, 86	5	5	1
Acetone	43	58	10	10	5
Carbon disulfide	76	78	5	5	5
1,1-Dichloroethene	96	61, 63	5	5	.5
1,1-Dichloroethane	63	65, 83	5	5	.5
1,2-Dichloroethene, total*	96	61, 98	5	5	.5
Chloroform	83	85	5	5	.5
1,2-Dichloroethane	62	98	5	5	.5
2-Butanone	43	72	10	10	5
1,1,1-Trichloroethane	97	99,61	5	5	.5
Carbon tetrachloride	117	119	5	5	.5
Vinyl acetate	43	86	10	10	5
Bromodichloromethane	83	85, 127	5	5	.5
1,1,2,2-Tetrachloroethane	83	85, 131	5	5	.5
1,2-Dichloropropane	63	112	5	5	.5
trans-1,3-Dichloropropene	75	39, 77	5	5	± 0.5
Trichloroethene	95	130, 97	5	5	.5
Dibromochloromethane	129	127	5	5	± 0.5
1,1,2-Trichloroethane	83	85, 97	5	5	.5
Benzene	78	--	5	5	.5
cis-1,3-Dichloropropene	75	39, 77	5	5	± 0.5
2-Chloroethylvinyl ether	63	65, 106	10	10†	5†
Bromoform	173	175	5	5	1
2-Hexanone	43	58, 100	10	10	5
4-Methyl-2-pentanone	43	58	10	10	5
Tetrachloroethene	164	129, 131	5	5	.5
Toluene	92	91	5	5	1
Chlorobenzene	112	77, 114	5	5	.5
Ethylbenzene	91	106	5	5	.5
Styrene	104	78	5	5	1
Xylenes, total**	106	91	5	5	.5
1,2-Dichlorobenzene	146	111, 148	5	5	.5
1,3-Dichlorobenzene	146	111, 148	5	5	.5

Table 10-6

CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS AND QUANTITATION LIMITS

Analyte	Primary Ion	Secondary Ion(s)	PQL Soils (µg/kg)	PQL 5-mL Water (µg/L)	PQL 25-mL Water (µg/L)
1,4-Dichlorobenzene	146	111, 148	5	5	.5
Trichlorofluoromethane	101	103	5	5	± 0.5
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*Includes cis- and trans-1,2-dichloroethene.
**Includes o-, m-, and p-xylenes.
†Compound not reported from acid-preserved samples.

Table 10-6.1

CHARACTERISTIC IONS FOR SURROGATE COMPOUNDS AND INTERNAL STANDARDS FOR VOLATILE ORGANIC COMPOUNDS

Analyte	Primary Ion	Secondary Ion(s)	PQL Soils (µg/kg)	PQL 5 mL Water (µg/L)	PQL 25 mL Water (µg/L)
Surrogate Compounds					
Dibromofluoromethane	113		5	5	5
4-Bromofluorobenzene	95	174, 176	5	5	5
1,2-Dichloroethane-d4	65	67, 102	5	5	5
Toluene-d8	98		5	5	5
Internal Standards					
Fluorobenzene	96	77	NA	NA	NA
Chlorobenzene-d5	117		NA	NA	NA
1,4-Dichlorobenzene-d4	152	115, 150	NA	NA	NA
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Table 10-6.2

MAXIMUM ALLOWABLE CONCENTRATIONS OF TCLP COMPOUNDS

Compound	Quantitation Limit (mg/L)	Maximum Allowable Limit (mg/L)
Benzene	0.05	0.05
Carbon tetrachloride	0.05	0.05
Chlorobenzene	0.05	100
Chloroform	0.05	5
1,2-Dichloroethane	0.05	0.5
1,1-Dichloroethylene	0.05	0.7
Methyl ethyl ketone	0.10	200
Tetrachloroethylene	0.05	0.7
Vinyl chloride	0.10	0.2
Trichloroethylene	0.05	0.5

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10.3 Preventing Contamination

10.3.1 Rinse all syringes used for transfer of samples or stock standard solutions a minimum of three times with methanol before switching to the next solution. Rinse sample syringes a minimum three times with purged ASTM Type II water.

10.3.2 Glassware Cleaning Procedure:

10.3.2.1 Wash all purge tubes and purge rods and glassware (Erlenmeyer, volumetrics, etc.) with hot tap water and soap if necessary.

10.3.2.2 Thoroughly rinse all glassware with ASTM Type II water and bake dry.

10.3.3 When a sample containing analytes in excess of two times the highest calibration standard is run, do not use that particular autosampler position for any other samples until a blank is run on that position demonstrating no contamination exists.

10.3.4 When a sample containing analytes in excess of two times the highest calibration standard is run, do not use the following run if any analytes from the previous run are detected above the QL.

10.4 Standard Solution Preparation

10.4.1 All stock standards commercially obtained shall be certified by the manufacturer. The stock standards are purchased as mixes of various compounds per mix. Keep certificates on file for all stocks received. Store all stocks and standards at –10 C or less.

10.4.2 Put a small amount of methanol in the volumetric being used for the preparation.

10.4.3 Prepare standards using Supelco™ and Restek™ or equivalent vendor stock solutions. Carefully add the correct amount of stock solution for each compound to the volumetric using syringes. Be sure to add the stock below the surface of the methanol. Dilute to volume, invert three times, and transfer the standard to a vial.

10.4.4 The compounds contained in each stock standard are listed in Table 8-1.

10.4.5 Prepare internal standards and/or surrogate standards using the table information (see Table 8-2).

10.4.6 Prior to analysis, make up running standards for Tekmar (soil and water), EST Archon (soil only),

and O.I. Analytical 4552 P&T (soil only) Units in a 5-mL Hamilton syringe barrel containing 5-mL of purged ASTM Type II water (see Tables 10-4.1, 10-4.6, and 10-4.9).

10.4.7 For Dynatech (water), EST Archon (water), and O.I. Analytical 4552 P&T (water) units, all running standards are made in a 50- or 100-mL volumetric flask containing purged ASTM Type II water and transferred to a VOA vial after preparation (see Tables 10-4.2 through 10-4.5, 10-4.7, and 10-4.8).

10.4.8 All vials holding stock and working standards are labeled with the following information:

- Standard name and concentration;
- Date prepared;
- Standard number - book number, page number, and preparation number (e.g., 249-178-1);
- Initials of preparer; and
- Expiration date

10.4.9 The following are the expiration dates for volatile stock and working standards.

10.4.9.1 Stocks - 1 month from date opened.

10.4.9.2 BFB - 1 month from date prepared.

10.4.9.3 Volatile working standards - 1 week from date prepared.

10.5 Tuning

10.5.1 Inject one (l) µL of the BFB (50 ng/µL) standard into the injection port of the GC. The injection time defines the beginning of a 12-hour clock. A tune precedes any initial calibration.

10.5.2 Select the scan which corresponds to the top of the BFB peak, average it with the scan before and after, and subtract from it one scan from the background region, no more than 20 scans prior to elution of the BFB peak. This constitutes the BFB tune check scan.

10.5.3 Evaluate the BFB tune check scan against the criteria listed in Table 10-5.

10.5.4 If tuning criteria cannot be met, do not proceed until corrective action is taken. This may include retuning or cleaning the source. If necessary, manually tune the instrument using tuning compound PFTBA. Repeat from step 10.5.1.

10.5.5 Save a hardcopy of each successful tune's parameters in a file.

10.6 Initial Calibration

10.6.1 Compound List and Quantitation Limits: see Table 10-6.

10.6.2 Perform the initial calibration at the six nominal levels specified in Tables 10-4.1 through 10-4.8.

10.6.3 The 10 compounds listed in Table 10-6 that begin at 10 µg/L (µg/kg) use the 5 points VSTD010, VSTD020, VSTD050, VSTD100, and VSTD200 for these analytes. The remaining compounds use all six standards, including VSTD005 (5 µg/L, 5 µg/kg). For ~~24~~25-mL water, seven compounds including 2-chloroethylvinylether listed in Table 10-6 use five points (VSTD005, 010, 020, 040, 050). ~~Eight~~ Four compounds listed in Table 10-6 use six points (VSTD001, 005, 010, 020, 040, 050). The remaining compounds use all seven points including VSTD0005.

10.6.4 Analyze each calibration standard as prepared in Tables 10-4.1 to 10-4.8. Preheat the calibration standards for a soil calibration for 1-3 minutes at 40 C before purging. Calibration standards for 25-mL water analyzed using O.I. Analytical 4560 P&T Units are heated while purging to 40 C using IR heater. (Calibration standards used for 5 mL water calibration do not go through a preheat stage.) This is the procedure to be used on all P&T autosamplers. Run all standards from low to high concentration. For methanol extract analysis, add an amount of methanol to each standard that would approximate the total amount that would be used for sample analysis of methanol extracts. The volume of organic-free purged reagent water used for calibration must be the same volume used for sample analysis. For EnCore™ prep closed system soil purge and trap units calibration standards should also contain approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~1 g), as the presence of the preservative will affect the

purging efficiencies of the analytes. Separate initial calibrations will be prepared for EnCore™ prep soils and all other soils.

10.6.5 Calculate relative response factors (RRF_x) for each compound and surrogate using the following equation:

$$RRF_x = (A_x/A_{is})(C_{is}/C_x)$$

where:

A_x = Area of the characteristic ion for the compound to be measured (see Tables 10-6 and 10-6.1).
A_{is} = Area of the characteristic ion for the associated internal standard (see Tables 11-1).
C_{is} = Concentration of the internal standard (ng).
C_x = Concentration of the compound to be measured (ng).

10.6.6 Calculate the mean relative response factor

$$\text{Mean RRF} = \overline{RRF_X} = \frac{\sum_{i=1}^{n} RRFi}{n}$$

for all compounds and surrogates. Calculate the % Relative Standard Deviation (%RSD) of the RRF_x values for the initial calibration using the following equations:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RRF_i - \overline{RRF}_x\right)^2}{n-1}} \qquad \%RSD = \frac{SD}{\overline{RRF_x}} \cdot 100$$

where:

RRF_x = Mean RRF for each compound "x" or surrogate.
RRF_i = Response factor for each calibration level for the compound/surrogate of interest.
N = Number of calibration levels.

10.6.7 The percent relative standard deviation (%RSD) should be less than or equal to 15% for each target analyte. The %RSD for each Calibration Check Compound (CCC) must be less than or equal to 30%. The CCCs are:

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

See Sections 10.6.10 through 10.6.14 for action if these criteria are not met.

10.6.8 System Performance Check Compounds (SPCC) must have the following minimum mean relative response factors:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

Note: The above minimum mean relative response factors are based on a 5 mL sample size only. See 10.6.15 for action if these criteria are not met.

10.6.9 If the %RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the mean relative response factor may be used for quantitation.
10.6.10 The initial calibration is valid as long as the grand mean of the % RSD values for all analytes in the calibration is less than 15%. This is determined by reviewing the RSD check program to verify that the average RSD of all compounds is less than 15%. If this is true, then the analytes' mean relative response factor may be used for quantitation of each analyte for which the response factor exceeded 15%. This document is filed with the initial calibration data. See client-specific section for limits to this application.
10.6.11 The mean RSD criterion applies to all analytes in the standards, regardless of whether or not they are of interest for a specific project.
10.6.12 The data user must be provided with either a summary of the initial calibration data or a specific list of those compounds for which the RSD exceeded 15% and the results of the mean RSD calculation.
10.6.13 If a %RSD greater than 30% is measured for any CCC, take corrective action before reattempting calibration. If a % RSD for any other compound is greater than 30%, a regression fit may be used for the calibration curve for that compound. For a first order regression, r must be $\geq$0.990. For a second order regression, at least 6 calibration levels must be used and the coefficient of determination must be greater or equal to 0.990. (In the rare event a third order regression is used, 7 points must be used. Approval must be obtained from the supervisor.)
10.6.14 The analyst may not pick and choose compounds from whichever combination of runs proves to be compliant for that compound. Complete runs must be used, and the entire 6-point calibration must be run under a single clock.
10.6.15 If SPCC mean response factors do not meet minimum criteria (see Section 10.6.8) the analyst must either:

- rerun one of two calibration points if they have anomalously low RRF; or
- take corrective action and perform a completely new calibration.

10.6.16 A second source initial calibration verification (ICV) standard is analyzed after each initial calibration in order to achieve special project requirements. See Section 13 for individual client-specific quality control requirements for the ICV.
10.6.17 The second source ICV standard is prepared and analyzed as a laboratory control standard (LCS) using the VOA MS standard (see Table 8-2). See Sections 10.9, 10.10, and 10.11 for LCS preparation.

10.7 Calibration Verification

10.7.1 Perform a calibration verification within every 12-hour clock, immediately after each tune (Section 10.5).
10.7.2 Purge a 50 µg/L or 50 µg/kg standard (The VSTD050 standard in Section 10.6.4). The ~~8260 Dyna IS SS,~~ 8260 TK IS SS, 8260 PTA IS SS, Archon 8260 IS SS, and 4552 "D," ~~and~~ 4552 "F," and 4552 "N" IS SS mixes are now used in place of the individual IS and SS mixes for those P&T units. For methanol ex-

tract analysis, add an additional 100 µL of methanol per 5 mL total volume of purged water (closed loop P&T units use 1.0 mL per 50 mL purged water). The volume of organic-free purged reagent water used for calibration must be the same volume used for sample analysis. For closed system EnCore™ prep soil purge and trap units, calibration standards should also contain approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~1 g), as the presence of the preservative will affect the purging efficiencies of the analytes.

10.7.3 The SPCC must have the minimum relative response factors noted in Section 10.6.8

10.7.4 Quantitate each compound of the continuing calibration standard against the average response factor of the initial calibration using the following equation.

$$C_x = (A_x/A_{is})\ (C_{is}/\overline{RRF_x})$$

where:

C_x = Mass or concentration of the compound being measured.
C_{is} = Mass or concentration of the associated internal standard.
A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the associated internal standard.
RRF_x = Average relative response factor of the compound being measured.

10.7.5 Calculate the % difference (%D) of each analyte according to the following equation:

$$\%D = \left((RRF - \overline{RRF_x}) / \overline{RRF_x}\right) \cdot 100$$

where:

$\underline{RRF}$ = Measured response factor for compound (x) from analysis of calibration verification standard.
$\overline{RRF_x}$ = Mean response factor from initial calibration for compound (x).

10.7.6 If the %D is less than or equal to 20% for each CCC, the initial calibration is valid.

10.7.7 If the %D is greater than 20% for any one CCC, take corrective action and reanalyze the calibration verification standard. If reanalysis failed to meet %D criteria, a new 6-point calibration must be generated.

10.7.8 If a client required sublist does not contain all of the SPCCs or CCCs, then all compounds reported must have a %D less than or equal to 20%.

10.7.9 The primary ion area counts of the internal standards should not vary by less than one half or more than two times of the primary ion counts of the internal standards from the midpoint level of the initial calibration. The absolute retention time of the internal standards should not vary by more than +/- 30 seconds of absolute retention time of the internal standards from the midpoint level of the initial calibration.

10.8 Method Blank - (Water Analysis) ~~Run after each calibration verification standard.~~ Processed before any samples.

10.8.1 Add appropriate 8260 IS/SS to 5 mL or 25 mL purged ASTM Type II water.

10.8.2 Purge, trap and analyze the method blank under the same conditions used for calibration. Quantitate each compound detected against the average response factor of the initial calibration using the equation in Setion 11.1.2.

10.8.3 Check that surrogate recoveries fall within acceptable limits (Table 12-2).

10.8.4 Check that no target compounds are found above the quantitation limit (PQL Table 10-6) (see Section 2.3.2 for exceptions).
10.8.5 If any criteria are out-of-control, do not run any samples until corrective action is completed and a valid blank is run.

10.8.6 Method Blank (Soil Analysis)
10.8.6.1 Add 5 g of analyte free sand to vial containing 5 mL of purged ASTM Type II water and stir bar.
10.8.6.2 Check that surrogate recoveries fall within acceptable limits (see Table 12-2).
10.8.6.3 Check that no target compounds are found above the quantitation limit (PQL Table 10-6) (see Section 2.3.2 for exceptions).
10.8.6.4 If any criteria are out-of-control, do not run any samples until corrective action is completed and a valid blank is run.

10.8.7 For Method 5035, the Closed Loop Purge and Trap Soil Analysis:
10.8.7.1 Remove the sample vial from storage and allow to warm to room temperature.
10.8.7.2 Weigh the vial and contents to the nearest 0.01 g and record this weight. Add 5 g of analyte-free sand to vial containing 1 g sodium bisulfate. Weigh vial and contents to nearest .01 g and record weight. Add 5 mL purged ASTM Type II water.
10.8.7.3 Shake the sample vial gently to ensure that the contents move freely and that stirring will be effective.
10.8.7.4 Place the sample vial in the tray (EST Archon, O.I. Analytical 4552).
10.8.7.5 Add 5 mL of purged ASTM Type II water and IS/SS (added automatically by P&T unit).

10.8.8 Method Blank (Methanol Extract Analysis)
10.8.8.1 Add 10 µL of 8260 TK IS/SS and 100 µL of methanol to 5 mL of purged ASTM Type II water.
10.8.8.2 For closed loop P&T analysis, add 1 mL of methanol to 50-mL volumetric flask filled with purged ASTM Type II water. 8260 IS/SS is added automatically. Check that surrogate limits fall within acceptable limits (see Table 12-2).
10.8.8.3 Purge, trap and analyze the method blank under the same conditions used for calibration.
10.8.8.4 Quantitate each compound detected against the average response factor of the initial calibration using the equation in Section 11.1.2.
10.8. 8.5 Check that no target compounds are found above the quantitation limit (PQL Table 10-6) (see Section 2.3.2 for exceptions).
10.8. 8.6 If any criteria are out-of-control, do not run any samples until corrective action is completed and a valid blank is run.

10.9 LCS (Water Analysis)
10.9.1 5 µL of VOA MS 50 spike and 10 µL 8260 TK IS/SS to 5 mL of purged ASTM Type II water. Prepare at frequency in Table 12-1. For 5-mL water analysis using Dynatech PTA, ~~Dynatrap,~~ O.I. Analytical 4552, and EST Archon add 20 µL VOA MS 250 to 100 mL purged ASTM Type II water. Invert three times and transfer to VOA vials for analysis. For 25-mL water analysis using Dynatech PTA, ~~Dynatrap,~~ O.I. Analytical 4552, and EST Archon add 4 µL VOA MS 250 to 100 mL purged ASTM Type II water. Invert three times and transfer to VOA vials for analysis.
10.9.2 Calculate the recovery of the LCS. If the recovery of any designated analyte is outside the range given in Table 12-2, identify and correct the problem.
10.9.3 If an acceptable tune, verification standard, method blank, and laboratory control sample have been completed, the processing of samples may begin.

10.10 LCS (Soil Analysis)

10.10.1 Add ~~10 µL 8260 TK IS/SS and~~ 5 µL VOA MS 50 to 5 mL purged ASTM Type II water and 5 g of analyte-free sand and stir bar. For analysis on Jake also add 10 µL TK 8260 IS/SS. Prepare at frequency in Table 12-1.

10.10.2 Calculate the recovery of the LCS. If the recovery of any designated analyte is outside the range given in Table 12-2, identify and correct the problem.

10.10.3 If an acceptable tune, verification standard, method blank, and laboratory control sample have been completed, the processing of samples may begin.

10.10.4 For Method 5035 the closed loop P&T soil analysis, add 5 g of analyte-free sand to pre-weighed VOA vial containing l g $NaHSO_4$ and 5 mL of purged ASTM Type II water and stir bar.

10.11 LCS (Methanol Extract)

10.11.1 Add 10 µL of 8260 TK IS/SS, 5 µL VOA MS 50 and 100 µL methanol to 5 mL of purged ASTM Type II water. Prepare at frequency in Table 12-1.

10.11.2 For closed loop P&T analysis, add 10 µL of VOA MS 250 plus 1 mL methanol to 50-mL volumetric flask filled with purged ASTM Type II water. 8260 IS/SS is added automatically.

10.11.3 Calculate the recovery of the LCS. If the recovery of any designated analyte is outside the range given in Table 12-2, identify and correct the problem.

10.11.4 If an acceptable tune, verification standard, method blank, and laboratory control sample have been completed, the processing of samples may begin.

10.11.5 Additional spike mixes other than VOA MS 50 and VOA MS 250, may be used in order to fulfill client-specific spike lists.

10.12 Sample Analysis

10.12.1 Complete all analysis within a 12-hour clock, beginning at the injection time of the BFB tune check run.

10.12.2 Sample Analysis (Water)

10.12.2.1 Add 10 µL of 8260 TK IS/SS to 5 mL of water sample. The ~~Dynatrap,~~ Dynatech PTA, EST Archon, and O.I. Analytical 4552 instruments add the IS/SS automatically. For closed loop P&T analysis, surrogate may be added directly to the 43-mL glass VOA sample vial if 8260 IS only is being automatically injected. For the ~~Dynatrap,~~ Dynatech PTA, EST Archon, and O.I. Analytical 4552 instruments, 8.6 µL of 8260 SSS is added to the VOA vial (1.72 µL SSS for 25 mL).

10.12.2.2 After analysis, record the pH of all water samples in the injection logbook.

10.12.3 Sample Analysis (Soil)

10.12.3.1 Add 5 mL purged ASTM Type II water and approximately 5 g of soil sample. For analysis on Jake also add 10 mL 8260 TK IS/SS.

10.12.3.2 Quantitate each compound detected against the average response factor of the initial calibration using the equation in Section 11.1.2.

10.12.3.3 Soil samples exceeding concentrations of 2000 µg/kg of target analytes should be analyzed as a methanol extract.

10.12.4 Preparation of Samples from EnCore™ Samplers and Analysis by Method 5035, the Closed Loop Purge and Trap Soil Analysis

10.12.4.1 Three Encore™ samplers (approximately 4-5 g) normally are received for each sampling site. Preserve two for low-level Method 8260B soil analysis and one for medium-level Method 8260B soil analysis. Within 48 hours of sample collection, place an empty prelabeled VOA vial containing a stir bar on a tared balance. Add 1.0 g of sodium bisulflate monohydrate crystal (0.2 g of bisulfate per gram of soil). Tare the balance. Open the Encore™ sampler and place soil from first sampler into the VOA vial. Record weight of soil (to two decimal places) in a logbook and on the label. Add 5 mL of purged ASTM Type II water and seal vial. Repeat the above process with the second Encore™ sampler and a second prelabeled VOA vial. These two samples are now preserved for future Method 8260B soil analysis. Prep a batch blank along with the samples using Ottawa sand for the soil. Return the vials to sample custody if not analyzing immediately. Place the third empty prelabeled VOA vial on the balance and tare. Open the Encore™ sampler and add the soil to the vial. Record the weight (two decimal places) of the soil in the logbook and on the label. Add 10 mL purge and trap methanol and seal. This sample is now preserved for future medium level Method 8260B soil analysis.

10.12.4.2 Remove the sample vial from storage and allow it to warm to room temperature.

10.12.4.3 Shake the sample vial gently to ensure that the contents move freely and that stirring will be effective.

10.12.4.4 Add 5 mL of purged ASTM Type II water. IS/SS and 5 mL water are added automatically by purge and trap unit. Place the sample vial in the instrument tray.

10.12.4.5 Quantitate each compound detected against the average response factor of the initial calibration using the equation in Section 11.1.2.

10.12.5 Preparation of Soil Samples by the Frozen Soil Technique

10.12.5.1 An empty prelabeled VOA vial containing a clean magnetic stir bar is weighed to the nearest 0.01 g on a tared balance. The weight is recorded on the VOA vial and also in a logbook. The vial is shipped for soil sampling. Also store a VOA vial containing 5 g of Ottawa sand in freezer to act as a storage blank.

10.12.5.2 After sampling, the soil sample is maintained at -10 C (or lower) until sample analysis. For sample analysis, the frozen soil vial is brought to room temperature, wiped clean of condensation, weighed to the nearest 0.01 g on a tared balance, and recorded in a logbook. The amount of soil analyzed is determined by subtracting the empty prelabeled VOA vial from the sampled soil VOA vial (formerly frozen). Weigh the vial and contents to the nearest 0.01g and record this weight (even if the sample weight was determined in the field). If the weight found in the laboratory is withiin +/- 5% of the sample weight determined in the field, the field weight will be used for all calculations. Any significant discrepancies between the field and laboratory weights should be reported to the data users.

10.12.5.3 The sample is purged and concentrated using a closed loop purge and trap apparatus. 10 mL of purged ASTM Type II water and IS/SS is added automatically to the sampled soil VOA vial by the purge and trap unit. The sample is stirred by the magnetic stir bar throughout the purge cycle.

10.12.6 Sample Analysis (Methanol)

10.12.6.1 Samples analyzed as methanol extracts are either soil methanol extracts or liquid (oil) methanol extracts.

10.12.6.2 Soil Methanol Extracts

10.12.6.2.1 Prepare a soil methanol extract by adding approximately 4 g of sample to 10 mL of methanol. Shake for 2 minutes. Add 100 µL of this extract and 10 µL 8260 TK IS/SS to 5 mL purged ASTM Type II water.

10.12.6.2.2 For closed loop purge and trap analysis, add 1 mL of the prepared extract to a 50-mL volumetric flask filled with purged ASTM Type II water. Transfer to a 43-mL glass VOA vial and place on autosampler. 8260 IS/SS is added automatically.

10.12.7 Liquid (Oil) Methanol Extracts

10.12.7.1 If two layers of sample exist in the sample container, both layers must be analyzed.

10.12.7.2 Samples analyzed as liquid (oil) methanol extracts are either soluble or insoluble in methanol.

10.12.8 Liquid (Oil) Methanol Extracts Soluble in Methanol

10.12.8.1 Prepare a methanol soluble liquid (oil) methanol extract by adding 1 mL of sample (record weight) to 9 mL of methanol. Shake for 2 minutes. Add 100 µL of this extract and 10 µL 8260 TK IS/SS to 5 mL of purged ASTM Type II water.

10.12.8.2 For closed loop purge and trap analysis, add 1 mL of the prepared extract to a 50-mL volumetric flask filled with purged ASTM Type II water. Transfer to a 43-mL glass VOA vial and place on autosampler, 8260 IS/SS is added automatically.

10.12.9 Liquid (Oil) Methanol Extracts Insoluble in Methanol

10.12.9.1 Prepare a Methanol Insoluble Liquid (oil) methanol extract by adding 1 mL of sample (record weight) to 10 mL of methanol. Shake for 2 minutes. Add 100 µL of this extract and 10 µL of 8260 TK IS/SS to 5 mL of purged ASTM Type II water.

10.12.9.2 For closed loop Purge and Trap analysis, add 1 mL of the prepared extract to a 50-mL volumetric flask filled with purged ASTM Type II water. Transfer to a 43-mL glass VOA vial and place on autosampler. 8260 IS/SS is added automatically.

10.12.10 Field Collected Methanol Extracts for NJ RCRA

10.12.10.1 See Section 13.4, Special Project Requirements.

10.12.11 Sample Analysis (TCLP Extracts)

10.12.11.1 Add 10 µL of 8260B TK IS/SS and 0.5 mL of TCLP extract to 4.5 mL of purged ASTM Type II water. For closed loop purge and trap analysis, add 5 mL of TCLP extract to a 50-mL volumetric flask and bring to volume with purged ASTM Type II water. Transfer to a 43-mL VOA vial for analysis (IS/SS is added automatically by the closed loop P&T units).

10.13 MS/MSD Analysis

10.13.1 Run one set for each 20 or fewer samples by matrix when sample volume allows (methanol extract, if performed, requires an MS/MSD). Analyze the MS/MSD at the same dilution as the sample analysis (volume permitting).

10.13.2 MS/MSD Analysis (Water)

10.13.2.1 Add 10 µL of 8260 TK IS/SS and 5 µL of VOA MS 50 spike mix to 5 mL of sample. For the ~~Dynatrap,~~ Dynatech PTA, EST Archon, and O.I. Analytical 4552 instruments, add 8.6 µL of VOA MS 250 to the entire 43-mL VOA vial. For 25-mL water analysis, add 1.72 µL VOA MS 250 to the entire 43-mL VOA vial. The appropriate IS/SS mix is added automatically.

10.13.2.2 Quantitate each compound detected against the average response factor of the initial calibration using the equation in Section 11.1.2.

10.13.2.3 Check quantitation report that recoveries of designated spike compounds are within criteria in Table 12-2.

10.13.3 MS/MSD Analysis (Soil)

10.13.3.1 Add 5 µL VOA MS 50 to 5 mL of purged ASTM Type II water and 5 g of soil sample. For soil analysis on Jake, also add 10 µL 8260 TK IS/SS.

10.13.3.2 Quantitate each compound detected against the average response factor. The initial calibration using the equation in Section 11.1.2.

10.13.3.3 Check quantitation report that recoveries of spiked compounds are within criteria in Table 12-2.

10.13.3.4 If all criteria are met, recovery report is acceptable.

10.13.3.5 For Method 5035 the closed loop P&T soil analysis:

10.13.3.6 Remove the sample vial from storage and allow it to warm to room temperature.

10.13.3.7 Weigh the vial and contents to the nearest 0.01 g and record this weight (even if the sample weight was determined in the field). Any significant discrepancies between the field and laboratory weights should be reported to the data users.

10.13.3.8 Shake the sample vial gently to ensure that the contents move freely and that stirring will be effective.

10.13.3.9 Add 5 mL of purged ASTM Type II water and 5 µL VOA MS 50 to the soil sample. IS/SS is added automatically by purge and trap unit.

10.13.3.10 Place the sample vial in the instrument tray (EST Archon or O.I. Analytical 4552).

10.13.3.11 Quantitate each compound detected against the average response factor of the initial calibration using the equation in Section 11.1.2.

10.13.3.12 Check quantitation report that recoveries of designated spike compounds are within criteria in Table 12-2.

10.13.4 MS/MSD Analysis (Methanol)

10.13.4.1 Prepare a methanol sample extract according to Sections 10.12.6 to 10.12.9.

10.13.4.2 Add 10 µL 8260 TK IS/SS and 5 µL VOA MS 50 to 5 mL of the purged ASTM Type II water containing the added sample extract.

10.13.4.3 For closed loop P&T, add 8.6 µL of VOA MS250 directly to the 43-mL glass VOA vial filled with the purged ASTM Type II water containing the added sample extract.

10.13.4.4 Check quantitation report that recoveries of designated spike compounds are within criteria in Table 12-2.

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 Quantitative Analysis

11.1.1 When a compound has been identified, the quantitation of that compound is based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used is the one nearest the retention time of that of a given analyte. See Table 11-1.

11.1.2 The data system calculates final results. The equations below may be used to calculate the concentration of each identified analyte in the sample as follows:

purged water samples:

$$\text{Concentration } (\mu g/L) = \frac{(A_x)\,(C_{is})(DF)}{(A_{is})\,(\overline{RRF_x})\,(V_o)}$$

where:

A_x = Area of characteristic ion for compound being measured.
A_{is} = Area of characteristic ion for the internal standard.
C_{is} = Amount of internal standard injected (ng).
DF = Dilution factor.
$\overline{RRF_x}$ = Mean relative response factor of compound being measured.
V_o = Volume of purged water (mL), taking into consideration any dilutions made.

Sediment/Soil/Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis), methanol extract

Low Level:

$$\text{Concentration } (\mu g/kg) = \frac{(A_x)\,(C_{is})}{(A_{is})\,(\overline{RRF_x})\,(W_s)} \cdot \frac{(Df)}{(D)}$$

where:

A_x = Area of characteristic ion for compound being measured.
A_{is} = Area of characteristic ion for the internal standard.
C_{is} = Amount of internal standard injected (ng).
D = % dry weight of sample/100, or 1 for a wet-weight basis.
DF = Dilution factor.
$\overline{RRF_x}$ = Mean relative response factor of compound being measured.
W_s = Mass of sample (g) placed in purge vessel with 5 mL purged water and purged.

Medium Level:

$$\text{Concentration } (\mu gkg) = \frac{(A_x)\,(C_{is})\,(V_t)(DF)}{(A_{is})\,(\overline{RRF_x})\,(V_i)\,(W_m)\,(D)}$$

where:

A_x = Area of characteristic ion for compound being measured.
A_{is} = Area of characteristic ion for the internal standard.
C_{is} = Amount of internal standard injected (ng).
D = % dry weight of sample/100, or 1 for a wet-weight basis.
DF = Dilution factor.
$\overline{RRF_x}$ = Mean relative response factor of compound being measured.
W_m = Mass of sample (g) extracted in methanol (medium-level soils).
V_t = Volume of methanol used to extract sample (mL), normally 10 mL.
V_i = Volume of extract added (mL) for purging, normally 0.100 mL.

11.1.3 TCLP samples are analyzed at a tenfold dilution (10x) and are reported in mg/L (divided by 1000).

11.2 Qualitative Identification of Mass Spectra:
11.2.1 Mass spectral identification criteria:
11.2.1.1 For each individual instrument, a reference spectrum must be generated for each target compound of interest using the analytical conditions mentioned in Section 10. The spectrum is obtained at the peak apex and may include subtraction of interferences or background.
11.2.1.1.1 The characteristic ions present in the reference spectrum must be present in the sample spectrum. The characteristic ions are listed in Table 10-5.
11.2.1.1.2 The relative intensities of the ions must agree within +/-30% (i.e., 30 absolute units relative to the normalized abundance of the ion of interest).
11.2.1.1.3 Ions in the spectra of interest which have a relative intensity >10% of the most abundant ion, but which are not found in the reference should be carefully considered to determine if they are a significant component of the compound, or an interference. Seek guidance from the mass spectral specialist to determine the appropriate action in this case.
11.2.1.1.4 When a spectra does not meet all of the above criteria, but in the technical judgement of the analyst the compound may be present, seek guidance from the mass spectral specialist, who will make the final identification determination.
11.2.1.1.5 Consider each target compound found based on the mass spectra identification criteria of Section 11.2 and the retention time of that compound. The relative retention time should be within +\- 0.06 units of the relative retention time determined in the daily calibration standard. If it does not agree, but in the technical judgement of the analyst the compound may be present, seek guidance from the mass spectral specialist. **This case may arise in samples with excessive matrix effects.**

11.3 Tentatively Identified Compounds: (only if requested)
11.3.1 Perform mass spectral library searches on all additional peaks found in the sample run up to a maximum of 10 peaks. Exclude any peaks with area counts less than 10% of the area count of the nearest internal standard. Also, exclude the carbon dioxide peak at the beginning of the chromatogram, and exclude any peaks that are identified in the current run as target, internal, or surrogate compounds.
11.3.2 All tentatively identified compounds will be assigned from the mass spectral library by the automatic search routine.

11.4 Quantitation of Tentatively Identified Compounds:
11.4.1 Quantitate the peak using the total ion area counts for that peak, and the total ion area counts of the nearest internal standard. Apply the following formula:

$$\text{Final Conc TIC} = \frac{\text{Conc(IS)} \cdot \text{AreaCount (TIC)} \cdot \text{Df}}{\text{Area Count(IS)} \cdot \text{Vol}}$$

where:

Final Conc (TIC)	=	Units µg/L or µg/kg, per matrix.
Conc(IS)	=	Internal standard (ng).
Vol	=	Weight or volume used (g or mL, per matrix).
Df	=	Dilution factor.

11.5 MS/MSD Percent Recovery and RPD Calculations

11.5.1 Calculate the recovery of each matrix spike compound in the matrix spike and matrix spike duplicate and report on the appropriate forms. Calculate the concentrations of the matrix spike compounds using the same equations as used for target compounds. Calculate the recovery of each matrix spike compound as follows:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \cdot 100$$

where,

SSR = Spike sample result.
SR = Sample result.
SA = Spike added.

11.5.2 Calculate the relative percent difference (RPD) of the recoveries of each compound in the matrix spike and matrix spike duplicate as follows:

$$RPD = \left| \frac{S-D}{1/2(S+D)} \right| \cdot 100$$

where:

RPD = Relative percent difference.
S = Matrix spike recovery.
D = Matrix spike duplicate recovery.

The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

11.6 SURROGATE PERCENT RECOVERY CALCULATION

11.6.1 Calculate the concentrations of the surrogate compounds using the same equations as used for the target compounds. Calculate the recovery of each surrogate using the following equation:

$$\%\,\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \cdot 100$$

11.6.2 Calculate surrogate standard recovery on all samples, blanks, matrix spikes and matrix spike duplicates. Determine if recovery is within limits (see Table 12-2) and report on appropriate form.

Table 11-1

VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS AND SURROGATES ASSIGNED FOR QUANTITATION

Fluorobenzene	Chlorobenzene – d5	1,4-Dichlorobenzene – d4
Chloromethane	2-Hexanone	1,1,2,2-Tetrachloroethane
Bromomethane	4-Methyl-2-pentanone	Bromofluorobenzene (surr)
Vinyl chloride	Tetrachloroethene	1,2-Dichlorobenzene
Chloroethane	Toluene	1,3-Dichlorobenzene
Methylene chloride	trans-1,3-Dichloropropene	1,4-Dichlorobenzene
Acetone	Chlorobenzene	
Carbon disulfide	Dibromochloromethane	
1,1-Dichloroethene	1,1,2-Trichloroethane	
1,1-Dichloroethane	Ethylbenzene	
trans-1,2-Dichloroethene	Styrene	
cis-1,2-Dichloroethene	Bromoform	
Chloroform	Xylene (m+p)	
1,2-Dichloroethane	Xylene (o)	
2-Butanone	Toluene-d8 (surr)	
1,2-Dichloroethane-d4 (surr)		
Trichlorofluoromethane		
Vinyl acetate		
Dibromofluoromethane (surr)		
1,1,1-Trichloroethane		
Carbon Tetrachloride		
Bromodichloromethane		
1,2-Dichloropropane		
Trichloroethene		
Benzene		
Cis-1,3-Dichloropropene		
2-Chloroethylvinyl ether		
Dichlorodifluoromethane		

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12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
BFB standard (50 ng) (BFB)	At beginning of every 12 hours of analysis	Inject 1 µL into GC/MS	See Table 10-5	Manual tune with PFTBA
Calibration verification standard (50 µg/L or 250 ng) (VSTD050)	Following BFB	See Section 10.7	%D of CCC <20% min. RF and SPCC according to Section 10.7.	Reanalyze adjust instrument conditions. Remake standard. Analysis may continue for analytes with high verification standard recovery if that analyte is non-detect in the sample. If problem cannot be corrected due to hold times, comment on effect on sample bias. Stop future analyses until problem identified and corrected.
Method Blank	One per batch	See Section 10.8	All compounds less than QL except (See Section 2.5.2). Surrogate standard (SS) recoveries as per E & E limits.	Reanalyze in same batch Stop sample analysis. Comment on effect on sample data.
Surrogate Recoveries (SS) (SUR)	Each sample, blank and QC analysis	See Section 10.12	All Recoveries as per E & E limits. See Control Limit Summary, 8260B. If statistical limits are <115%, then recoveries up to 115% are acceptable without corrective action.	Check for errors. Reanalyze sample, unless obvious matrix effects. Comment on effect on sample data.
Internal Standard responses and retention times (IS)	Each sample, blank and QC analysis	See Section 10.12	Retention times for any IS in all standards must vary less than 30 seconds from last calibration check. Areas for any IS must vary less than a factor of 2 from last calibration check.	Inspect for malfunction. If corrections are made, reanalyze affected samples.

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS)	One per batch of 20 or fewer samples for client-specified compounds	See Section 10.9-10.11	Recovery as per E & E limits. See Section 12.1	Stop analysis until problem identified and corrected. If problem cannot be corrected due to hold times, comment on effect on sample bias. Stop future analyses until problem identified and corrected.
Matrix Spike (MS) Matrix Spike Duplicate with each MS (MSD)	One per 20 or fewer samples in a batch (sample volume permitting). Requires a minimum of three VOA vials collected.	See Section 10.13	Project requirements if specified	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.

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Control Limits for Routine Quality Control Samples

Table 12-2

QUALITY CONTROL RECOVERY LIMITS

QC Type	Parameters Charted	Type of Samples Charted	Source of Limits	Frequency Updated
Surrogates	Dibromofluoromethane 1,2-Dichloroethane-d4 Toluene-d8 4-Bromofluorobenzene	Soil/water	E & E lab limits. See Control Limit Summary, 8260B	Yearly
LCS	All target compounds	Soil/purged water	E & E lab limits. See Control Limit Summary, 8260B	Yearly

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12.1 Reporting of LCS
12.1.1 Spike with all target compounds.
12.1.2 In the absence of client requirements to the contrary, the specified number of sporadic QC failures given in Table 13-1 may be used.
12.1.3 If a client does not specify or does not desire a full list, then a reduced spiking list of 1,1-dichloroethene, trichloroethene, benzene, toluene, and chlorobenzene may be reported. However, all reduced list compounds must pass and all spiked compounds must remain in the quantitation report. These additional compounds enter the LIMS and are monitored as ongoing recovery studies (control charting).

13.0 SPECIAL PROJECT REQUIREMENTS

Client-Specific Quality Control Requirements
13.1 **Jacobs; Otis** - LCS and LCSD required.

13.2 **U.S. Army Corps of Engineers (ACE)**
13.2.1 Method Quantitation Limit (MQL) is between 3-10 times MDL. The low calibration standard concentration must be ≥3 times MDL. The Method Reporting Limit (MRL) may slide between two times the MDL and project-specific or regulatory requirements. Results between the MQL and MRL must be reported as estimated (J flagged). Results below the MRL must be reported "ND."
13.2.2 If using the grand mean approach for RSD (Section 10.6.20), no single analyte may be >30% RSD.
13.2.3 A second source initial calibration verification (ICV) standard is analyzed after each initial calibration curve. A maximum of ±20% of expected recovery of each target analyte in the ICV is determined from the initial calibration. Note the number of allowable QC failures as given in Table 13-1 below.

Table 13-1

NUMBER OF ALLOWABLE QC FAILURES

Number of Compounds in Calibration Standard	Number of Allowable QC Failures
5 – 15	1
16 – 30	2
31 – 45	3
46 – 60	4
61 – 75	5
76 – 90	6

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13.2.4 Continuing calibration verification (CCV) must be done daily before sample analysis and every 12 hours of analysis time. Percent difference for CCC must be < 20. The concentration of all other target analytes and surrogates must be within 20% difference of their expected values.
13.2.5 Method blanks containing common laboratory solvents below one-half and up to the reporting limit will be acceptable.
13.2.6 The LCS must contain all target analytes unless project specifies otherwise. Recoveries for water LCS must be 80-120%, solid LCS 75-125%. Sporadic marginal failures (Table 13-1) of 60-140% can be accepted.
13.2.7 After verifications of tuning, the STD and LCS must be loaded together. When the LCS has passed all requirements, load the method blank and samples on the purge and trap unit. The results must be reported for TIC compounds.

13.2.8 ACE criteria are summarized in Table 13-2.

Table 13-2

Summary of Method Quality Objectives for Method 8260 VOCs

QC Element	Target Analyte/Surrogate	Poor Purgers/Gases/Sporadic Marginal Failures[1]
Initial Calibration (Shell document section 9.2.2.4)	Instrument Evaluation: SPCCs: minimum RF values per method requirements CCCs: verify %RSD ≤ 30% Primary Evaluation: $r \geq 0.995$, %RSD ≤ 15%, $r^2 \geq 0.990$ Alternative Evaluation: Mean %RSD for all target analytes ≤ 15%	No allowance Alternative Evaluation: Maximum allowable %RSD for each target analyte ≤ 30%
ICV (Shell document section 9.3)	%Rec = 80% - 120%	Sporadic Marginal Failures[1]: %Rec = 60% - 140%
CCV (Shell document section 9.5/9.5.2/9.5.2.4)	Instrument Evaluation: SPCCs: minimum RF values per method requirements CCCs: verify %D ≤ 30% Primary Evaluation (CCCs): %Drift ≤ 20%, %D ≤ 20%	Primary Evaluation (remaining target analytes): Qualitative, see text
MB (Shell document section 10.2.1/11.4.1)	Target Analytes: Analytes < MDL Check Sample (~2X MDL)	Common Lab Contaminants: Analytes < MQLs
LCS (10.2.2/11.4.2)	Water: %Rec = 80% - 120% Solids: %Rec = 75% - 125%	Sporadic Marginal Failures[1]: %Rec = 60% - 140%
MS (Shell document section 10.2.3/11.4.3/11.4.3.2)	%Rec = 70% - 130%	Sporadic Marginal Failures[1]: %Rec = 60% - 140%
MSD/MD (Shell document section 10.2.4/11.4.4)	Water: RPD ≤ 30% Solids: No RPD Limits	Water: RPD ≤ 40% Solids: No RPD Limits
Surrogates (Shell document section 10.2.5/11.4.5)	%Interference-Free Matrix: Water: %Rec = 80% - 120% Solids: %Rec = 75% - 125% Project Sample Matrix: %Rec = 70% - 30%	No Applicable

[1] The number of Sporadic Marginal Failure (SMF) allowances depend upon the number of target analytes reported from the analysis. For instance, if the full list of 68 compounds are reported from the GC/MS analysis, then five (5) SMFs are allowed to the expanded criteria presented for the ICV and LCS. If the MS includes only a subset of compounds, allow only one (1) SMF for this QC element. Refer to Section Shell document section 9.3 for additional information on the application of sporadic marginal failures.

Source: USACE, Shell for Analytical Chemistry Requirements, Version 1.0, November 2, 1998.

13.3 AFCEE - General Requirements

13.3.1 Reporting:

13.3.1.1 MDL may not be > 1/2 reporting limit (RL) unless a variance was obtained.

13.3.1.2 A calibration standard at or below the RL must be included.

13.3.1.3 Apply a "U" flag if analyte result <MDL; an "F" flag if MDL ≤ analyte result ≤ RL.

13.3.2 Calibration

13.3.2.1 Analyze an initial calibration verification standard form (ICV) a second source after 6 point curve. File with calibration curve. All compounds should be less than 15% D.

All initial calibration curves, inorganic or organic, must be **verified with an independent source (vendor) standard** prior to sample analysis unless an independent vendor is unavailable and a variance was obtained.

13.3.2.2 Quantitate each compound of the ICV standard against the average response factor of the initial calibration according to the equation in 10.7.4. Calculate the percent Difference (%D) of each analyte according to the equation in 10.7.5. All compounds must have a % D <15% (or as per client requirements).

13.3.2.3 The daily (organic) and continuing (organic and inorganic) calibration verifications are not required to be independent, however, the continuing calibration verification may not be used as the LCS (volatiles, mercury, any test without a preparation).

13.3.3 Batching. A batch is 20 environmental samples. These 20 samples *include* the MS/MSD or MS/MD.

13.3.3.1 An AFCEE analytical batch is the environmental samples that are similar in composition (matrix) and are prepared at the same time and with the same **lot of reagents.**

13.3.3.2 A batch is not to exceed 20 samples. **The sample duplicate, MS, MSD are considered an environmental sample, therefore, the actual number of unique samples is less than 20.**

13.3.4 Laboratory Control Sample

13.3.4.1 Laboratory Control Sample for solid must be prepared using **Ottawa sand** unless a variance was obtained.

13.3.4.2 The LCS must be prepared from second source material.

13.3.4.3 The LCS must contain all target analytes unless noted differently under specific method requirements or a variance was obtained.

13.3.4.4 The LCS must be spiked at a concentration ≤ the midpoint of the calibration curve for each analyte unless a variance was obtained.

13.3.4.5 Ottawa sand must be used as the solid matrix to prepare the solid LCS.

13.3.5 Matrix Spike/Matrix Spike Duplicate

13.3.5.1 The MS and MSD must be spiked at a concentration ≤ midpoint of the calibration curve for each analyte unless a variance was obtained.

13.3.5.2 AFCEE samples must be used for spiking.

13.3.6 Surrogates (Organic analyses)

13.3.6.1 **Whenever one surrogate is outside limits,** corrective action must be performed.

13.3.7 When **retention time** is outside windows, perform corrective action.

13.3.8 Method blank must be less than RL unless a variance was obtained.

13.3.9 AFCEE Project-Specific Method Requirements

13.3.9.1 Holding time for waters is 7 days if unpreserved.

13.3.9.2 Retention times must be updated at each initial calibration and each calibration verification unless a variance was obtained.

13.3.9.3 The AFCEE term second source calibration verification is the laboratory term initial calibration verification (ICV), run once after a curve is analyzed. Do not confuse with the AFCEE term ICV (see below).

13.3.9.4 The AFCEE term of initial calibration verification is the same as the laboratory term daily calibration check. It may not be prepared from the same material used to prepare the LCS. Use second source material for the LCS and use calibration material for the AFCEE term ICV and continuing calibration verifications.
13.3.9.5 RSD for CCC must be ≤ 30%, RSD for all other calibration analytes must be ≤15%.
13.3.9.6 Second source calibration must be performed after a five-point curve and must be ±25% of expected.
13.3.9.7 Continuing calibration verification must be done daily before sample analysis and every 12 hours of analysis time. Percent difference for CCC must be <20% and all calibration analytes within ±20 % of expected.
13.3.9.8 Calibration check standard IS retention time must be ±30 seconds and IS area within 1/2 to 2 times that of last calibration verification (12 h).

13.4 START, LONG BEACH
13.4.1 MTBE oxygenate compounds are analyzed as additional compounds. These compounds include:

1) Methyl tert-butyl ether, primary ion = 73, QL = 5 µg/L, quantitating from IS = fluorobenzene.
2) Ethanol, primary ion = 45, QL = 2,000 µg/L, quantitating from IS = fluorobenzene.
3) tert-Butanol, primary ion = 59, QL = 100 µg/L, quantitating from IS = fluorobenzene.
4) Di-isoproypyl ether, primary ion = 45, QL = 5 µg/L, quantitating from IS = fluorobenzene.
5) Ethyl tert-butyl ether, primary ion = 59, QL = 5 µg/L, quantitating from IS = fluorobenzene.

13.4 NJ RCRA – Field Collected Methanol Extracts
13.4.1 Collection jars are prepared and pre-weighed in the laboratory before shipment to the field.
13.4.2 4-oz. Screw-top jars are filled with 24 mL methanol and spiked with 1 mL 8260B surrogate solution.
13.4.3 Jars with caps are weighed and the weight recorded in the NJ Methanol Extraction Field Prep Log. Each log entry and corresponding jar is numbered with the standard lab designation of "logbook-page-line number."
13.4.4 An extra jar is prepared as above for a trip blank.
13.4.5 Field technicians add approximately 10 g of soil to each jar except the trip blank.
13.4.6 Jars are returned to the laboratory where the analyst reweighs the jar and subtracts that weight from the original jar weight to determine sample weight.
13.4.7 Samples are analyzed as normal methanol samples. The sample weight used in calculation is the value determined above, and the correction factor is 1250.

13.5 DEC – Tentatively Identified Compounds (TICs)
13.5.1 Perform mass spectral library searches on all additional peaks found in the sample run up to a maximum of 10 peaks. Exclude any peaks with area counts less than 10% of the area count of the nearest internal standard. Also, exclude the carbon dioxide peak at the beginning of the chromatogram, and exclude any peaks that are identified in the current run as target, internal, or surrogate compounds.
13.5.2 Compare each TIC with the top three library spectra generated by the automatic search routine. Use as a guide to assign the identification name to the TIC if the match of the best fit is >90%. If the compound of interest has multiple isomers of identical spectra, assign a classification name to the compound (e.g., trimethylbenzene isomer). If in the technical judgement of the analyst, the spectra represents a member of a class of compounds, that classification should be assigned to the compound (e.g., unknown hydrocarbon). If no identification can be assigned to the compound, assign the name "Unknown."

13.6 SAIC – Soil Analysis

13.6.1 All soils are prepared and analyzed using the frozen soil technique (See Section 10.12.5).

13.7 Illinois EPA

13.7.1 An ICV must be analyzed with the mean %D < 15%.

13.8 North Carolina DENR (ACC Seymour Johnson)

13.8.1 An MS/MSD pair must be analyzed every **10** samples.

13.8.2 The daily calibration verification must be at mid-concentration (100 µg/L for most compounds) at calibration range, NOT at 50 µg/L).

14.0 SAMPLE DISPOSAL

14.1 See SOP A.10, Waste Disposal

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: TPH by TNRCC, Method TX1005			
Reviewer: C. Lewandowski		Peer Reviewer: None	
Implementation Date: 8/2/99		Last Annual Review Date: 11/6/00	
Revision: 0	Status: Final	Method: TX1005	Minor Revision Date: 3/26/2001

1.0 SCOPE AND APPLICATION

1.1 This method outlines the procedure for determining the presence of C6 - C28 (peak start/end) hydrocarbons and the quantitation of these compared to a gasoline/ diesel fuel standard. This method can be employed for both aqueous or soil samples following the extraction procedures outlined in this SOP. Other fuel types can be analyzed including jet fuel, kerosene and fuel oil.

2.0 METHOD SUMMARY

2.1 This method involves extraction of a soil with methanol and n-pentane or a water sample with n-pentane.

2.2 A (2.0 µL) aliquot of sample extract is injected into a capillary column gas chromatograph equipped with an FID detector and calibrated for gasoline and diesel range organics analysis. The total chromatographic area representing peak start of C6 to peak end of C28 in the sample is compared to the corresponding area in the standard for quantitation.

2.3 This method is based in part on USEPA Methods 8000,8015, and 8100, SW-846, "Test methods for Evaluation of Solid Waste," 3rd edition. It was developed at Shell Development Company. It is used primarily for the petroleum storage tank division of the Texas Natural Resource Council.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 TNRCC Method 1005 (revised 4/13/98).
4.2 Training SOP QA.5, A.23, and A.29.
4.3 Data Review SOP A.25.
4.4 Laboratory Quality Assurance Manual.
4.5 MDL SOP A.18.
4.6 Corrective Action Procedure, QA.1

5.0 DEFINITIONS/ACRONYMS

5.1 **Total Petroleum Hydrocarbons (TPH)** - Defined as all gas chromatographic peaks eluting after the solvent (n-pentane) starting with and including n-hexane (C6) to noctacosane (C28). This definition includes aliphatic and aromatic hydrocarbons. The TPH for each range C6-C10 and >C10-C28 are analyzed simultaneously, but are quantified separately as part of the whole. The petroleum hydrocarbons in a sample (if any) may not encompass the entire range. There may be non-hydrocarbon compounds that elute in this

range (such as chlorinated solvents, ketones, alcohols, etc.). However, such compounds usually appear as discrete peaks and do not match the typical petroleum product fingerprints. These compounds are quantified as part of the TPH, but the sample should be flagged as containing non-petroleum compounds.

5.2 Approximate Boiling Point/Carbon Number Distribution - Defined as the subdivision of the chromatogram into sections that correspond to the boiling and/or volatility of n-alkanes. The gas chromatographic separation is achieved using a column that separates components based in boiling point differences. This separation can be correlated to approximate carbon number. For example, >C10 to≤ C28 indicates those hydrocarbons that elute after decane and up to and including octacosane. This range includes most,but not all, of the hydrocarbons within the carbon range. Branching lowers the boiling point of hydrocarbons relative to their n-alkane isomer. Cyclization, or ring structures, raises the boiling point higher than the n-alkanes of the same carbon number.

5.3 Locator Mix Standard - Used to determine the ranges C6-C10 and >C10-C28. The locator mix standard must contain, but is not limited to C6, C10, and C28 n-alkanes.

5.4 Analytical Batch - A set of one to 20 samples prepared on the same day.

5.5 Continuing Calibration Standard: Midpoint calibration standard analyzed every 10 samples to verify the calibration curve.

5.6 Laboratory Control Sample - A processed QC sample prepared by spiking reagent water or sand with the intermediate gas/diesel stock used in preparing the calibration standard.

5.7 MDL - Method Detection Limit as determined by 40 CFR Part 136, Appendix B. Method detection limits must be determined yearly for both water and soil matrices. See SOP A.18 for procedure.

5.8 PQL - Practical Quantitation Limit is the concentration above the MDL that can be reasonably obtained and is used as the limit of reporting.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Other organic compounds falling within the retention time window are measurable by this method and are included in the DRO results.

6.2 Contamination by carryover may occur especially when dealing with heavy weight hydrocarbons such as motor oils. If carryover is expected, additional solvent blanks should be run after potential problem samples in order to minimize the interference.

7.0 INSTRUMENTATION AND EQUIPMENT

Gas chromatograph equipped with flame ionization detector (FID) and data system.
Column: 60-m, 0.25-mm RTX-5 (or equivalent).

7.1 Apparatus Set-up

7.1.1 Gas chromatographic conditions, Nominal. Conditions may be altered to optimize.

Carrier gas: Helium at ~5 mL/min
Injector Temp.: 250 C
Detector Temp.: 325 C
Column Temp.: 35 C/2 min; Ramp @ 10 C/min to final temp of 320 C hold 8 min.

7.2 Analytical balance capable of accurately weighing to 0.0001 gram should be used for preparation of standards. A top loading balance capable of weighing to the nearest 0.01 gram should be used for obtaining sample weights.

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standards and Reagents.

8.2 All stock standards expire one year from receipt unless the vendor supplies an expiration date on the standard container. All working standards expire 6 months from preparation or the expiration date of its stock, whichever is sooner.

8.3 All standards are stored at 2-6 C.

8.4 Table 8-2 outlines standard preparation.

8.5 The ~~DRO~~ TXTPH Locator Mix standard refers to the n-alkane standard used to establish retention time windows.

Summary of Standards and Reagents

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Methylene chloride ($MeCl_2$)	JT Baker Ultra Resi-analyzed or equivalent	Neat
Methanol (MeOH)	JT Baker Ultra Resi-analyzed or equivalent	Neat
Hexane	JT Baker Ultra Resi-analyzed or equivalent	Neat
Pentane	JT Baker Ultra Resi-analyzed or equivalent	Neat
#2 Diesel fuel	Exxon, Mobil or equivalent	Neat
Gasoline (89 Octane)	Exxon, Mobil or equivalent	Neat
~~Diesel Range Organics (DRO) (Normal alkane standard)~~	~~Restek, Supelco or equivalent~~	~~2000 µg/mL~~
Restek Calibration Mix (TXTPH Mix Catalog No. 31483)	Restek	10,000 µg/mL
TXTPH Locator Mix Catalog No. 31482	Restek	200 µg/mL
TXTPH Matrix Spike Mix Catalog No. 31484)	Restek	10,000 µg/mL

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Standard Solution Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
TNRCC/TX1005 (diesel/gasoline) ~~Intermediate~~ Stock	#2 Diesel Fuel Gasoline	1.25 g of #2 Diesel Fuel 1.25 g of Gasoline to 50 mL 1:1 MeOH:$MeCl_2$	25,000 µg/mL (nominal) 25,000 µg/mL (nominal)
TX1005 Low Calibration Standard	~~TNRCC/TX1005 Intermediate~~ Restek Calibration Mix	~~20 µL to 10 mL $MeCl_2$~~ 25 µL to 5 mL pentane	50 µg/mL ~~each (gas/diesel)~~
TX1005 M Low Calibration Standard	~~TNRCC/TX1005 Intermediate~~ Restek Calibration Mix	~~40 µL to 10 mL $MeCl_2$~~ 50 µL to 5 mL pentane	100 µg/mL ~~each (gas/diesel)~~
TX1005 Mid (CCV) Calibration Standard	~~TNRCC/TX1005 Intermediate~~ Restek Calibration Mix	~~100 µL to 10 mL $MeCl_2$~~ 250 µL to 10 mL pentane	250 µg/mL ~~each (gas/diesel)~~
TX1005 M High Calibration Standard	~~TNRCC/TX1005 Intermediate~~ Restek Calibration Mix	~~200 µL to 10 mL $MeCl_2$~~ 250 µL to 5 mL pentane	500 µg/mL ~~each (gas/diesel)~~
TX1005 High Calibration Standard	~~TNRCC/TX1005 Intermediate~~ Restek Calibration Mix	~~500 µL to 10 mL $MeCl_2$~~ 625 µL to 5 mL pentane	1250 µg/mL ~~each (gas/diesel)~~
~~N-Alkane Locator Mix~~	~~Diesel Range Organics (DRO)~~ ~~Hexane (C6)~~ ~~Restek Calibration Mix~~	~~250 µL DRO~~ ~~1 µL of hexane to 10 mL $MeCl_2$~~	~~50 µg/mL (C10-C28)~~ ~~100 µg/mL (C6)~~
TX1005 Locator Mix	Restek TXTPH Locator Mix	1000 µL to 10 mL pentane	20 µg/mL
TX1005 Reference Standard	Restek TX1005 Stock	100 µL to 10 mL pentane	250 µg/mL

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9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/ Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Solid	Standard	14 days from collection	14 days from collection	4-, 8-, or 16-oz. jar
Water	Standard	14 days from collection	14 days from collection	40-mL VOA vial w/$NaHSO_4$ to pH<2

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10.0 PROCEDURE

10.1 Extraction

10.1.1 Soil Extraction. Extract soil samples using a vortex mixer or shake technique.

10.1.1.1 Weigh 10 grams of sample into a 40-mL VOA vial with Teflon® cap. Record the weight to the nearest 0.01 grams. Add 2 mL of methanol to extract the water from the soil and neutralize surface charges on the clay particles, cap and vortex for 20 seconds. DO NOT ADD AN EXCESS OF METHANOL due to the tendency to form an emulsion. Add 10 mL of n-pentane (top layer), cap the vial and proceed with the extraction. To aid in the separation of pentane from methanol, 1-2 mL of water ~~can~~ should be added to the mixture. Transfer adequate volume of top (pentane) layer into two autosampler vials, one for storage and one for analysis.

10.1.1.2 For a method blank, weigh 10 grams of Ottawa sand and extract like a sample.

10.1.1.3 For laboratory control samples (LCS) weigh 10 grams of Ottawa sand. For matrix spikes, weigh a separate 10-gram portion of sample. Add ~~100 µL of TX1005 Intermediate~~ 250 µL Restek TXTPH MS and extract like a sample.

10.1.1.4 Extract blanks, samples, LCS, matrix spike, matrix spike duplicates by vortexing for at least one minute.

10.1.1.5 If particulate is suspended in the solvent layer or an emulsion forms, centrifugation may be necessary to obtain a clear solvent layer. Transfer a portion of the extract to autosampler vials for direct analysis of the extract for total petroleum hydrocarbons. The extract may be stored in vials with Teflon® caps. Extracts should be stored in GSP at <6 C.

10.1.2 Water extraction. Extract water samples using vortex mixer.

10.1.2.1 Remove sample vial from refrigeration and allow to come to ambient temperature. Dry the outside of the vial with a paper towel(if the vial is filled with no head space remove ~10 mL [pour off top]). Weigh the vial and its contents on a top loading balance and record the weight to the nearest 0.01g.

10.1.2.2 Add exactly 3.0 mL of n-pentane using a glass syringe through the septum.

10.1.2.3 For a method blank, fill a clean VOA vial with 30 mL of reagent water and extract like a sample.

10.1.2.4 For LCS, fill a clean VOA vial with 30 mL of reagent water and add ~~50 µL of TX1005 Intermediate 125~~ 750 µL Restek TXTPH MS or 300 µL TNRCC/TX1005 stock and extract like a sample.

10.1.2.5 Extract blanks, samples, LCS, matrix spike, matrix spike duplicates by vortexing for at least one minute.

10.1.2.6 Remove extract by pipette and store in Teflon® capped vials in GSP at <6 C.

10.1.2.7 Discard the water from the VOA vial and dry the vial, lid, and septum.

10.1.2.8 Reassemble the vial and weigh it on a top loading balance and record the weight to the nearest 0.01 gram.
10.1.2.9 Determine the sample volume by subtracting the dry vial weight obtained in Section 10.1.2.8 from the initial weight obtained in Section 10.1.2.1.

10.2 Apparatus Set-up
10.2.1 Gas chromatographic conditions, Nominal. Conditions may be altered to optimize.
Carrier gas : Helium at ~5 mL/min
Injector Temp.: 250 C
Detector Temp.: 325 C
Column Temp.: 35 C/2 min; Ramp @ 10 C/min to final temp of 320 C hold 8 min.
10.2.2 2 µL injections are used.

10.3 Retention Time Windows
10.3.1 Prior to analysis, and at least yearly, establish retention time windows by injecting the Normal Alkane Standard (C6 - C28) three times over a minimum 72-hour period. The retention time window is defined as ± 3 times the standard deviation of the absolute retention time for each of the components in the n-alkane locator mix.
10.3.2 Retention time windows must be established for each GC column used for this analysis and whenever a new column is installed.

10.4 Initial Calibration
10.4.1 Perform a calibration at the five levels of the gasoline/diesel (TX1005) calibration standards, 50, 100, 250, 500 and 1250 µg/mL (Low, Mlow, Mid, Mhigh, High)
10.4.2 Calculate the area response versus the amount injected (area/ concentration) for each point (calibration factor, CF) for all three ranges (C6-C10,>C10-C28,C6-C28). The calibration is acceptable if the %RSD (relative standard deviation) of the five points is ≤25% over the working range for each range. Linear regression analysis may be used so long as the correlation coefficient (r) is at least 0.995.
10.4.3 If an acceptable initial calibration cannot be achieved, perform corrective maintenance (Table 10-1) and reanalyze the initial 5-point calibration. If the initial calibration fails again, prepare a new calibration curve (Table 8-2) and reanalyze.

10.5 Continuing Calibration Verification
10.5.1 Verify the initial calibration by analysis of the TX1005 mid calibration standard (continuing calibration verification standard; CCV) at 250 µg/mL each (gasoline/diesel). The percent difference in response from the average initial response must not exceed 25% for any of the ranges or a new initial calibration must be performed. The % difference is the difference between the average response factor from the calibration curve and the response factor of the daily continuing standard.

$$\%\,\text{difference} = \frac{|CF_D - CF_C|}{CF_C} \cdot 100$$

where:

CF_D = Calibration factor of continuing standard.
CF_C = Average calibration factor from the calibration curve.

10.5.2 Initiate a 24-hour quantitation window by injecting the Normal Alkane Standard to establish retention times.
10.5.3 Prior to sample analysis, a pentane blank must be analyzed to determine that a normal baseline exists under current operating parameters. The area is generated by projecting a horizontal baseline between the peak start of C6 and the peak end of C28 retention times. When quantitating samples **DO NOT** baseline subtract.
10.5.4 The continuing calibration verification standard (CCV) at the midpoint concentration must be analyzed at a minimum of every ten samples and at the end of each sequence. If the standard does not fall within 25% of the true value, all samples prior to the failed QC and since the last acceptable CCV standard must be reanalyzed.
10.5.5 If an acceptable CCV cannot be analyzed, it may be necessary to clean the injector, bake out the column or detector, or replace the FID jet.
10.5.6 If an acceptable CCV can be run after the corrective action steps in Section 10.5.5, sample analysis may continue. If not, a new 5-point curve must be run (see Section 10.4).

10.6 Analysis of Extracts
10.6.1 Extracts of samples are analyzed by 2 µL direct injection.
10.6.2 Using the chromatographic conditions listed, peak areas from peak start of C6 to peak end of C28 are used to determine the total diesel fuel and gasoline.
10.6.3 Total area and the partial areas (from peak start of C6 to peak end of C10 and peak end of C10 to peak end of C28) in these windows are used for all sample calculations.
10.6.4 Typical **daily run sequence** is as follows:
10.6.4.1 n-Pentane solvent blank
10.6.4.2 n-Alkane Locator Mix
10.6.4.3 Mid Calibration Standard (CCV)
10.6.4.4 Method blank
10.6.4.5 Laboratory Control Sample
10.6.4.6 Eight-sample analyses
10.6.4.7 n-Pentane solvent blank
10.6.4.8 CCV
10.6.4.9 10 samples
10.6.4.10 n-pentane solvent blank
10.6.4.11 n-Alkane Locator Mix
10.6.4.12 CCV
10.6.4.13 Repeat 10.6.4.4 and 10.6.4.12 to end of sequence.

Instrument Conditions and Maintenance

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Gas Chromatograph	Retention time shift	Change septum	Daily or as needed
Gas Chromatograph	High Background	Change injection port liner	As needed
Gas Chromatograph	High Background	Clip 6-8" from column	As needed

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11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 Quantitation is based on baseline to baseline integration.

$$\text{Soils}: \frac{mg}{kg} = Ax \frac{m}{As} \frac{1}{Vi} \frac{Vt}{M}$$

$$\text{Water}: \frac{mg}{L} = Ax \frac{m}{As} \frac{1}{Vi} \frac{Vt}{Vo}$$

where:

Ax = Area of component investigated.
m = Mass of standard ~~in~~ (ng).
As = Area response of standard.
Vi = Volume of extract injected (µL).
Vt = Volume of ~~methylene chloride~~ pentane extraction concentrate (mL).
M = Mass of sample extracted (g).
Vo = Volume of sample extracted (mL).

11.1 Practical Quantitation limits are based on common amounts (10 grams to 10 mL, 30 mL to 3.0 mL), usual matrix background, and determined method detection limits.

Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL	
		Water (mg/L)	Soil (mg/kg)
Gasoline, C6 – C10 (peak start- peak end)	TLS	5	50
Diesel, C10 - C28 (peak end – peak end)	TLS	5	50
Total Pet. Hydr., C6 - C28 (peak start- peak end)	TLS	5	50
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Key Type:

T = Compound/analyte is target compound routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike.
L = Compound/analyte is routinely used as a LCS spike.
Q = Compound/analyte is used as a surrogate spike.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

12.1 Refer to the tables below and the following sections for corrective actions.

12.2 The instrument must be able to achieve adequate separation and resolution of peaks and analytes of interest.

12.3 The n-hexane (nC6) peak must be adequately resolved from the n-pentane solvent in the chromatographic run (C6 and C5 must separate).

12.4 Retention time windows must be established for each analyte and/or carbon range of interest each time a new GC column is installed, and verified and/or adjusted on a daily basis by use of the n-alkane locator mix.

12.5 The method blank must not have petroleum hydrocarbons above the practical quantitation limit (PQL).

12.6 The LCS, matrix spike (MS) and matrix spike duplicate (MSD) are each spiked with the gasoline and diesel mixture. If not within the limits of Table 12-3 the problem must be corrected before further samples are analyzed. File a corrective action report. All samples extracted with a method blank or LCS failing to meet criteria must be reextracted. MS and MSD may be determined to have matrix interference.

12.7 A Laboratory Control Sample (LCS, Reference Sample) is to be extracted for every 20 or fewer samples.

12.8 Any data which must be submitted with an out of control event because of hold time considerations or matrix effects or other non-correctable circumstances must be accompanied by a corrective action report.

Routine Quality Control Samples

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Method Blank	1 per 20 or fewer samples	30 mL ASTM Type II water for water sample or 10 g of target analyte free sand for soil sample treated as sample.	Less than quantitation limit.	Reextract and reanalyze samples having concentrations greater than quantitation but less than 5 times blank value.
Laboratory Control Sample (LCS)	1 per 20 or fewer samples	~~50 µL of TX1005 Int.~~ 125 µL of Restek TXTPH MS added to 30 mL of ASTM Type II water for water sample, or ~~100 µL of TX1005 Int.~~ 250 µL Restek TXTPH MS added to 10 g of target analyte-free sand for soil samples.	70-130% recovery	Reextract.
Diesel Mid Calibration Standard (Continuing Calibration Standard CCV)	Daily at start of sample analysis Every 10 samples and at end of sequence	See Table 8-2	Daily ±25%D of average CF (see Section 10.4.1) ±25% of true value	Repeat initial calibration Identify problem. Correct, repeat samples since last acceptable CCV Standard.
Matrix Spike/ Matrix Spike Duplicate	1 set of 20 or fewer samples in a batch	~~50 µL of TX1005 Int.~~ ~~125 µL Restek TXTPH MS~~ 300 µL TX1005 Stock or 750 µL Restek TXTPH MS added to 30 mL of sample, or ~~100 µL of TX1005 Int.~~ 250 µL Restek TXTPH MS added to 10 g sample	70-130% recovery. Compare against LCS limits for bias. 30% RPD	Evaluate results in conjunction with other QC information (e.g., LCS recoveries) to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate. Reextract batch

Revision: 0	Status: Final	Method: TX1005	Minor Revision Date: 3/26/2001

Table 12-2

CONTROL CHARTED QUALITY CONTROL SAMPLES

QC Type	Analytes Charted	Matrix Charted
LCS	Gasoline and diesel spike	Water/Soil

Revision: 0	Status: Final	Method: TX1005	Minor Revision Date: 3/26/2001

Table 12-3

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
LCS	Soil Soil	Gasoline Diesel Fuel	250 mg/kg 250 mg/kg	70-130%	NA	Method
LCS	Water Water	Gasoline Diesel Fuel	~~41.7~~ 250 mg/L ~~41.7~~ 250 mg/L	70-130%	NA	Method
MS/MSD	Soil Soil	Gasoline Diesel Fuel	250 mg/kg 250 mg/kg	70-130%	30%	Method
MS/MSD	Water Water	Gasoline Diesel Fuel	41.7 mg/L 41.7 mg/L	70-130%	30%	Method

Revision: 0	Status: Final	Method: TX1005	Minor Revision Date: 3/26/2001

13.0 SPECIAL PROJECT REQUIREMENTS

13.1 U.S. Army Corps of Engineers (ACE)

13.1.1 Method Quantitation Limit (MQL) is between 3-10 times MDL. The low calibration standard concentration must be ≥3 times MDL. The Method Reporting Limit (MRL) may slide between two times the MDL and project specific or regulatory requirements. Results below the MRL must be reported "ND." Results between the MQL and MRL must be reported as estimated (J flagged).

14.0 SAMPLE DISPOSAL

14.1 See SOP A.10, Waste Disposal.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: PCB Analysis by Method 8082			
Reviewer: R. Neary		Peer Reviewer: K. Willy	
Implementation Date: 6/13/01		Last Annual Review Date: 6/13/01	
Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) describes the procedure for identifying and quantifying polychlorinated biphenyl compounds in surface waters, groundwater, soils, sediments, and oils by a gas chromatographic procedure.

2.0 METHOD SUMMARY

2.1 Extracts of water or soil samples (SOP EXT.4 or EXT.19) or dilutions of oils and wipes for PCB are analyzed on a gas chromatograph equipped with capillary columns and electron capture detectors. The GC is calibrated by injecting 5 levels of the analytes of interest and measuring response. An aliquot of a sample is injected into the GC and quantitative analysis is performed on the resulting chromatogram by comparing sample response to standard response. Qualitative Confirmation is accomplished by simultaneously injecting onto a second column.

2.2 The following deviations from the method are taken.

2.2.1 The target list of compounds usually reported by this method is listed in Table 11-1. This SOP does not cover use of this method for specific PCB congeners.

2.2.2 Minor changes in chromatographic conditions may be used to optimize separation and sensitivity.

2.2.3 DCB is the primary surrogate, TCMX is a secondary surrogate.

2.2.4 Due to the likelihood that one or more surrogates may be interfered with, the following criteria are applied.

2.2.4.1 DCB in Laboratory Control Samples and method blanks must be recovered within in-house limits.

2.2.4.2 DCB recovery will be determined for all samples; if recovery is outside in-house limits, TCMX recoveries will be evaluated and reported.

2.2.4.3 Cases where both DCB and TCMX recoveries fail to meet criteria, and matrix effects are not evident, will require re-extraction and reanalysis of affected samples.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

3.3 Waste containing ≥50 ppm total PCB must be segregated and identified as "PCB Waste"

4.0 REFERENCES

4.1 SW-846 3rd Edition, Update III, June 1997, Methods 8000B, 8082, 3660B, 3665A.

4.2 Training SOP QA.5, A.23, and A.29.

4.3 Data Review SOP A.25.

4.4 Laboratory Quality Assurance Manual (L-001).

4.5 MDL SOP A.18.
4.6 Corrective Action Procedure, QA.1.
4.7 Use checklist C-055 for analyst and peer review.

5.0 DEFINITIONS/ACRONYMS

5.1 **MDL** - Method Detection Limit as determined by 40 CFR Part 136, Appendix B. Method detection limits must be determined yearly for both water and soil matrixes. See SOP A.18 for procedure.
5.2 **PQL** - Practical Quantitation Limit is the concentration above the MDL that can be reasonably obtained and is used as the limit of reporting (Table 11-1).
5.3 **TCMX** - Tetrachlorometaxylene.
5.4 **DCB** – Decachlorobiphenyl.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 **Phthalates** may cause false positives. Care should be exercised to reduce exposure of samples to plastics.
6.2 Elemental Sulfur – if present in the samples, may cause interferences. Sulfur clean-up using copper may be required. Refer to sulfur cleanup in SOP EXT.4 or SOP EXT.19.
6.3 Heavy baseline and organics in samples may be cleaned up using acid-partitioning. Refer to Section 10.5.5.

7.0 INSTRUMENTATION AND EQUIPMENT

Varian 3400 or Hewlett Packard 5890 equipped with electron capture detector and autosampler
PE Nelson data system with TURBO*CHROM Software
Restek RTX-5, 30 m column x 0.53mm 1.0 µm film (or equivalent)
Restek RTX-35, 30 m column x 0.53mm 0.5 µm film (or equivalent)
Volumetric flasks, Fisher or equivalent
Gastight syringes, Krackeler or equivalent
Screwcap vials with Teflon-lined septa, Krackeler or equivalent
Disposable Wiretrol® pipettes, Krackeler, Restek, or equivalent

8.0 REAGENTS AND MATERIALS

8.1 **See Table 8-1, Summary of Standards, Reagents, and Consumables**

Table 8-1

SUMMARY OF STANDARDS AND REAGENTS

Description	Source	Concentration
AR1016/1260 MIX	Restek or equivalent	1000 µg/mL each
AR 1254 MIX	Restek or equivalent	1000 µg/mL
AR 1248 MIX	Restek or equivalent	1000 µg/mL
AR 1242 MIX	Restek or equivalent	1000 µg/mL
AR 1232 MIX	Restek or equivalent	1000 µg/mL

Table 8-1

SUMMARY OF STANDARDS AND REAGENTS

Description	Source	Concentration
AR 1221 MIX	Restek or equivalent	1000 µg/mL
Pesticide Surrogate Mix	Restek or equivalent	200 µg/mL TCMX and 200 µg/mL DCB
Copper 30-40 Mesh	Supelco	Neat

Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

8.2 Standard Solution Preparation

8.2.1 All stock solutions commercially obtained shall be certified. Certificates are to be kept on file for all stocks received. All stocks and standards are to be stored at 2-6 C or lower.

8.2.2 Stock standards expire one year from the date opened, unless an earlier vendor supplied expiration date supercedes.

8.2.3 Working standards expire six months from date prepared unless expiration is superceded by stock expiration.

8.2.4 Unopened stock standards expire or per vendor-specified date; if not provided 1 year from Date of Receipt.

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted To _____	Final Concentration
Pest. Surr. Int.	Pesticide Surrogate Mix	50 µL to 10 mL hexane	10 µg/mL
AR1660L	AR1016/1260 Mix Pest Surr. Int	2.5 µL plus 25 µL to 50 mL hexane	See Table 8-3
AR1660ML	AR1016/1260 Mix Pest Surr. Int	5 µL plus 50 µL to 50 mL hexane	See Table 8-3
AR1660M	AR1016/1260 Mix Pest Surr. Int	10 µL plus 100 µL to 50 mL hexane	See Table 8-3
AR1660 MH	AR1016/1260 Mix Pest Surr. Int	20 µL plus 200 µL to 50 mL hexane	See Table 8-3
AR1660H	AR1016/1260 Mix Pest Surr. Int	50 µL plus 500 µL to 50 mL hexane	See Table 8-3
AR1254L	AR1254 Mix Pest Surr Int	2.5 µL plus 25 µL to 50 mL hexane	See Table 8-4
AR1254ML	AR1254 Mix Pest Surr Int	5 µL plus 50 µL to 50 mL hexane	See Table 8-4
AR1254M	AR1254 Mix Pest Surr Int	10 µL plus 100 µL to 50 mL hexane	See Table 8-4

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted To _____	Final Concentration
AR1254MH	AR1254 Mix Pest Surr Int	20 µL plus 200 µL to 50 mL hexane	See Table 8-4
AR1254H	AR1254 Mix Pest Surr Int	50 µL plus 500 µL to 50 mL hexane	See Table 8-4
AR1221L	AR1221 Mix Pest Surr Int	5 µL plus 25 µL to 50 mL hexane	See Table 8-5
AR1221ML	AR1221 Mix Pest Surr Int	10 µL plus 50 µL to 50 mL hexane	See Table 8-5
AR1221M	AR1221 Mix Pest Surr Int	20 µL plus 100 µL to 50 mL hexane	See Table 8-5
AR1221MH	AR1221 Mix Pest Surr Int	40 µL plus 200 µL to 50 mL hexane	See Table 8-5
AR1221H	AR1221 Mix Pest Surr Int	50 µL plus 500 µL to 50 mL hexane	See Table 8-5
AR1232L	AR1232 Mix Pest Surr Int	2.5 µL plus 25 µL to 50 mL hexane	See Table 8-6
AR1232ML	AR1232 Mix Pest Surr Int	5 µL plus 50 µL to 50 mL hexane	See Table 8-6
AR1232M	AR1232 Mix Pest Surr Int	10 µL plus 100 µL to 50 mL hexane	See Table 8-6
AR1232MH	AR1232 Mix Pest Surr Int	20 µL plus 200 µL to 50 mL hexane	See Table 8-6
AR1232H	AR1232 Mix Pest Surr Int	50 µL plus 500 µL to 50 mL hexane	See Table 8-6
AR1242L	AR1242 Mix Pest Surr Int	2.5 µL plus 25 µL to 50 mL hexane	See Table 8-7
AR1242ML	AR1242 Mix Pest Surr Int	5 µL plus 50 µL to 50 mL hexane	See Table 8-7
AR1242M	AR1242 Mix Pest Surr Int	10 µL plus 100 µL to 50 mL hexane	See Table 8-7
AR1242MH	AR1242 Mix Pest Surr Int	20 µL plus 200 µL to 50 mL hexane	See Table 8-7
AR1242H	AR1242 Mix Pest Surr Int	50 µL plus 500 µL to 50 mL hexane	See Table 8-7
AR1248L	AR1248 Mix Pest Surr Int	2.5 µL plus 25 µL to 50 mL hexane	See Table 8-8
AR1248ML	AR1248 Mix Pest Surr Int	5 µL plus 50 µL to 50 mL hexane	See Table 8-8
AR1248M	AR1248 Mix Pest Surr Int	10 µL plus 100 µL to 50 mL hexane	See Table 8-8

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted To _____	Final Concentration
AR1248MH	AR1248 Mix Pest Surr Int	20 µL plus 200 µL to 50 mL hexane	See Table 8-8
AR1248H	AR1248 Mix Pest Surr Int	50 µL plus 500 µL to 50 mL hexane	See Table 8-8

Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

Table 8-3

COMPONENTS AND CALIBRATION CONCENTRATIONS OF AR 1660

Compound	µg/mL				
	AR1660L	AR1660ML	AR1660M	AR1660MH	AR1660H
TCMX	0.005	0.010	0.020	0.040	0.100
AR1016	0.050	0.100	0.200	0.400	1.000
AR1260	0.050	0.100	0.200	0.400	1.000
DCB	0.005	0.010	0.020	0.040	0.100

Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

Table 8-4

AR1254 CALIBRATION LEVELS

Compound	µg/mL				
	AR1254L	AR1254ML	AR1254M	AR1254MH	AR1254H
TCMX	0.005	0.010	0.020	0.040	0.100
AR1254	0.050	0.100	0.200	0.400	1.000
DCB	0.005	0.010	0.020	0.040	0.100

Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

Table 8-5

FINAL CONCENTRATIONS (µg/mL) OF AR1221 – 5-POINT CALIBRATON

Compound	Level				
	Low	Med. Low	Med	Med High	High
TCMX	0.005	0.010	0.020	0.040	0.100
AR1221	0.100	0.200	0.400	0.800	1.000
DCB	0.005	0.010	0.020	0.040	0.100

Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

Table 8-6

FINAL CONCENTRATIONS (µg/mL) OF AR1232 - 5 POINT CALIBRATION

Compound	Level				
	Low	Med. Low	Med	Med High	High
TCMX	0.005	0.010	0.020	0.040	0.100
AR1232	0.050	0.100	0.200	0.400	1.000
DCB	0.005	0.010	0.020	0.040	0.100

Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

Table 8-7

FINAL CONCENTRATIONS (µg/mL) OF AR1242 - 5 POINT CALIBRATION

Compound	Level				
	Low	Med. Low	Med	Med High	High
TCMX	0.005	0.010	0.020	0.040	0.100
AR1242	0.050	0.100	0.200	0.400	1.000
DCB	0.005	0.010	0.020	0.040	0.100

Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

Table 8-8

FINAL CONCENTRATIONS (µg/mL) AR1248 - 5 POINT CALIBRATION

Compound	Level				
	Low	Med. Low	Med	Med High	High
TCMX	0.005	0.010	0.020	0.040	0.100
AR1248	0.050	0.100	0.200	0.400	1.000
DCB	0.005	0.010	0.020	0.040	0.100

Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Water or Solid	Standard	Seven for water; 14 for soil.	The PCB extracts from sample preparation are to be analyzed within 40 days from extraction date.	Extracts are stored in a secure area in a separate refrigerator from original samples at 2-6 C.
Water or Solid	NYSDEC	Five for water and soils from verified time of sample receipt.	The PCB extracts from sample preparation are to be analyzed within 40 days from extraction date.	Extracts are stored in a secure area in a separate refrigerator from original samples at 2-6 C.

Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

10.0 PROCEDURE

10.1 Apparatus Setup

10.1.1 The GC is equipped with two columns connected by a "Y" connector to one injection point and 2 electron capture detectors.

10.1.2 The carrier gas flow and temperature profile may vary somewhat to optimize analytical separation.

10.1.3 Recommended temperatures are set as follows: Injector = 200 C; Detector = 300 C Temperature program: Column Initial = 150 C. Initial Hold = 1.0 min. Column Rate = 5 C/min. Column Final = 280 C. Final Hold = minimum of 10 minutes and may be extended due to presence of late-eluting compounds in extracted samples.

10.1.4 The autosampler injects 1.0 µL of sample extract into each GC column. (i.e., 2.0 µL total injection).

10.1.5 Transfer of extracts to instrument autosampler vials shall be accomplished with disposable pipettes or graduated Wiretrol® micropipettes.

10.2 Establishing Retention Time Windows

10.2.1 Make three injections of mid level Aroclor 1016/1260 standard over 72-hour period.

10.2.2 Calculate the multicomponent retention time windows based on five peaks of the Aroclor 1016/1260 mixture. The windows will apply to all multicomponents. Pattern recognition is still the primary means of identification.

10.2.3 The retention time window is defined as plus or minus 3 times the standard deviation from the retention time of the initial mid-level standards in the continuing calibration sequence. However, analyst experience should weigh heavily. Pattern recognition should primarily be used for multi-response products.

10.2.4 Retain this data so that it can be easily retrieved.

10.2.5 To facilitate analyte identification, the retention time windows are set as percentages (±% realtime of daily calibration) into the data system. These values are slightly wider than calculated windows.

10.3 Initial Calibration

10.3.1 Compound list and Quantitation Limits (Table 11.1).

10.3.2 Analyze an instrument blank (hexane) to verify the analytical system is clean (no target compounds are present greater than the quantitation limit) before Standards.

10.3.3 The initial calibration of PCBs is performed at the five levels identified with suffix L, ML, M, MH, and H (see Tables 8.3 - 8.8.). See Table 10-1 for specific calibrations.

10.3.4 Initial calibration for PCBs only require five-point calibration for AR1660. Should any additional PCB be detected, or suspected, calibration must be verified for that Aroclor. If not verified, new initial calibration must be done for that Aroclor.

10.3.5 Note: Hexane = Instrument blank.

Table 10-1

INITIAL CALIBRATION SEQUENCE
HEXANE
AR1660L
AR1660ML
AR1660M
AR1660MH
AR1660H
AR1660 Reference (2nd source) (ICV)

See Section 10.3.4, Following 1660 calibration, the instrument is recalibrated for any additional suspected or identified PCBs.

10.3.6 Inject 1.0 µL per column (i.e., 2 µL total injection) of each calibration standard. Tabulate area responses against the mass injected. The results are used to prepare a calibration curve for each analyte.

10.3.7 Calculate the ratio of the response to the amount injected, defined as the calibration factor (CF), for each analyte at each standard concentration.

$$\text{Calibration Factor (CF)} = \frac{\text{Total Area of Peaks *}}{\text{Mass Injected (in nanograms)}}$$

**Use the total area of the 5 largest peaks for quantitation. The number and specific peaks may need to be adjusted, depending if sample matrix interferences or pattern weathering exists (see Section 11.2).*

10.3.8 Calculate the mean calibration factor for all Aroclors and surrogates. Calculate the % Relative Standard Deviation (%RSD) of the CF_x values for the initial calibration using the following equations:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}_x\right)^2}{n-1}} \qquad \%RSD = \frac{SD}{\overline{CF}_x} \cdot 100$$

where:

SD = Standard derivation using sample population (n-1).
CF_x = Mean CF for each Aroclor "x."
CF_i = Calibration factor for each calibration level "i" for the Aroclor "x" of interest.
n = Number of calibration levels.

10.3.9 If the % RSD of any Aroclors is 20% or less, linearity through the origin is assumed and the calibration factor is assumed to be constant over the calibration range, and the mean calibration factor may be used for quantitation.
10.3.10 If any analytes are greater than the 20% RSD, the initial calibration is valid as long as no one analyte is greater than 40% RSD and the mean of the RSD values for all analytes in the calibration is less than 20% (Grand Mean approach). Note: The analyst and the data user must be aware that the use of the approach listed in this Section (i.e., the average of all RSD values ≤ 20%) will lead to greater uncertainty of those analytes for which the RSD is greater than 20%. The analyst and the data user should review the associated quality control results carefully, with particular attention to the matrix spike and laboratory control sample results to determine if the calibration linearity poses a significant concern. If this approach is not acceptable for a particular application, then the analyst may need to adjust the instrument operating conditions and/or the calibration range until the RSD is ≤ 20%, or apply a regression analysis.
10.3.10.1 The mean RSD criterion applies to all analytes in the standards, regardless of whether or not they are of interest for a specific project.
10.3.10.2 The data user must be provided with either a summary of the initial calibration data or a specific list of those compounds for which the RSD exceeded 20% and the results of the mean RSD calculation.
10.3.11 If the RSD of the calibration or response factors is greater than 20% over the calibration range, then linearity through the origin cannot be assumed. The analyst may then utilize a linear regression equation that is not forced through the origin.
10.3.12 Linear regression can also be used for analytes that do meet the RSD limits. Calibration is valid when the regression calculation generates a correlation coefficient for each analyte of interest greater than or equal to 0.995.
10.3.13 If the linear correlation coefficient is <0.995, a second order curve may be used if at least 6 calibration standards are used. If the coefficient of determination of the second order curve is ≥0.990, use this second order curve for quantitation of the analyte of interest. Additional concentration levels of standards **are required** for higher order curves. Refer to Method 8000 for guidance. Do not apply higher order curves without consulting the supervisor.
10.3.14 If initial calibration requirements mentioned in the above sections cannot be met, then calibration is not valid and, therefore, the analyst should check instrument operation conditions and recalibrate.
10.3.15 Analyze a reference standard from a second source (ICV) immediately following an initial calibration to verify the curve. The curve is deemed acceptable provided each analyte in the reference standard calibration is within 15% difference of the mean calibration factor or within 15% of the theoretical concentration, depending on whether quantitation will be by mean calibration factor or from regression analysis, respectively. The Grand Mean Approach may be used for the ICV except where instructed otherwise for specific clients. See Section 13.
10.3.16 Quantitation of samples must continue to be done using the procedure initially used.
10.3.17 When PCBs other than 1016 or 1260 are unexpectedly found at a given site, standard curves for those analytes must be run and checked against samples for pattern recognition. If positively identified, calibration must be established before samples can be quantified.

10.4 Daily Calibration and Continuing Calibration Verification

10.4.1 The working calibration curve or calibration factor must be verified at the beginning of the analytical sequence, and if a lapse in time >12 hours, has occurred from last continuing standard.

10.4.2 An instrument blank (hexane) is run to verify that the analytical system is clean (no target compounds are present > PQL).

10.4.3 A calibration check standard equivalent to the mid-level calibration standard must be analyzed at the beginning of the analytical sequence and after no more than every 20 samples. Use AR1016/1260 mid-level (AR1660M).

10.4.4 Continuing calibration and daily calibration verifications must be judged against the quantitation method used at the start.

10.4.5 Calculate % Drift and % Difference using the equations below. When using a curve:

$$\%\ \text{Drift} = \frac{\text{Calculated Conc.} - \text{Theoretical Conc.}}{\text{Theoretical Conc.}} \cdot 100$$

When using mean calibration factor:

$$\%\ \text{Difference} = \frac{\text{CF Verification Standard} - \overline{CF_x}}{\overline{CF_x}} \cdot 100$$

10.4.6 If the mean calibration factor is used for quantitation, then the calibration factor of the analyte must be ≤ 15% difference from the mean calibration factor.

10.4.7 If the regression curve is used for quantitating analytes in samples, then the concentration of the analyte in the calibration check standard must be ≤ 15% drift from the theoretical concentration (i.e., 85-115% recovery).

10.4.8 If the percentage difference or drift is greater than 15% for one or more analytes, the standard is acceptable provided no one analyte %D exceeds 30% and the mean of all analytes' %D in the standard is ≤15% difference or drift (Grand Mean approach). However, the discussion in Section 10.3.10 applies.

10.4.9 Instrument blanks (hexane) are allowed before CCV to minimize chances for carryover into the CCV to avoid invalidation of the curve due to random carryover. Indication of carryover from one sample to another requires reanalysis of the potentially contaminated sample only.

10.5 Calibration Verification Corrective Action

10.5.1 If initial or continuing calibration criteria is not met, stop sample analysis immediately. Inspect the system. Minor corrective action may be performed such as changing the septum or injection port liner.

10.5.2 If after the minor corrective action a compliant CCV is analyzed, sample analysis may continue. Any samples not bracketed by valid CCVs must be reanalyzed and bracketed by compliant CCVs.

10.5.3 If minor corrective action does not fix the problem, major corrective action may be taken such as cleaning the injection port, clipping the column ends, baking out the detectors, or installing new columns. If major corrective action is taken, a new calibration curve must be run and all associated samples reanalyzed.

10.6 Sample Analysis

10.6.1 Once a successful initial calibration is complete, the primary analytical daily or batch sequence may begin (See Table 10-2).

Table 10-2 Daily Calibration Sequence
Instrument Blank
Aroclor 1660 M
Alternate PCBs as needed.
Samples, not to exceed 20, 10 recommended.
Aroclor 1660 M.
Alternate PCBs as needed.
Samples, not to exceed 20, 10 is recommended.
Repeat

10.6.2 After every 20 samples or less, an Aroclor 1660 mid-level standard is analyzed. If any PCB is detected in a sample at a level that exceeds the linear range of the calibration curve, the sample must be diluted to within the calibrated range.
10.6.2.1 Instrument blanks (hexane) may be interspersed throughout the analytical run to check for carryover or when the system is run unattended (autosampler).
10.6.3 Additional instrument blanks may be run after suspect samples or before standards to help eliminate matrix carry over.
10.6.4 Peak detection is prevented by the presence of elemental sulfur. Samples are copper treated prior to analysis; however, further treatment may be required for those samples that contain larger amounts of sulfur.
10.6.5 If samples are suspected to contain matrix interference (samples are colored) or after initial anlaysis samples have matrix interference, samples must under go sulfuric acid cleanup. This procedure is as follows.
10.6.6 Sulfuric Acid Cleanup
10.6.6.1 Using a disposable pipette, transfer 1 mL of the hexane extract to a 2-mL vial and, in a fume hood, carefully add 0.5 mL of the concentrated sulfuric acid. **Caution: Make sure that there is no exothermic reaction nor evolution of gas prior to proceeding.**
10.6.6.2 Cap the vial tightly and vortex for 30 seconds. Caution: Stop the vortexing immediately if the vial leaks. AVOID SKIN CONTACT, SULFURIC ACID BURNS.
10.6.6.3 Allow the phases to separate for at least 1 minute. Examine the top (hexane) layer, it should not be highly colored nor should it have a visible emulsion or cloudiness.
10.6.6.4 If a clean phase separation is achieved, proceed to Section 10.6.6.7.
10.6.6.5 If the hexane layer is colored or the emulsion persists for several minutes, remove the hexane layer from the vial and place in a 2.0-mL vial. Add another 0.5-mL portion of the clean sulfuric acid and perform another acid cleanup.
10.6.6.6 Vortex the sample for one minute and allow the phases to separate.
10.6.6.7 Analyze the hexane layer only.

10.7 Confirmation Analysis
10.7.1 Confirmation is not required by Method 8082.
10.7.2 If required by the client, a PCB is confirmed as present if the recognizable pattern is present on the confirmation column.
10.7.3 The confirmation analysis is qualitative only.

Instrument Maintenance

Table 10-3

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
V3400/HP5890	Retention time shift	Change septum	Daily (or as needed)
V3400/HP5890	High baseline	Bake out column, detector	As needed
V3400/HP5890	Non-linear calibration	Replace detector; replace column	As needed
Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 If quantitating from a linear curve, the equation below is provided as a manual check.

$$\text{Concentration} = \text{mg/kg}, \mu\text{g/L} = \left[\frac{y-b}{m}\right]\frac{Vt}{S}\cdot Df$$

where:

y = Area of target analyte from initial curve,
b = Intercept (area),
m = Slope (area /[µg/mL]),
S = Mass of soil sample extracted in grams (g) or (volume of water sample extracted in liters L),
Vt = Final volume of the extract in milliliters (mL).
Df = Dilution factor. The dilution factor equals µL of extract used to make dilution + µL clean solvent used to make dilution divided by µL of extract used to make dilution.

11.2 Due to weathering effects or matrix interference, it is not always possible to quantitate samples using the same 5 peaks used in calibrating. All samples must be quantitated using a minimum of 3 peaks for each PCB and these same peaks from the calibration must be used to calculate concentrations. In cases where there are multiple PCBs detected, fewer peaks may be used. Peaks used for quantitation must be clearly identified on chromatograms of standards and samples.

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL	
		Soil (µg/kg)	Water (µg/L)
Aroclor-1016	T,L,S	20	0.50
Aroclor-1221	T	40	1.0
Aroclor-1232	T	20	0.50
Aroclor-1242	T	20	0.50
Aroclor-1248	T	20	0.50
Aroclor-1254	T	20	0.50
Aroclor-1260	T,L,S	20	0.50
TCMX	Q	NA	NA
DCB	Q	NA	NA
Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

Key Type:

NA = Not applicable.
T = Compound/analyte is target compound routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike (MS).
L = Compound/analyte is routinely used as a LCS spike (LCS).
Q = Compound/analyte is used as a surrogate spike (SUR).

Table 11-1.1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS (WIPES)

Compound/Analyte	Type	PQL
		µg /Wipe
Aroclor-1016	T,L,S	1.5
Aroclor-1221	T	3.0
Aroclor-1232	T	1.5
Aroclor-1242	T	1.5
Aroclor-1248	T	1.5
Aroclor-1254	T	1.5
Aroclor-1260	T,L,S	1.5
TCMX	Q	NA
DCB	Q	NA
Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

Key Type:

T = Compound/analyte is target compound routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike.
L = Compound/analyte is routinely used as a LCS spike.
Q = Compound/analyte is used as a surrogate spike.

Table 11-1.2

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL Oil µg /g
Aroclor-1016	T,L,S	5
Aroclor-1221	T	10
Aroclor-1232	T	5
Aroclor-1242	T	5
Aroclor-1248	T	5
Aroclor-1254	T	5
Aroclor-1260	T,L,S	5
TCMX	Q	NA
DCB	Q	NA

Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

Key Type:

NA = Not applicable.
T = Compound/analyte is target compound routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike (MS).
L = Compound/analyte is routinely used as a LCS spike (LCS).
Q = Compound/analyte is used as a surrogate spike (SUR).

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

12.1 Refer to the Quality Control Table below and the following sections for corrective actions.

12.2 Each sample is spiked with the surrogates DCB and TCMX. The DCB is the primary surrogate. Calculate its recovery. If peaks interfere with DCB, TCMX should be evaluated for acceptance. Proceed with corrective action when two surrogates are out of limits for a sample.

12.3 The sample is to be reextracted and reanalyzed unless it can be documented (chromatogram) that factors such as matrix effects are responsible. A corrective action report must be filed.

12.4 If any quality control criteria cannot be met, but the problem is correctable, the sample(s) affected must be reanalyzed.

12.5 Data that must be submitted with out of control events must be accompanied by a corrective action report.

12.6 If any criteria cannot be met, but the problem is correctable, the sample(s) affected must be reanalyzed.

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Acceptance Criteria	Corrective Action
Method Blank	1 per preparation batch of 20 or fewer samples	PQL	Reanalyze samples having concentrations > Quantitation limits.
Matrix Spike (MS) Matrix Spike Duplicate (MSD)	1 per preparation batch of 20 or fewer samples	- Project requirements if specified. - Water 20% RPD - Soil 35% RPD	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Laboratory Control Sample (LCS)	1 per extraction batch of 20 or fewer samples	Laboratory limits	Notify Project Manager immediately. Reextract and reanalyze the entire batch.
LCS Duplicate	Client Specified	Client specified	Client specified
Surrogate (SUR)	Every sample, blanks and QC	Laboratory Control Limit. DCB must meet acceptance limits for method blanks and LCS samples. At least 1 surrogate must meet criteria in samples.	Reextract batch for blank or LCS failure. Reextract affected sample(s) unless matrix effect is evident from chromatogram.

Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

Table 12-2

CONTROL CHARTED QUALITY CONTROL SAMPLES

QC Type		Analytes Charted	Matrix Charted
MS/MSD		Per client	Soil, Water, Oil, Wipes
LCS		Analytes per customer request	Soil, Water, Oil, Wipes
Surrogate (SUR)		DCB	Soil, Water, Oil, Wipes
Secondary Surrogate (SUR)		TCMX	Soil, Water, Oil, Wipes
Revision: 1	Status: Final	Method: 8082	Minor Revision Date: None

13.0 SPECIAL PROJECT REQUIREMENTS

13.1 AFCEE

13.1.1 % Difference or drift may not be averaged.

13.2 U.S. Army Corps of Engineers (ACE)

13.2.1 Method Quantitation Limit (MQL) is between 3-10 times MDL. The low calibration standard concentration must be ≥3 times MDL. The Method Reporting Limit (MRL) may slide between two times the MDL and project-specific or regulatory requirements. The MRL may be higher or lower than the MQL. Results below the MQL must be reported as estimated (J flagged).

13.3 North Carolina DENR

13.3.1 If quantitating from a curve, a zero standard must be incorporated as well as five non-zero standards.

13.3.2 Alternate use of a midconcentration range continuing verification standard with a lower standard every 12 hours. The standard AR1660M is NOT at midconcentration of the calibration range.

13.3.3 Two MS/MSD pairs must be prepared and analyzed every 10 samples.

14.0 SAMPLE DISPOSAL

14.1 See waste disposal SOP A.10.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Pesticide Analysis by Method 8081A			
Reviewer: R. Neary		Peer Reviewer: L. Gutierrez	
Implementation Date: 11/08/00		Last Annual Review Date: 11/03/00	
Revision: 2	Status: Final	Method: 8081A	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) describes the procedure for identifying and quantifying organochlorine pesticides in surface waters, groundwater, soils, TCLP extracts, and sediments by a gas chromatographic procedure. Although not part of the method, it can be used as a screening tool for the presence of PCBs.

2.0 METHOD SUMMARY

2.1 Extracts of water, soil samples, or TCLP extracts (SOP EXT.4, EXT.19, EXT.58) for pesticides are analyzed on a gas chromatograph equipped with capillary columns and electron capture detectors. The GC is calibrated by injecting 5 levels of the analytes of interest and measuring response. An aliquot of a sample is injected into the GC and quantitative analysis is made by comparing sample response to standard response. Qualitative Confirmation is accomplished by simultaneously injecting onto a second column.

2.2 The following deviations from the method are taken.

2.2.1 The target list of compounds usually reported by this method is denoted with a "T" in Table 11-1. The list can be expanded to include other pesticides analyzable by this method (see Sections 1.9, 1.10 of Method 8081A).

2.2.2 Minor changes in chromatographic conditions may be used to optimize separation and sensitivity.

2.2.3 Due to the likelihood that one or more surrogates may be interfered with, the following criteria are applied.

2.2.3.1 All surrogates in Laboratory Control Samples and method blanks must be recovered within in-house limits.

2.2.3.2 Surrogates in each sample must be recovered within limits, unless matrix interferences are evident in the chromatogram.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E Inc., Analytical Services Center Chemical Hygiene Plan, located in the QA Library Island shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Methods 8000B, 8081A, (Rev 1, 12/96), SW-846 3rd Edition, Update III, June 1997.
4.2 Training SOP QA.5, A.23, and A.29.
4.3 Data Review SOP A.25.
4.4 Laboratory Quality Assurance Manual.
4.5 MDL SOP A.18.
4.6 Corrective Action Procedure, QA.1.
4.7 Checklist C-081 for review.

5.0 DEFINITIONS/ACRONYMS

5.1 MDL – Method Detection Limit as determined by 40 CFR Part 136, Appendix B. Method detection limits must be determined yearly for both water and soil matrixes. See SOP A.18 for procedure.

5.2 PQL – Practical Quantitation Limit is the concentration above the MDL that can be reasonably obtained and is used as the limit of reporting.

5.3 TCLP – Toxicity Characteristic Leaching Procedure.

5.4 TCL – Target Compound List.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Phthalates may cause false positives. Confirmation column analysis should eliminate this interference.

6.2 Elemental Sulfur – Present in the samples, may cause interferences. Sulfur clean-up using copper may be required. Refer to the procedure outlined in Section 10.6.5.

7.0 INSTRUMENTATION AND EQUIPMENT

Varian 3400 or Hewlett Packard 5890, 6890 equipped with electron capture detector and autosampler
PE Nelson data system with TURBO*CHROM Software
Restek RTX-5, 30 m column x 0.53 mm 1.0 µm film (or equivalent)
Restek RTX-35, 30 m column x 0.53 mm 0.5 µm film (or equivalent)
Volumetric flasks, Fisher or equivalent
Gastight syringes, Krackeler or equivalent
Screwcap vials with Teflon®-lined septa, Krackeler or equivalent
Disposable Wiretrol® pipettes, Krackeler, Restek, or equivalent

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Reagents and Consumables

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Component	Concentration (µg/mL)
Pesticide Mix A	Supelco or equivalent	alpha-BHC	5
		gamma-BHC	5
		Heptachlor	5
		Endosulfan I	5
		Dieldrin	10
		Endrin	10
		4,4'-DDD	10
		4,4'-DDT	10
		Methoxychlor	50

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Component	Concentration (µg/mL)
Pesticide Mix B	Supelco or equivalent	beta-BHC	5
		delta-BHC	5
		Aldrin	5
		Heptachlor epoxide	5
		gamma Chlordane	5
		alpha Chlordane	5
		4,4'-DDE	10
		Endosulfan II	10
		Endrin aldehyde	10
		Endosulfan sulfate	10
		Endrin ketone	10
Pesticide Reference Mix A	Supelco or equivalent	alpha-BHC	8.0
		gamma-BHC	8.0
		4,4'-DDD	16
		4,4'-DDT	16
		DCB	16
		Dieldrin	16
		Endosulfan I	8.0
		Endrin	16
		Heptachlor	8.0
		Methoxychlor	80
		TCMX	8.0
Pesticide Reference Mix B	Supelco or equivalent	Aldrin	8.0
		beta-BHC	8.0
		delta-BHC	8.0
		alpha-Chlordane	8.0
		gamma-Chlordane	8.0
		4,4'-DDE	16
		Endosulfan II	16
		Endosulfan sulfate	16
		Endrin aldehyde	16
		Endrin ketone	16
		Heptachlor epoxide	8.0
		TCMX	8.0
		DCB	16
Pesticide Surrogate Mix	Restek or equivalent	TCMX and DCB	200 µg/mL (TCMX and DCB)

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Component	Concentration (µg/mL)
Technical Chlordane Calibration	Restek or equivalent	Technical chlordane	1000 µg/mL
Technical Chlordane Reference Stock	Supelco or equivalent	Technical chlordane	5000 µg/mL
Toxaphene Calibration	Restek or equivalent	Toxaphene	1000 µg/mL
Toxaphene Reference Stock	Supelco or equivalent	Toxaphene	5000 µg/mL
Endrin/4'4-DDT Mix	Supelco or equivalent	Endrin/DDT	500 µg/mL each
Copper, 30-40 mesh	Supelco		Metal
Revision: 2	Status: Final	Method: 8081A	Minor Revision Date: None

8.2 Standard Solution Preparation

8.2.1 All stock solutions commercially obtained shall be certified. The single-response pesticide stock standards are purchased as mixes of several compounds per mix. Certificates are to be kept on file for all stocks received. All stocks and standards are to be stored at 6 C or lower.

8.2.2 Stock standards expire one year from the date opened, unless an earlier vendor-supplied expiration date supercedes.

8.2.3 Working standards expire six months from date prepared unless expiration is superceded by stock expiration.

8.2.4 Unopened stock standards expire by vendor-specified date; if not provided one year from date of receipt.

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted To _____	Final Concentration
Pesticide Surrogate Intermediate	Pesticide Surrogate Mix	500 µL to 10 mL hexane	10 µg/mL
INDA L (INDA 1)	Pesticide Mix A Pesticide Surrogate Intermediate	25 µL plus 25 µL to 50 mL hexane	See Table 8-3
INDA ML (INDA 2)	Pesticide Mix A Pesticide Surrogate Intermediate	50 µL plus 50 µL to 50 mL hexane	See Table 8-3
INDA M (INDA 3)	Pesticide Mix A Pesticide Surrogate Intermediate	200 µL plus 200 µL to 100 mL hexane	See Table 8-3
INDA MH (INDA 4)	Pesticide Mix A Pesticide Surrogate Intermediate	100 µL plus 100 µL to 25 mL hexane	See Table 8-3

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted To _____	Final Concentration
INDA H (INDA 5)	Pesticide Mix A Pesticide Surrogate Intermediate	250 µL plus 250 µL to 25 mL hexane	See Table 8-3
INDB L (INDB 1)	Pesticide Mix B Pesticide Surrogate Intermediate	25 µL plus 25 µL to 50 mL hexane	See Table 8-4
INDB ML (INDB 2)	Pesticide Mix B Pesticide Surrogate Intermediate	50 µL plus 50 µL to 50 mL hexane	See Table 8-4
INDB M (INDB 3)	Pesticide Mix B Pesticide Surrogate Intermediate	200 µL plus 200 µL to 100 mL hexane	See Table 8-4
INDB MH (INDB 4)	Pesticide Mix B Pesticide Surrogate Intermediate	100 µL plus 100 µL to 25 mL hexane	See Table 8-4
INDB H (INDB 5)	Pesticide Mix B Pesticide Surrogate Intermediate	250 µL plus 250 µL to 25 mL hexane	See Table 8-4
Toxaphene Low (TOX 1)	Toxaphene Pesticide Surrogate Intermediate	5 µL plus 25 µL to 50 mL hexane	See Table 8-5
Toxaphene Med/Low (TOX 2)	Toxaphene Pesticide Surrogate Intermediate	10 µL plus 50 µL to 50 mL hexane	See Table 8-5
Toxaphene Med (TOX 3)	Toxaphene Pesticide Surrogate Intermediate	20 µL plus 100 µL to 50 mL hexane	See Table 8-5
Toxaphene Med/High (TOX 4)	Toxaphene Pesticide Surrogate Intermediate	25 µL plus 200 µL to 50 mL hexane	See Table 8-5
Toxaphene High (TOX 5)	Toxaphene Pesticide Surrogate Intermediate	50 µL plus 500 µL to 50 mL hexane	See Table 8-5
Technical Chlordane, Intermediate	Technical Chlordane, Intermediate	500 µL to 5 mL hexane	100 µg/mL
Chlordane Low (CLR 1)	Technical Chlordane, Intermediate Pesticide Surrogate Intermediate	10 µL plus 25 µL to 50 mL hexane	See Table 8-6
Chlordane Med/Low (CLR 2)	Technical Chlordane, Intermediate Pesticide Surrogate Intermediate	25 µL plus 50 µL to 50 mL hexane	See Table 8-6

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted To _____	Final Concentration
Chlordane Med (CLR 3)	Technical Chlordane, Intermediate Pesticide Surrogate Intermediate	50 µL plus 100 µL to 50 mL hexane	See Table 8-6
Chlordane Med/High (CLR 4)	Technical Chlordane, Intermediate Pesticide Surrogate Intermediate	100 µL plus 200 µL to 50 mL hexane	See Table 8-6
Chlordane High (CLR 5)	Technical Chlordane, Intermediate Pesticide Surrogate Intermediate	250 µL plus 500 µL to 50 mL hexane	See Table 8-6
Pesticide Reference A	Restek Pesticide Mix A	31.25 µL to 50 mL hexane	See Table 8-7
Pesticide Reference B	Restek Pesticide Mix B	31.25 µL to 50 mL hexane	See Table 8-8
Technical Chlordane Reference Intermediate	Technical Chlordane Reference Stock	100 µL of Technical Chlordane Reference Stock into 50 mL of hexane	10 µg/mL
Technical Chlordane Reference	Technical Chlordane Reference Intermediate Stock	100 µL of Technical Chlordane Reference Intermediate Stock into 10 mL of hexane	0.10 µg/mL
Toxaphene Reference	Toxaphene Reference Stock	4 µL of Toxaphene Reference Stock into 50 mL of hexane	0.400 µg/mL
Eval	Endrin/DDT Mix Pesticide Surrogate Intermediate	10 µL plus 200 µL to 100 mL hexane	See Table 8-9

Revision: 2	Status: Final	Method: 8081A	Minor Revision Date: None

Table 8-3

COMPONENTS AND CALIBRATION CONCENTRATIONS OF PESTICIDE INDA

	(µg/mL)				
Compound	**INDA L**	**INDA ML**	**INDA M**	**INDA MH**	**INDA H**
alpha-BHC	0.0025	0.0050	0.0100	0.0200	0.0500
gamma-BHC	0.0025	0.0050	0.0100	0.0200	0.0500
Heptachlor	0.0025	0.0050	0.0100	0.0200	0.0500
Endosulfan I	0.0025	0.0050	0.0100	0.0200	0.0500
Dieldrin	0.0050	0.0100	0.0200	0.0400	0.1000
Endrin	0.0050	0.0100	0.0200	0.0400	0.1000
4,4'-DDD	0.0050	0.0100	0.0200	0.0400	0.1000

Table 8-3

COMPONENTS AND CALIBRATION CONCENTRATIONS OF PESTICIDE INDA

Compound	(µg/mL)				
	INDA L	INDA ML	INDA M	INDA MH	INDA H
4,4'-DDT	0.0050	0.0100	0.0200	0.0400	0.1000
Methoxychlor	0.0250	0.0500	0.1000	0.2000	0.5000
TCMX	0.0050	0.0100	0.0200	0.0400	0.1000
DCB	0.0050	0.0100	0.0200	0.0400	0.1000

Revision: 2 | Status: Final | Method: 8081A | Minor Revision Date: None

Table 8-4

COMPONENTS AND CALIBRATION CONCENTRATIONS OF PESTICIDE INDB

Compound	µg/mL				
	INDB L	INDB ML	INDB M	INDB MH	INDB H
beta-BHC	0.0025	0.0050	0.0100	0.0200	0.0500
delta-BHC	0.0025	0.0050	0.0100	0.0200	0.0500
Aldrin	0.0025	0.0050	0.0100	0.0200	0.0500
Heptachlor epoxide	0.0025	0.0050	0.0100	0.0200	0.0500
gamma Chlordane	0.0025	0.0050	0.0100	0.0200	0.0500
alpha Chlordane	0.0025	0.0050	0.0100	0.0200	0.0500
4,4'-DDE	0.0050	0.0100	0.0200	0.0400	0.1000
Endosulfan II	0.0050	0.0100	0.0200	0.0400	0.1000
Endrin aldehyde	0.0050	0.0100	0.0200	0.0400	0.1000
Endosulfan sulfate	0.0050	0.0100	0.0200	0.0400	0.1000
Endrin ketone	0.0050	0.0100	0.0200	0.0400	0.1000
TCMX	0.0050	0.0100	0.0200	0.0400	0.1000
DCB	0.0050	0.0100	0.0200	0.0400	0.1000

Revision: 2 | Status: Final | Method: 8081A | Minor Revision Date: None

Table 8-5

FINAL CONCENTRATIONS OF TOXAPHENE - 5-POINT CALIBRATION LEVEL
(µg/mL)

Compound	Low	Med. Low	Med	Med High	High
Toxaphene	0.100	0.200	0.400	0.500	1.000
TCMX	0.005	0.010	0.020	0.040	0.100
DCB	0.005	0.010	0.020	0.040	0.100

Revision: 2 | Status: Final | Method: 8081A | Minor Revision Date: None

Table 8-6

FINAL CONCENTRATIONS OF CHLORDANE - 5-POINT CALIBRATION LEVEL (µg/mL)

Compound	Low	Med. Low	Med	Med High	High
Chlordane, Technical	0.020	0.050	0.100	0.200	0.500
TCMX	0.005	0.010	0.020	0.040	0.100
DCB	0.005	0.010	0.020	0.040	0.100

Revision: 2	Status: Final	Method: 8081A	Minor Revision Date: None

Table 8-7

FINAL CONCENTRATIONS OF PESTICIDE REFERENCE MIX A (µg/mL)

Compound	Concentration
Alpha-BHC	0.005
Gamma-BHC	0.005
Heptachlor	0.005
Endosulfan I	0.005
Dieldrin	0.01
Endrin	0.01
4,4'-DDD	0.01
4,4'-DDT	0.01
Methoxychlor	0.05
DCB	0.01
TCMX	0.005

Revision: 2	Status: Final	Method: 8081A	Minor Revision Date: None

Table 8-8

FINAL CONCENTRATIONS OF PESTICIDE REFERENCE MIX B (µg/mL)

Compound	Concentration
alpha-Chlordane	0.005
gamma-Chlordane	0.005
Beta-BHC	0.005
Delta-BHC	0.005
Aldrin	0.005
Heptachlor epoxide	0.005
4,4'-DDE	0.01
Endosulfan II	0.01
Endrin aldehyde	0.01

Table 8-8

FINAL CONCENTRATIONS OF PESTICIDE REFERENCE MIX B
(µg/mL)

Compound	Concentration
Endosulfan sulfate	0.01
Endrin ketone	0.01
DCB	0.01
TCMX	0.005

Revision: 2	Status: Final	Method: 8081A	Minor Revision Date: None

Table 8-9

FINAL CONCENTRATIONS OF EVAL MIX
(µg/mL)

Compound	Concentration
Endrin	0.050
4,4'-DDT	0.050
TCMX	0.020
DCB	0.020

Revision: 2	Status: Final	Method: 8081A	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/ Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Water or Solid	Standard	7 for water; 14 for soil (For TCLP: 14 days to preparative extraction)	The pesticide extracts from sample preparation are to be analyzed within 40 days from extraction date.	Extracts are stored in a secure area in a separate refrigerator from original samples at 2-6 C.
Water or Solid	NYSDEC	5 for water or soil from verified time of sample receipt (For TCLP: 5 days to preparative extraction)	The pesticide extracts from sample preparation are to be analyzed within 40 days from extraction date.	Extracts are stored in a secure area in a separate refrigerator from original samples at 2-6 C.

Revision: 2	Status: Final	Method: 8081A	Minor Revision Date: None

10.0 PROCEDURE

10.1 Apparatus Setup

10.1.1 The GC is equipped with two columns connected by a "Y" connector to one injection point and two electron capture detectors.

10.1.2 The carrier gas flow and temperature profile may vary somewhat to optimize analytical separation.

10.1.3 Recommended temperatures are set as follows: Injector = 200 C; Detector = 325 C Temperature program: Column Initial = 150 C. Initial Hold = 1.0 min. Column rate = 5 C/min. Column final = 280 C. Final Hold = minimum of 10 minutes and may be extended due to presence of late-eluting compounds in extracted samples.

10.1.4 The autosampler injects 2.0 μL of sample extract into each GC column.

10.1.5 Transfer of extracts to instrument autosampler vials shall be accomplished with disposable pipettes or graduated Wiretrol® micropipettes, depending if dilutions are being made.

10.2 Establishing Retention Time Windows

10.2.1 Make three injections of all compounds and multi-response products over a 72-hour period.

10.2.2 Calculate the standard deviation of the three retention times for each single component standard.

10.2.3 Calculate the multicomponent retention time windows for five peaks based on the toxaphene and chlordane mixture. The windows will apply to all multicomponents. Pattern recognition is still the primary means of identification.

10.2.4 The retention time window is defined as plus or minus 3 times the standard deviation from the retention time of the initial mid-level standards in the continuing calibration sequence. However, analyst experience should weigh heavily. Pattern recognition should primarily be used for multi-response products.

10.2.5 Calculate retention-time windows for each single component analyte, toxaphene and chlordane mid-level on each GC column whenever a new GC column is installed.

10.2.6 Retain this data so that it can be easily retrieved.

10.2.7 To facilitate analyte identification, the retention time windows are set as percentages (±% realtime of daily calibration) into the data system. These values are slightly wider than calculated windows.

10.3 Column Priming

10.3.1 If the column has not been used for more than a day, it is recommended that the column be deactivated or primed by injecting a standard mixture approximately 20 times more concentrated than the mid-level standard. Inject prior to initial or daily calibration.

10.4 Initial Calibration

10.4.1 Analyze an instrument blank (hexane) to verify the analytical system is clean (no target compounds are present greater than the quantitation limit) before standards.

10.4.2 An evaluation mixture (EVAL) is analyzed to check for the degradation of 4,4'-DDT and endrin. Calculate breakdown from the EVAL as follows:

$$\%\ \text{breakdown for } 4,4-\text{DDT} = \frac{\text{Total DDT degradation peak area (DDE + DDD)}}{\text{Total DDT peak area (DDT + DDE + DDD)}} \cdot 100$$

$$\%\ \text{Breakdown for Endrin} = \frac{\text{Total endrin degradation peakarea (endrin aldehyde + endrin ketone)}}{\text{Total endrin peak area (endrin + endrin aldehyde + endrin ketone)}} \cdot 100$$

If degradation of DDT or endrin exceeds 15% for either compound, take corrective action before proceeding with calibration. Corrective action may include but is not limited to snipping the column or changing the injection port liner.

10.4.3 The initial calibration of single-component pesticides and the multi-response components are performed at the five levels identified with suffix L, ML, M, MH, and H (see Tables 8-3 and 8-4). See Table 10-1 for specific calibration sequence. The initial single component pesticide calibration (Pesticide Mix A and B) contain some compounds that are not required for TCLP analysis.

10.4.4 Initial calibration for multi-response technical chlordane and toxaphene consists of choosing five of the largest responding peaks to gauge response. Because of the varying patterns of technical chlordane from manufacturers and sites, the peaks applicable to samples must be examined to ensure pattern match.

10.4.4.1 Only the mid-level standards for toxaphene and technical chlordane are analyzed unless determined (after sample analysis) that a full calibration is needed.

10.4.4.2 Technical chlordane may be quantitated and reported if the sample pattern resembles that of the quantitation standard. Generally, the chlordane pattern varies considerably. Therefore, a-chlordane, g-chlordane and heptachlor (the major components) are quantitated and reported separately unless otherwise required.

Table 10-1

INITIAL CALIBRATION SEQUENCE

Instrument Blank (hexane)
EVAL
INDA L
INDA ML
INDA M
INDA MH
INDA H
INDB L
INDB ML
INDB M
INDB MH
INDB H
Chlordane M
Toxaphene M
Pesticide Reference A
Pesticide Reference B
EVAL

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10.4.5 Calculate the ratio of the response to the amount injected, defined as the calibration factor (CF), for each analyte at each standard concentration.

$$\text{Calibration Factor (CF)} = \frac{\text{Total Area of Peak *}}{\text{Mass Injected (in nanograms)}}$$

*For multi-response pesticides, use the total area of 3-5 unique peaks used for quantitation. The number and specific peaks may be adjusted depending upon sample matrix or pattern weathering.

10.4.6 Calculate the mean calibration factor for all compounds and surrogates. Calculate the % Relative Standard Deviation (%RSD) of the CF_x values for the initial calibration using the following equations:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}_x\right)^2}{n-1}} \qquad \%RSD = \frac{SD}{\overline{CF}_x} \cdot 100$$

where:

SD = Standard derivation using sample population (n-1).
CF_x = Mean CF for each compound "x."
CF_i = Calibration factor for each calibration level "i" for the compound "x" of interest.
n = Number of calibration levels.

10.4.7 If the % RSD of any compound is 20% or less, linearity through the origin is assumed and the calibration factor is assumed to be constant over the calibration range, and the mean calibration factor may be used for quantitation.
10.4.8 If any analytes are greater than the 20% RSD, the initial calibration is valid as long as no one analyte is greater than 40% RSD and the mean of the RSD values for all analytes in the calibration is less than 20% (Grand Mean approach). Note: The analyst and the data user must be aware that the use of the approach listed in this Section (i.e., the average of all RSD values $\leq 20\%$) will lead to greater uncertainty of those analytes for which the RSD is greater than 20%. The analyst and the data user should review the associated quality control results carefully, with particular attention to the matrix spike and laboratory control sample results to determine if the calibration linearity poses a significant concern. If this approach is not acceptable for a particular application, then the analyst may need to adjust the instrument operating conditions and/or the calibration range until the RSD is $\leq 20\%$, or apply a regression analysis.
10.4.8.1 The mean RSD criterion applies to all analytes in the standards, regardless of whether or not they are of interest for a specific project.
10.4.8.2 The data user must be provided with either a summary of the initial calibration data or a specific list of those compounds for which the RSD exceeded 20% and the results of the mean RSD calculation.
10.4.9 If the RSD of the calibration or response factors is greater than 20% over the calibration range, then linearity through the origin cannot be assumed. The analyst may then utilize a linear regression equation that is not forced through the origin.
10.4.10 Linear regression can also be used for analytes that do meet the RSD limits in Section 10.4.8. Calibration is valid when the regression calculation generates a correlation coefficient for each compound of interest greater than or equal to 0.995.
10.4.11 If the linear correlation coefficient is <0.995, a second order curve may be used if at least 6 calibration standards are used. If the coefficient of determination of the second order curve is $\geq$0.990, use this second order curve for quantitation of the compound of interest. Additional concentration levels of stan-

dards are required for higher order curves. Refer to Method 8000 for guidance. Do not apply higher order curves without consulting the supervisor.

10.4.12 When toxaphene or technical chlordane, are suspected or found at a given site, standard curves for those analytes must be run and checked against samples for pattern recognition. If positively identified, calibration must be established before samples can be quantified.

10.4.12.1 Technical chlordane may be quantitated and reported if the sample pattern resembles that of the quantitation standard. Generally, the chlordane pattern varies considerably. Therefore, a-chlordane, g-chlordane and heptachlor (the major components) are quantitated and reported separately unless otherwise required.

10.4.13 If initial calibration requirements mentioned in the above sections cannot be met, then calibration is not valid and, therefore, the analyst should check instrument operation conditions and recalibrate.

10.4.14 Analyze a reference standard from a second source (ICV) immediately following an initial calibration to verify the curve. The curve is deemed acceptable provided each analyte in the reference standard calibration is within 15% difference of the mean calibration factor or within 15% of the theoretical concentration, depending on whether quantitation will be by mean calibration factor or from regression analysis, respectively. The Grand Mean Approach may be used for the ICV except where instructed otherwise for specific clients. See Section 13.

10.4.15 Quantitation of samples must continue to be done using the procedure initially used.

10.5 Daily Calibration and Continuing Calibration Verification

10.5.1 The working calibration curve or calibration factor must be verified after initial curve at the beginning of the analytical sequence and if a lapse in time >12 hours, has occurred from last continuing standard.

10.5.2 An instrument blank (hexane) is run to verify that the analytical system is clean (no target compounds are present > PQL.)

10.5.3 The EVAL standard is analyzed to check degradation. The degradation of DDT or endrin must not exceed 15% for each compound.

10.5.4 A calibration check standard (calibration verification, continuing standard) equivalent to the mid-level calibration standard must be analyzed at the beginning of the analytical sequence and after every 10 samples or no longer than 12 hours (INDA and INDB).

10.5.5 Continuing calibration and daily calibration verifications must be judged against the quantitation method used at the start. That is, if using a curve, judge standards as acceptable if $\leq \pm$ 15% Drift of their true value; if using the mean calibration factor, judge standards' calibration factor as acceptable if $\leq \pm 15\%$ difference against the mean calibration factor.

Calculate % Drift and % Difference using the equations below. When using a curve:

$$\%\ \text{Drift} = \frac{\text{Calculated Conc.} - \text{Theoretical Conc.}}{\text{Theoretical Conc.}} \cdot 100$$

When using mean calibration factor:

$$\%\ \text{Difference} = \frac{\text{CF Verification Standard} - \overline{CF_x}}{\overline{CF_x}} \cdot 100$$

10.5.6 If the mean calibration factor is used for quantitation, then the calibration factor of the analyte must be ≤ 15% difference from the mean calibration factor.
10.5.7 If the regression curve is used for quantitating analytes in samples, then the concentration of the analyte in the calibration check standard must be ≤ 15% drift from the theoretical concentration (i.e., 85-115% recovery).
10.5.8 If the percentage difference or drift is greater than 15% for one or more analytes, the standard is acceptable provided no one analyte %D exceeds 30% and the mean of all analytes' %D in the standard is ≤15% difference or drift (Grand Mean approach). However, the discussion in Section 10.4.10 applies.
10.5.9 Instrument blanks (hexane) are allowed before CCV to minimize chances for carryover into the CCV to avoid invalidation of the curve due to random carryover. Indication of carryover from one sample to another requires reanalysis of the potentially contaminated sample only.

10.6 Calibration Verification Corrective Action
10.6.1 If initial or continuing calibration criteria is not met, stop sample analysis immediately. Inspect the system. Minor corrective action may be performed such as changing the septum or injection port liner.
10.6.2 If after the minor corrective action a compliant CCV (continuing calibration verification [INDAM, INDBM]) is analyzed, sample analysis may continue. Any samples not bracketed by valid CCVs must be reanalyzed and bracketed by compliant CCVs.
10.6.3 If minor corrective action does not fix the problem, major corrective action may be taken such as cleaning the injection port, clipping the column ends, baking out the detectors, or installing new columns. If major corrective action is taken, a new calibration curve must be run and all associated samples reanalyzed.

10.7 Sample Analysis
10.7.1 Once a successful initial calibration is complete, the primary analytical daily or batch sequence may begin (see Table 10-2) for pesticide analyses.

Table 10-2

Daily Run Sequence

Instrument Blank (hexane)
EVAL
INDAM
INDBM
Toxaphene
Technical Chlordane
Samples, not to exceed 12 hours
Instrument Blank
EVAL
INDAM
INDBM
Samples, not to exceed 12 hours
Instrument Blank
EVAL
INDAM
INDBM

10.7.2 Dilute TCLP extracts 20-fold before injection. Table 11-1 PQL accounts for this factor and preparation factor.
10.7.3 If any pesticide is detected in a sample at a level that exceeds the linear range of the calibration curve, the sample must be diluted to within the calibrated range. The dilution of samples may also be required to bring the chromatogram on scale even when no analytes of interest are detected.
10.7.4 Instrument blanks (hexane) may be interspersed throughout the analytical run to check for carryover or when the system is run unattended (autosampler).
10.7.5 Peak detection is prevented by the presence of elemental sulfur. The samples are copper treated to remove sulfur at the preparatory stage. See extraction SOP.

10.8 Confirmation Analysis
10.8.1 Tentative identification of an analyte occurs when a peak falls within the retention time window of a target compound on the primary column and it is at, or above, the quantitation limit for that compound. Confirmation on a secondary GC column is then required.
10.8.2 The confirmation analysis is run simultaneously with the primary analysis but, if a separate confirmation is needed, the analysis should not exceed 40 days from date of extraction and only mid-level standards of compounds requiring confirmation need be analyzed. In addition, if confirmation of compounds at or near the quantitation limit is required, analysis of the lowest calibration standard may be done to demonstrate quantitation limits can adequately be met.
10.8.3 An analyte is confirmed as present if it falls within the retention time window for that compound on the confirmation column as well as on the primary column.
10.8.4 The confirmation analysis is qualitative only.

Instrument Conditions and Maintenance
See Table 10-3, Routine Maintenance Procedures.

Table 10-3

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
V3400/HP5890	Retention time shift	Change septum	Daily (or as needed)
V3400/HP5890	High degradation	Change injection liners, cut end of column	As needed
V3400/HP5890	High baseline	Bake out column, detector	As needed
V3400/HP5890	Non-linear calibration	Replace detector; replace column	As needed

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11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 If quantitating from a linear curve, the following equation is provided as a manual check.

$$\text{Concentration} = \text{mg/kg}, \mu\text{g/L} = \left[\frac{y - b}{m}\right]\frac{Vt}{S} \cdot Df$$

where:

y = Area of target analyte from initial curve,
b = Intercept area,
m = Slope (area /[µg/mL]),
S = Mass of soil sample extracted in grams(g) or volume of water sample (or TCLP extract) extracted in liters (L),
Vt = Final volume of the extract in milliliters (mL).
Df = Dilution factor. The dilution factor equals µL of extract used to make dilution + µL clean solvent µL of extract used to make dilution.

11.2 For TCLP extracts, multiply the result above by 1 mg/1000 µg to convert to the required TCLP reporting limits of mg/L.

Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound	Type	PQL		
		Soil (µg/kg)	Water (µg/L)	TCLP Extracts/ (Regulatory Level) (mg/L)
Aldrin	T, S	1.0	0.025	
α BHC	T, S	1.0	0.025	
β BHC	T, S	1.0	0.025	
γ BHC (Lindane)	T, S	1.0	0.025	0.0025/(0.4)
δ BHC	T, S	1.0	0.025	
α Chlordane	T, S	1.0	0.025	
γ Chlordane	T, S	1.0	0.025	
Chlordane	M	10.0	0.20	0.020/(0.030)
4,4'-DDD	T, S	2.0	0.05	
4,4'-DDE	T, S	2.0	0.05	
4,4'-DDT	T, S	2.0	0.05	
Dieldrin	T, S	2.0	0.05	
Endolsulfan I	T, S	2.0	0.05	
Endolsufan II	T, S	2.0	0.05	
Endosulfan Sulfate	T, S	2.0	0.05	
Endrin	T, S	2.0	0.05	0.005/(0.02)

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound	Type	PQL		
		Soil (µg/kg)	Water (µg/L)	TCLP Extracts/ (Regulatory Level) (mg/L)
Endrin Aldehyde	T, S	2.0	0.05	
Endrin Ketone	T, S	2.0	0.05	
Heptachlor	T, S	2.0	0.025	0.0025/(0.008)
Heptachlor Epoxide	T, S	2.0	0.025	0.0025/(0.008)
Methoxychlor	T, S	10.0	0.25	0.025/(10.0)
Toxaphene	T, S	50.0	1.0	0.10/(0.5)
Tetrachlorometaxylene (TCMX)	Q	NA	NA	
Decachlorobiphenyl (DCB)	Q	NA	NA	
DBCP	M	TBD	TBD	
Diallate	M	TBD	TBD	
Isodrin	M	TBD	TBD	
Hexachlorobenzene	M	TBD	TBD	
Hexachlorocyclopentadiene	M	TBD	TBD	
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Key Type:

NA = Not Applicable.
TBD = To Be Determined, if required.
T = Compound/analyte is target compound routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike.
L = Compound/analyte is routinely used as a LCS spike.
Q = Compound/analyte is used as surrogate spike.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

12.1 Refer to the Quality Control Table below and the following sections for corrective actions.

12.2 Each sample is spiked with the surrogates DCB and TCMX. The DCB is the primary surrogate. Calculate its recovery. If peaks interfere with DCB, then the TCMX should be evaluated for acceptance. Proceed with corrective action when two surrogates are out of limits for a sample.

12.3 The sample is to be reextracted and reanalyzed unless it can be documented (chromatogram) that factors such as matrix effects are responsible. A corrective action report must be filed.

12.4 If any quality control criteria cannot be met, but the problem is correctable, the sample(s) affected must be reanalyzed.

12.5 Data that must be submitted with out of control events must be accompanied by a corrective action report.

12.6 If any criteria cannot be met, but the problem is correctable, the sample(s) affected must be reanalyzed.

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Acceptance Criteria	Corrective Action
Method Blank	1 per batch of 20 or fewer samples.	< PQL	Reanalyze samples having concentrations ≥ PQL
Matrix Spike (MS) Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples	- Project requirements, if specified - 20% for waters - 35% for soils	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples	Laboratory limits	Notify Project Manager immediately. Re-extract and reanalyze the entire batch.
Surrogate	Every sample, blanks and QC	Laboratory limits. Evaluate DCB recovery. In event of interference evaluate TCMX recovery.	Reextract batch for blank or LCS failure. Reextract affected sample(s) unless matrix effect is evident from chromatogram.
TCLP Blank	1 per batch of 20 or fewer sample TCLP extracts	< TCLP PQL	Reanalyze samples having concentrations ≥ PQL

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Table 12-2

CONTROL LIMITS FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameters Charted	Type of Samples Charted	Source of Limits	Frequency Updated
Surrogates	TCMX, DCB	Water Pesticides	In-House	Annual
Surrogates	TCMX, DCB	Soil Pesticides	In-House	Annual
LCS	All compounds	Water/Soil	In-House	Annual

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Table 12-3.1

CRITERIA ASSIGNED TO ROUTINE QC SAMPLES

QC Type	Matrix	Analytes	Nominal Spike Amount	Recovery	RPD	Source
LCS MS MSD	Water	alpha-BHC	0.1000 each µg/L (0.01 µg/mL on column)	Table 12-1 Table 12-1 Table 12-1	-- -- Table 12-1	In-house In-house In-house
		beta-BHC				
		gamma-BHC				
		delta-BHC				
		Heptachlor				
		Aldrin				
		Heptachlor epoxide				
		alpha-chlordane				
		gamma-chlordane				
		Endosulfan I				
		4,4'-DDE				
		Dieldrin				
		Endrin				
		Endosulfan II				
		4,4'-DDD				
		4,4'-DDT				
		Endrin aldehyde				
		Endosulfan sulfate				
		Methoxychlor	1.000 (0.1 µg/mL on column)			
Surrogate	Water	DCB, TCMX	0.2 µg/L each (0.02 µg/mL on column)	Table 12-1	--	In-house
LCS MS MSD	Soil	alpha-BHC	3.33 µg/kg (0.01 µg/mL on column) each	Table 12-1 Table 12-1 Table 12-1	-- -- Table 12-1	In-house In-house In-house
		beta-BHC				
		gamma-BHC				
		delta-BHC				
		Heptachlor				
		Aldrin				
		Heptachlor epoxide				
		alpha-chlordane				
		gamma-chlordane				
		Endosulfan I				
		4,4'-DDE				
		Dieldrin				
		Endrin				

Table 12-3.1

CRITERIA ASSIGNED TO ROUTINE QC SAMPLES

QC Type	Matrix	Analytes	Nominal Spike Amount	Recovery	RPD	Source
		Endosulfan II				
		4,4'-DDD				
		4,4'-DDT				
		Endrin aldehyde				
		Endosulfan sulfate				
		Methoxychlor	33.3 µg/kg (0.1 µg/mL on column)			
Surrogate	Soil	DCB, TCMX	6.66 µg/kg each (0.02 µg/mL on column)	Table 12-1	--	In-house

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Table 12-3.2

CRITERIA ASSIGNED TO QUALITY CONTROL SAMPLES - TCLP

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
LCS MS MSD	TCLP Extract	Endrin, gamma-BHC, heptachlor, heptachlor epoxide, methoxychlor	0.040 mg/L includes preparation and dilution factors (0.04 µg/mL on column)	See Table 12-1	--	In-house
Surrogates	TCLP Extract	TCMX, DCB	0.020 mg/L each includes preparation and dilution factors (0.02 µg/mL on column)	See Table 12-1	--	In-house

Revision: 2	Status: Final	Method: 8081A	Minor Revision Date: None

13.0 SPECIAL PROJECT REQUIREMENTS

Client-Specific Quality Control Requirements

13.1 AFCEE

13.1.1 Second column must be fully calibrated and results quantitative.

13.1.2 LCS must contain all target analytes. The QC samples in Table 12-3.1 are prepared in Organic Preparation. Additionally, the following Table 13-1, QC Samples for toxaphene, are prepared.

13.1.3 Follow AFCEE-assigned limits for QC samples.

Table 13-1

QC SAMPLES FOR TOXAPHENE

QC Type	Matrix	Nominal Spike Amount
LCS MS MSD	Water	4 µg/L (0.04 µg/mL on column)
LCS MS MSD	Soil	133 µg/kg (0.04 µg/mL on column)

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13.1.4 Both INDAM and INDBM must be analyzed and in control every 10 samples.
13.1.5 All AFCEE projects are subject to project-specific variances.
13.1.6 MDL may not be >1/2 reporting limit (RL) unless a variance was obtained.
13.1.7 A calibration standard at or below the RL must be included.

13.2 U.S. Army Corps of Engineers (ACE)

13.2.1 Method Quantitation Limit (MQL) is between 3-10 times MDL. The low calibration standard concentration must be ≥3 times MDL. The Method Reporting Limit (MRL) may slide between two times the MDL and project-specific or regulatory requirements. Results below the MRL must be reported "ND." Results between the MQL and MRL must be reported as estimated (J flagged).
13.2.2 If using the grand mean approach for RSD, no single analyte may be greater than 40% RSD.
13.2.3 If using linear regression, the correlation coefficient must be ≥0.995.
13.2.4 Initial Calibration Verification. The initial calibration curve shall be verified as accurate with a standard purchased or prepared from an independent source. This initial calibration verification (ICV) involves the analysis of a standard containing all of the target analytes, typically in the middle of the calibration range, each time the initial calibration is performed. The % recovery of each target analyte in the ICV is determined from the initial calibration and compared with the specifications for the CCV in each method (except for mercury by CVAA) as outlined in the Shell Document Tables 7 - 14.
Note for methods which report several (>5) target analytes, a small percentage of sporadic marginal failures may be tolerated (i.e., will not trigger reextraction and analysis of the entire batch). The number of target analytes reported for the method will dictate the number of allowable QC failures as given below.
Refer to the individual method tables for details on the implementation of this concept.

Number of Target Analytes	Number of Allowed Marginal Failures
5 - 15	1
16 - 30	2
31 - 45	3
46 - 60	4
61 - 75	5
76 - 90	6
91 - 105	7

The marginal failure allowance entails the application of an expanded acceptance criterion. If these QC criteria are not met, a new initial calibration must be performed.

13.2.5 For method blanks, check that no target compounds are found above one-half the required quantitation limit.

13.2.6 ACE criteria are summarized in Table 13-2.

Table 13-2

SUMMARY OF METHOD QUALITY OBJECTIVES FOR METHOD 8081 PESTICIDES

QC Element	Target Analyte/Surrogate	Sporadic Marginal Failure
DDT/Endrin %Breakdown (ACE Shell Document Section 8.2)	DDT & Endrin %Breakdown ≤15% each	Not Applicable
Initial Calibration (ACE Shell Document Section 9.2.2.2)	Primary Evaluation: r ≥0.995, %RSD ≤20%, r^2 ≥0.990 Alternative Evaluation: Mean %RSD for all target analytes ≤20%	No allowance Alternative Evaluation: Maximum allowable %RSD for each target analyte ≤40%
ICV (ACE Shell Document Section 9.3/9.3.1)	%Rec = 85% - 115%	Sporadic Marginal Failures[1]: %Rec = 70% - 130%
CCV (ACE Shell Document Section 9.5/9.5.2/9.5.2.2)	Primary Evaluation: %Drift ≤15%, %D ≤15% Alternative Evaluation: Mean %Drift/%D for all target analytes ≤15%	No allowance Alternative Evaluation: Maximum allowable %Drift/%D for each target analyte ≤30%
MB (ACE Shell Document Section 10.2.1/1.4.1)	Analytes < MDL Check Sample (~2X MDL)	Not Applicable
LCS (ACE Shell Document Section 10.2.2/11.4.2)	Water: %Rec = 50% - 130% Solids: %Rec = 50% - 130%	Sporadic Marginal Failures[1]: %Rec = 30% - 150%
MS (ACE Shell Document Section 10.2.3/11.4.3/11.4.3.2)	%Rec = 40% - 140%	Sporadic Marginal Failures[1]: %Rec = 30% - 150%
MSD/MD (ACE Shell Document Section 10.2.4/11.4.4)	RPD ≤50%	RPD ≤60%
Surrogates (ACE Shell Document Section 10.2.5/11.4.5)	Interference-Free Matrix: Water: %Rec = 50% - 130% Solids: %Rec = 50% - 130% Project Sample Matrix: %Rec = 40% - 140%	Not Applicable
Target Analyte Confirmation (12.3)	RPD ≤40%	RPD ≤40%

[1]The number of Sporadic Marginal Failure (SMF) allowances depend upon the number of target analytes reported from the analysis. For instance, if the full list of 21 compounds are reported from the GC/ECD analysis, then two (2) SMFs are allowed to the expanded criteria presented. If the MS includes only a subset of compounds, allow only one (1) SMF for that QC element. Refer to Section 9.3 of ACE Shell Document for additional information on the application of sporadic marginal failures.

13.3 North Carolina DENR

13.3.1 A mid-concentration (of the calibration range) continuing verification standard must be analyzed. The INDA M and INDB M standards are not mid-concentration. Prepare a standard at half that of INDA H and INDB H. Alternate use of this standard with the lower standards INDA M and INDB M every 12 hours.

13.3.2 Two MS/MSD pairs must be prepared and analyzed every 10 samples.

14.0 SAMPLE DISPOSAL

14.1 See waste disposal, SOP A.10.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Analysis of Soil or Water Samples for Chlorinated Herbicides by Method 8151A			
Reviewer: R. Neary		Peer Reviewer: D. Fournier	
Implementation Date: 11/7/00		Last Annual Review Date: 11/2/00	
Revision: 4	Status: Final	Method: 8151A	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP details the esterification and GC/ECD analysis of extracted soil, aqueous, and waste samples for chlorinated herbicides by Method 8151A.

2.0 METHOD SUMMARY

2.1 This SOP provides procedures for derivitazition of acids to methyl esters and chromatographic conditions for the analysis of chlorinated acid herbicide in water, soil, and waste samples.

2.2 Samples are extracted and hydrolyzed following SOP Ext.7 (soil) and Ext.8 (water).

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves and a lab coat. For more information see the E & E, Inc. Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 Diazomethane is a potentially explosive gas that is subject to rapid decomposition upon exposure to strong light, heat or rough surfaces. In this method, diazomethane is codistilled with ether, a highly flammable liquid. The greatest hazard is present during the distillation process, due to the use of heat. This procedure is widely used and safe if the proper precautions are used. Lab coat, safety glasses and protective gloves are to be worn at all times. The entire procedure must be performed in a fume hood. The distillation portion of the procedure should be performed with the apparatus behind a safety shield with the hood window pulled down. Before use, always check glassware for chips, cracks and scratches, particularly the distillation apparatus pieces. If any defects are found, do not use it. When cleaning the glassware, never use soap or a brush, as they could scratch or etch the glass. Do not use the Diazald kit glassware for any other purpose. It is also recommended that the MSDS for the reagents being used be read before performing the procedure.

3.3 Due to the dangers of working with diazomethane, all work must be performed under an approved hood with a safety shield. Diazomethane must not be stored (unless frozen in an amount not to exceed 20 mL). Any unused diazomethane solution must be decomposed immediately after use with acetic acid and disposed of properly.

3.4 The laboratory's usual list of compounds reported are asterisked in Table 10-2. Reporting can be extended to other compounds.

3.5 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Method 8151A, (Rev. 1, 12/96) Method 8000B, SW-846 3rd Ed. Updated III, June 1997.

4.2 SOP Ext.7 (soil) and Ext.8 (water).

4.3 Training SOP QA.5, A.23, and A.29.

4.4 Data Review SOP A.25.

4.5 Laboratory Quality Assurance Manual.
4.6 MDL SOP A.18.
4.7 Corrective Action Procedure, QA.1

5.0 DEFINITIONS/ACRONYMS

5.1 **MDL** - Method Detection Limit as determined by 40 CFR Part 136, Appendix B. Method detection limits must be determined yearly for both water and soil matrices. See SOP A.18 for procedure.
5.2 **PQL** - Practical Quantitation Limit is the concentration above the MDL which can be reasonably obtained and is used as the limit of reporting.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Method interferences may be caused by solvents, glassware, and other reagents. To prevent such interferences, only high purity solvents and reagents must be used. All glassware must be thoroughly cleaned and rinsed with organic-free water. Prior to use, glassware should be solvent rinsed with the next solvent to be used.
6.2 Organic acids, especially chlorinated ones, cause interferences with the determination by methylation.
6.3 Alkaline hydrolysis and subsequent extraction removes many chlorinated hydrocarbons and phthalates esters, but may result in formation of aldol condensates if any acetone remains from the extraction process, which results in the loss of Dinoseb.
6.4 Herbicides react readily with alkaline substances. Glassware must be acid rinsed and then water rinsed to a constant pH. Sodium Sulfate must be acidified. Glass wool must be acid treated.
6.5 Sample extracts must be dry prior to esterification or poor recoveries will result.
6.6 Diethyl ether preserved with ethanol should not be used.

7.0 INSTRUMENTATION AND EQUIPMENT

Varian 3400 or Hewlett Packard 5890A equipped with electron capture detectors, split/splitless injectors, and an autosampler.
PE Nelson data system with TURBO Software.
RTX-5 30-m or 60-m x 0.32-mm ID 0.50 or 1.0 μm film thickness.
RTX-35 30-m or 60-m x 0.32-mm ID 0.50 μm film thickness.

8.0 REAGENTS AND MATERIALS

- Diazald Kit (Aldrich)
- Volumetric flasks; 10-mL, 50-mL
- Screw cap vials; 1.0-mL, 15-mL
- Gastight syringes, various sizes from 10-µL through 500-µL, Hamilton brand or equivalent
- Wiretrol® disposable pipettes, various sizes from 10-µL through 200-µL
- Safety shield
- Beaker; 500-mL and 50-mL
- Ring stand and clamps
- Graduated cylinder, 25-mL
- Ice bath
- Test tubes, screw top with Teflon®-lined caps

See Table 8-1, Summary of Standard and Reagents.

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Compound	Concentration (µg/mL)
5-Hydroxydicamba Methyl Ester Derivative	Chem Service	5-Hydroxydicamba Methyl Ester Derivative	100
DCAA Methyl Ester surrogate	Accustandard or equivalent	DCAA methyl ester	100
8151 Herbicides Mix Methyl Ester Derivatives	Accustandard (M-8151) or equivalent	Acifluorfen methyl ester	100
		Bentazon methyl ester	100
		Chloramben methyl ester	100
		2,4-D methyl ester	100
		2,4-DB methyl ester	100
		Dalapon methyl ester	100
		DCPA methyl ester	100
		Dicamba methyl ester	100
		Methyl-3,5-dichlorobenzoate	100
		Dichlorprop methyl ester	100
		Dinoseb methyl ester	100
		4-Nitroanisole	100
		Pentachloroanisole	100
		Picloram methyl ester	100
		2,4,5-T methyl ester	100
		2,4,5-TP methyl ester	100
		MCPA methyl ester	10,000
		MCPP methyl ester	10,000
8151 Herbicides Mix Methyl Ester Derivatives Second source	Protocol Analytical Supplies, Inc.	Acifluorfen methyl ester	100
		Bentazon methyl ester	100
		Chloramben methyl ester	100
		2,4-D methyl ester	100
		2,4-DB methyl ester	100
		Dalapon methyl ester	100

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Compound	Concentration (µg/mL)
		DCPA methyl ester	100
		Dicamba methyl ester	100
		3,5-Dichlorobenzoic acid methyl ester	100
		Dichloroprop methyl ester	100
		Dinoseb methyl ether	100
		4-Nitrophenol methyl ester	100
		Pentachlorophenol methyl ester	100
		Picloram methyl ester	100
		2,4,5-TP methyl ester	100
		2,4,5-T methyl ester	100
		MCPA methyl ester	10,000
		MCPP methyl ester	10,000
8151 Herbicides Mix Underivitized	Accustandard or equivalent	Acifluorfen	100
		Bentazon	100
		Chloramben	100
		2,4-D	100
		2,4-DB	100
		Dalapon	100
		DCPA Diacid	100
		Dicamba	100
		3,5-Dichlorobenzoic acid	100
		Dichlorprop	100
		Dinoseb	100
		4-Nitrophenol	100
		Pentachlorophenol	100
		Picloram	100
		Silvex (2,4,5-TP)	100

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Compound	Concentration (µg/mL)
		2,4,5-T	100
		MCPA	10,000
		MCPP	10,000
5-Hydroxydicamba	Chem Service	5-Hydroxydicamba	100
ASTM Type II Water	In-house System		NA
DCAA Acid (surrogate)	Chem Service		2000
Hexane, Ultra Resi-analyzed	J.T. Baker or equivalent		Neat
Diazald: N-methyl-N-nitroso-p-toluene sul-fonamide	Aldrich Chemical		Neat
Potassium Hydroxide (KOH)	J.T. Baker or equivalent		Neat
Silicic Acid, 100 mesh, chromatographic grade	Aldrich Chemical		Neat
Diethyl Ether, preserved with BHT or unpreserved.	J.T. Baker or equivalent		Neat
Acetone, Ultra-Resi grade	J.T. Baker or equivalent		Neat
(2-ethoxyethoxy)ethanol, also known as di(ethylene glycol) ethyl ether	Aldrich Chemical		Neat
Acetic Acid, Reagent Grade	J.T. Baker or equivalent		Neat
Ethanol	J.T. Baker or equivalent		Neat

Revision: 4	Status: Final	Method: 8151A	Minor Revision Date: None

8.1 Prepare standards in clean glassware rinsed with hexane. Record preparation in the standard preparation logbook.

8.2 Store all standards in refrigerator at 2-6 C. Unopened stocks expire one year from receipt unless another date is specified by the manufacturer. Working standards expire six months from preparation date unless preceded by expiration of the stock.

Standard Solution Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
5-Hydroxdicamba Methyl Ester Intermediate	5-Hydroxdicamba Methyl Ester Derivitative	1 mL derivitized and diluted to 10 mL hexane	10 µg/mL
8151 Low	8151 Herbicides Mix Methyl Ester Derivatives 5-Hydroxydicamba Methyl Ester Intermediate DCAA Methyl Ester	5.0 µL plus 50.0 µL plus 5.0 µL to 10 mL hexane	See Table 8-3
8151 Med Low	8151 Herbicides Mix Methyl Ester Derivatives 5-Hydroxydicamba Methyl Ester Intermediate DCAA Methyl Ester	10.0 µL plus 100 µL plus 10.0 µL to 10 mL hexane	See Table 8-3
8151 Mid	8151 Herbicides Mix Methyl Ester Derivatives 5-Hydroxydicamba Methyl Ester Intermediate DCAA Methyl Ester	20.0 µL plus 200 µL plus 20.0 µL to 10.0 mL hexane	See Table 8-3
8151 Med High	8151 Herbicides Mix Methyl Ester Derivatives 5-Hydroxydicamba Methyl Ester Intermediate DCAA Methyl Ester	40.0 µL plus 40.0 µL plus 40.0 µL to 10.0 mL hexane	See Table 8-3
8151 High	8151 Herbicides Mix Methyl Ester Derivatives 5-Hydroxydicamba Methyl Ester Intermediate DCAA Methyl Ester	50.0 µL plus 50.0 µL plus 100 µL to 10.0 mL hexane	See Table 8-3

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
Potassium Hydroxide Solution	Potassium Hydroxide (KOH)	2.5 g into 4 mL water	62.5% (w/v)
5% Acetic Acid Solution	Acetic Acid	5 mL to 100 mL ASTM Type II water	5% (v/v)
8151 Herbicide Spike	8151 Herbicides Mix Underivitized 5-Hydroxydicamba	350 µL 350 µL to 10 mL acetone	3.5 µg/mL all except MCPP and MCPA @ 350 µg/mL
8151 Surrogate	DCAA Acid	125 µL to 50 mL acetone	5 µg/mL
ICV	8151 Herbicides Mix Methyl Ester Derivative Second Source DCAA Methyl Ester (No second source available for 5-hydroxydicamba Methyl Ester Intermediate)	20.0 µL plus 200 µL plus 20.0 µL to 10.0 mL hexane	Same concentrations as mid level calibration standard

Revision: 4	Status: Final	Method: 8151A	Minor Revision Date: None

Table 8-3

METHOD 8151 CALIBRATION CONCENTRATIONS
HERBICIDE METHYL ESTER (µg/mL)

Compound	Low	Med Low	Mid	Med High	High
Dalapon	0.0500	0.100	0.200	0.400	0.500
3,5- Dichlorobenzoic acid*	0.0500	0.100	0.200	0.400	0.500
4-Nitrophenol	0.0500	0.100	0.200	0.400	0.500
Dicamba	0.0500	0.100	0.200	0.400	0.500
Dichloroprop	0.0500	0.100	0.200	0.400	0.500
2,4-D	0.0500	0.100	0.200	0.400	0.500
Pentachlorophenol	0.0500	0.100	0.200	0.400	0.500
2,4,5 – TP (Silvex)	0.0500	0.100	0.200	0.400	0.500
Chloramben*	0.0500	0.100	0.200	0.400	0.500
5-Hydroxydicamba*	0.0500	0.100	0.200	0.400	0.500
2,4,5-T	0.0500	0.100	0.200	0.400	0.500
2,4-DB	0.0500	0.100	0.200	0.400	0.500
Dinoseb	0.0500	0.100	0.200	0.400	0.500

Table 8-3

METHOD 8151 CALIBRATION CONCENTRATIONS
HERBICIDE METHYL ESTER (µg/mL)

Compound	Low	Med Low	Mid	Med High	High
Bentazon*	0.0500	0.100	0.200	0.400	0.500
Picloram*	0.0500	0.100	0.200	0.400	0.500
Acifluorfen*	0.0500	0.100	0.200	0.400	0.500
DCAA (Surrogate)	0.0500	0.100	0.200	0.400	1.00
DCPA*	0.0500	0.100	0.200	0.400	0.500
MCPP	5.00	10.00	20.00	40.00	50.00
MCPA	5.00	10.00	20.00	40.00	50.00
Revision: 4	Status: Final	Method: 8151A		Minor Revision Date: None	

* Compounds not routinely reported

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/ Project	Preparation (Days)	Analysis	Container Type and Preservative
Solid	Standard	14	Within 48 hours after derivitization	4,8,16-oz. jar 2-6 C
Water	Standard	7	Within 48 hours after derivitization	1-L or ½-g Amber 2-6 C
	NYSDEC	5 from date of receipt for soils or waters	Within 48 hours after derivitization	Same as above
Revision: 4	Status: Final	Method: 8151A	Minor Revision Date: None	

10.0 PROCEDURE

10.1 Preparation of Diazomethane

10.1.1 Prepare the fume hood by clearing the hood of all other equipment and reagents and cleaning a work area at least two feet in width.

10.1.2 Place the safety shield in hood.

10.1.3 Assemble the Diazald distillation apparatus as shown in the attached Aldrich bulletin, #AL-180, using a 100-mL generator flask. Two people must be present in the room at all times when diazomethane is being prepared and when methylation of samples is in progress.

10.1.4 Add the following reagents in the order listed to a 50-mL round bottom generator flask: 2-(2-ethoxyethoxy) ethanol (14 mL), ether (8 mL), potassium hydroxide solution (4 mL). (This solution must be at room temperature before use.)

10.1.5 Turn on water to the condenser.

10.1.6 Place safety shield in front of apparatus.

Aldrich Bulletin #AL-180
(Only hard copy available in binder/QA file)

10.1.7 Place receiver flask and ether trap in ice water bath.
10.1.8 Prepare an ether solution of 5 g of Diazald in 45 mL ether (this will produce enough diazomethane for approximately 50 samples, scale down to 1 g in 9 mL for each set of 10 samples). Check the separatory funnel for leaks and close the stop cock.
10.1.9 Add the Diazald/ether solution to the separatory funnel.
10.1.10 Rinse beaker used to dissolve Diazald with 2 to 3 mL of ether and add this to the separatory funnel.
10.1.11 Turn heating mantel on low setting, slowly increasing heat until the ether begins to boil (approximately 55 on a Variac).
10.1.12 Wait for about 2 mL (30 drops) of ether to distill over into the receiving flask.
10.1.13 Slowly add the Diazald/ether solution to the reaction flask, about 0.2 to 0.5 mL at a time, over a period of about 20 minutes. Do not allow the reaction flask to overheat at this stage (temperature should be approximately 65 C).
10.1.14 After all of the Diazald is added, add 10 mL ether to the separatory funnel and slowly add this to the reaction flask. When this has distilled over and the distillate is clear, shut off the heating mantel and lower it from the reaction flask.
10.1.15 Remove receiving flask and cap loosely with Teflon® cap, place flask back in ice bath. Immediately proceed to methylation of samples. This is subsequently refined to a Diazomethane solution.

10.2 Preparation of Methyl Ester Derivative
10.2.1 Obtain the sample extracts prepared as in SOP Ext.7 or Ext. 8. They should be in 15-mL screw-top vials at this point; if not, transfer them.
10.2.2 Add 1 mL to 2 mL of diazomethane solution to each tube. The extracts should be yellow in color after addition of diazomethane. If not, additional diazomethane is required.
10.2.3 Cap and allow the samples to warm to room temperature and sit for approximately 1 hour with occasional swirling (do not shake). If the samples turn clear, add 2 mL of the diazomethane solution. If the samples are heavily colored, add additional diazomethane.
10.2.4 After allowing the samples to react for 1 hour, adjust the sample volume to 10.0 mL with hexane, using a premeasured 15-mL vial as a guide. Mix well.
10.2.5 Decompose the unreacted diazomethane by adding approximately 10 mg of silicic acid.

10.3 Clean Up
10.3.1 Clean up must be completed on the same day as the preparation.
10.3.2 Never use metal brushes when cleaning the Diazald glassware
10.3.3 Add 100 mg silicic acid to decompose any residual diazomethane.
10.3.4 Take special care to neutralize the spent potassium hydroxide solution in the reaction flask with acetic acid.
10.3.5 Disassemble the distillation apparatus and clean with acetone, water, then acetone.
10.3.6 Dry glassware and return it to the storage box. Inspect glassware for contamination and reclean, if needed, with acetone.
10.3.7 Cleaning solution: Add 0.5 grams of silicic acid to the cleaning solution (acetone) to destroy any trace of diazomethane.
10.3.8 Return all reagents to refrigeration for storage.

10.4 Gas Chromatograph Parameters
10.4.1 The following parameters are recommended but may vary somewhat to optimize analytical separation.
- Carrier gas: Helium
- Make-up gas: Nitrogen

- Column flow: 8 mL/min
- Injection Temperature: 200 C
- Detector Temperature: 300 C

Temperature Program:

- Inject at 60 C and hold for 4 minutes
- Program at 10 C/min. to 140 C, hold 0 min.
- Program 4 C/min. to 265 C, hold 5 minutes for 30-meter column, hold 20 minutes for 60-meter column.
- Injection Volume: 2µL
- Make-up Flow: 30 mL/min.

Instrument Conditions and Maintenance

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Gas Chromatograph	Retention time shift	Change septum, check gas lines for leaks	As needed
Capillary Column	High baseline, change in response	Change injection liner, change "Y" splitter, cut end of column, bake out column or injector at higher temperature	As needed
Injection Port	Worn septum	Change septum	Daily or at the beginning of new sequence
Electron Capture Detector	Non-linear calibration	Replace detector and/or column	As needed

Revision: 4	Status: Final	Method: 8151A	Minor Revision Date: None

10.5 Initial Calibration

10.5.1 Analyze and prepare a calibration curve using the standards in Table 8-3. Inject these standards in order of concentration. The calibration standards are reported in µg/mL.

10.5.2 Calculate the ratio of the response to the amount injected, defined as the calibration factor (CF), for each analyte at each standard concentration.

$$\text{Calibration Factor (CF)} = \frac{\text{Total Area of Peak}}{\text{Mass Injected (in nanograms)}}$$

10.5.3 Calculate the mean calibration factor for all compounds and surrogates. Calculate the % Relative Standard Deviation (%RSD) of the CF_x values for the initial calibration using the following equations:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}_x\right)^2}{n-1}} \qquad \%RSD = \frac{SD}{\overline{CF}_x} \cdot 100$$

where:

SD = Standard derivation using sample population (n-1).
CF_x = Mean CF for each compound "x."
CF_i = Calibration factor for each calibration level "i" for the compound "x" of interest.
n = Number of calibration levels.

10.5.4 If the % RSD of any compound is 20% or less, linearity through the origin is assumed and the calibration factor is assumed to be constant over the calibration range, and the mean calibration factor may be used for quantitation.
10.5.5 If any analytes are greater than the 20% RSD, the initial calibration is valid as long as no one analyte is greater than 40% RSD and the mean of the RSD values for all analytes in the calibration is less than 20% (Grand Mean approach). Note: The analyst and the data user must be aware that the use of the approach listed in this Section (i.e., the average of all RSD values ≤20%) will lead to greater uncertainty of those analytes for which the RSD is greater than 20%. The analyst and the data user should review the associated quality control results carefully, with particular attention to the matrix spike and laboratory control sample results to determine if the calibration linearity poses a significant concern. If this approach is not acceptable for a particular application, then the analyst may need to adjust the instrument operating conditions and/or the calibration range until the RSD is ≤20%, or apply a regression analysis.
10.5.5.1 The mean RSD criterion applies to all analytes in the standards, regardless of whether or not they are of interest for a specific project.
10.5.5.2 The data user must be provided with either a summary of the initial calibration data or a specific list of those compounds for which the RSD exceeded 20% and the results of the mean RSD calculation.
10.5.6 If the RSD of the calibration or response factors is greater than 20% over the calibration range, then linearity through the origin cannot be assumed. The analyst may then utilize a linear regression equation that is not forced through the origin.
10.5.7 Linear regression can also be used for analytes that do meet the RSD limits in Section 10.2.6. Calibration is valid when the regression calculation generates a correlation coefficient for each compound of interest greater than or equal to 0.995.
10.5.8 If the linear correlation coefficient is <0.995, a second order curve may be used if at least 6 calibration standards are used. If the coefficient of determination of the second order curve is ≥0.990, use this second order curve for quantitation of the compound of interest. Additional concentration levels of standards **are required** for higher order curves. Refer to Method 8000 for guidance. Do not apply higher order curves without consulting the supervisor.
10.5.9 If initial calibration requirements mentioned in the above sections cannot be met, then calibration is not valid and, therefore, the analyst should check instrument operation conditions and recalibrate.
10.5.10 Analyze a reference standard from a second source (ICV) immediately following an initial calibration to verify the curve. The curve is deemed acceptable provided each analyte in the reference standard calibration is within 15% difference of the mean calibration factor or within 15% of the theoretical concentration, depending on whether quantitation will be by mean calibration factor or from regression analysis, respectively. The Grand Mean Approach may be used for the ICV except where instructed otherwise for specific clients. See Section 13.
10.5.11 Quantitation of samples must continue to be done using the procedure initially used.
10.5.12 Table 10-2 summarizes the sequence when initial calibration is performed.

Table 10-2 INITIAL CALIBRATION SEQUENCE	
Instrument Blank (hexane)	
8151 Low	Calibration Standards
8151 Med Low	
8151 Mid	
8151 Med High	
8151 High	
ICV	
Samples, not to exceed 10	
CCV	
Samples, not to exceed 10	
CCV	

Revision: 4	Status: Final	Method: 8151A	Minor Revision Date: None

10.6 Daily Calibration and Continuing Calibration Verification

10.6.1 The working calibration curve or calibration factor must be verified after initial curve at the beginning of the analytical sequence and if a lapse in time >12 hours, has occurred from last continuing standard.

10.6.2 A calibration check standard (calibration verification, continuing standard) equivalent to the mid-level calibration standard must be analyzed at the beginning of the analytical sequence and after every 10 samples, or no longer than 12 hours (8151 Mid Level Standard CCV).

10.6.3 Continuing calibration and daily calibration verifications must be judged against the quantitation method used at the start. That is, if using a curve, judge standards as acceptable if $\leq \pm 15\%$ Drift of their true value; if using the mean calibration factor, judge standards' calibration factor as acceptable if $\leq \pm 15\%$ difference against the mean calibration factor.

Calculate % Drift and % Difference using the equations below. When using a curve:

$$\%\ \text{Drift} = \frac{\text{Calculated Conc.} - \text{Theoretical Conc.}}{\text{Theoretical Conc.}} \cdot 100$$

When using mean calibration factor:

$$\%\ \text{Difference} = \frac{\text{CF Verification Standard} - \overline{CF_x}}{\overline{CF_x}} \cdot 100$$

10.6.4 If the mean calibration factor is used for quantitation, then the calibration factor of the analyte must be ≤15% difference from the mean calibration factor.

10.6.5 If the regression curve is used for quantitating analytes in samples, then the concentration of the analyte in the calibration check standard must be ≤15% drift from the theoretical concentration (i.e., 85-115% recovery).

10.6.6 If the percentage difference or drift is greater than 15% for one or more analytes, the standard is acceptable provided no one analyte %D exceeds 30% and the mean of all analytes' %D in the standard is ≤15% difference or drift (Grand Mean approach). However, the discussion in Section 10.2.7 applies.

10.6.7 Instrument blanks (hexane) are allowed before CCV to minimize chances for carryover into the CCV to avoid invalidation of the curve due to random carryover. Indication of carryover from one sample to another requires reanalysis of the potentially contaminated sample only.

10.7 Calibration Verification Corrective Action

10.7.1 If initial or continuing calibration criteria is not met, stop sample analysis immediately. Inspect the system. Minor corrective action may be performed such as changing the septum or injection port liner.

10.7.2 If after the minor corrective action a compliant CCV is analyzed, sample analysis may continue. Any samples not bracketed by valid CCVs must be reanalyzed and bracketed by compliant CCVs.

10.7.3 If minor corrective action does not fix the problem, major corrective action may be taken such as cleaning the injection port, clipping the column ends, baking out the detectors, or installing new columns. If major corrective action is taken, a new calibration curve must be run and all associated samples reanalyzed.

10.8 Sample Analysis

10.8.1 Analysis of samples should take place no later than 48 hours from esterification.

10.8.2 Once a successful initial calibration is complete, the primary analytical daily or batch sequence may begin (see Table 10-3).

Table 10-3

DAILY RUN SEQUENCE

Instrument Blank (hexane)
CCV (8151 Mid Level Standard)
Samples, not to exceed 10
CCV
Samples, not to exceed 10
CCV

Revision: 4	Status: Final	Method: 8151A	Minor Revision Date: None

10.8.3 If any analyte is detected in a sample at a level that exceeds the linear range of the calibration curve, the sample must be diluted to within the calibrated range. The dilution of samples may also be required to bring the chromatogram on scale even when no analytes of interest are detected.

10.8.4 Instrument blanks (hexane) may be interspersed throughout the analytical run to check for carryover or when the system is run unattended (autosampler).

10.9 Establishing Retention Time Windows

Make three injections of all compounds over a 72-hour period.

10.9.1 Calculate the standard deviation of the three retention times for each component.

10.9.2 The retention time window is defined as plus or minus three times the standard deviation from the retention time of the initial mid-level standards in the continuing calibration sequence.

10.9.3 Calculate retention-time windows for each analyte on each GC column whenever a new GC column is installed.

10.10 Confirmation Analysis

10.10.1 Tentative identification of an analyte occurs when a peak falls within the retention time window of a target compound on the primary column and it is at, or above, the quantitation limit for that compound. Confirmation on a secondary GC column is then required.

10.10.2 The confirmation analysis may be run simultaneously with the primary analysis but, if a separate confirmation is needed, the analysis should not exceed 40 days from date of extraction and only mid-level standard and MDL standard of compounds requiring confirmation need be analyzed.

10.10.3 An analyte is confirmed as present if it falls within the retention time window for that compound on the confirmation column as well as on the primary column.

10.10.4 The confirmation analysis is qualitative only.

11.0 DATA REDUCTION/EVALUATION/REPORTING

Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL	
		Water µg/L	Soil µg/kg
Dalapon	TSL	0.500	50
3,5-Dichlorobenzoic acid	C	0.500	33
4-Nitrophenol	TSL	0.500	50
Dicamba	TSL	0.500	33
Dichloroprop	TSL	0.500	33
2,4-D	TSL	0.500	33
Pentachlorophenol	TSL	0.500	33
Silvex	TSL	0.500	33
Chloramben	C	0.500	83
5-Hydroxydicamba	C	0.500	50
2,4,5-T	TSL	0.500	33
2,4-DB	TSL	0.500	33
Dinoseb	C	0.500	33
Bentazon	C	0.500	50
Picloram	C	0.500	90
Acifluorfen	C	0.500	33
DCPA	C	0.500	33
MCPA	TSL	50	1670
MCPP	TSL	50	1670

Revision: 4	Status: Final	Method: 8151A	Minor Revision Date: None

Key Type:

T = Compound/analyte is target compound routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike.
L = Compound/analyte is routinely used as a LCS spike.
Q = Compound/analyte is used as s surrogate spike.
NA = Not available.

11.1 QUANTITATION

11.2 Compound list and reported detection limits are as shown in Table 11-1.

11.3 Calculation of analyte concentration using mean calibration factor is as follows:

$$\text{Concentration } (\mu g/L \text{ or } \mu g/kg) = \frac{A_x}{\overline{CF}} \frac{V_t}{V_i} \frac{1}{S}$$

where:

$\overline{CF}$ = Mean calibration factor (area/ng) from initial calibration.
A_x = Area of target analyte.
Vi = Volume of extract injected (μL).
V_t = Volume of total extract (mL).
S = Sample size, volume water (L) or mass soil (kg).

11.4 Calculation of Analyte Concentration Using Regression of Linear Curve

$$\text{Concentration } (\mu g/L \text{ or } \mu g/kg) = \left[\frac{y-b}{m}\right] \frac{V_t}{S}$$

where:

y = Area of target analyte.
b = Curve intercept (area).
m = Slope of curve (area/(µg/mL).
V_t, S = As above.

11.5 Conversion of methyl esters to free acid values is automatically done by the PE Nelson Data System utilized in the GC lab for data reduction. Ester values are multiplied by the factors listed below.

Compound	Factor
Dalapon	0.911
3,5-Dichlorobenzoic acid	0.932
4-Nitrophenol	0.909
Dicamba	0.940
Dichloroprop	0.944
2,4-D	0.940
Pentrachlorophenol	0.950
Silvex	0.950
Chloramben	0.936
5-Hydroxydicamba	0.942
2,4,5-T	0.948
2,4-DB	0.947
Dinoseb	0.945
Bentazon	0.942
Picloram	0.945
Acifluorfen	0.958
DCPA	0.958
DCAA	0.936
MCPP	0.939
MCPA	0.938

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Initial Calibration Verification (ICV)	After initial curve	ICV (Table 8-2)	20% RSD (See also Section 10.5)	Identify and correct problem, recalibrate
Calibration Verification (CCV)	At start of analytical sequence	8151 Mid Calibration Standard (Table 8-2)	≤15% difference or ≤ 15% drift (See also Section 10.6)	Identify and correct problem, recalibrate
Method Blank	1 per batch of 20 or fewer samples	See Extraction SOP	Less than quantitation limit	Reanalyze samples having concentrations between PQL and 5 times PQL. Blank still above quantitation limits, reextract batch.

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1 pair per batch of 20 or fewer samples	Perform esterification of MS/MSD provided from sample preparation	- Project requirements if specified. - Compare against LCS limits for bias.	Project requirements if any, otherwise evaluate results in conjunction with other QC information (e.g., LCS recoveries) to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Continuing Standard	Every 10 samples and at end	8151 Mid Calibration Standard (Table 8-2)	≤15% difference or ≤15% drift (see also Section 10.6)	Identify problem, correct, document. Rerun samples not bracketed by acceptable standards.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples	Perform esterification of LCS provided from sample preparation	Laboratory limits	Reextract batch

Revision: 4	Status: Final	Method: 8151A	Minor Revision Date: None

Control Limits for Routine Quality Control Samples

Table 12-2

CONTROL LIMITS FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameters Charted	Type of Samples Charted	Source of Limits	Frequency Updated
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Refer to Table 12-3	Soil and water	Current E & E limits	Annually
Laboratory Control Sample (LSC)	Refer to Table 12-3	Soil and water	Current E & E limits	Annually
Surrogate (SUR)	Refer to Table 12-3	Soil and water	Current E & E limits	Annually

Revision: 4	Status: Final	Method: 8151A	Minor Revision Date: None

Table 12-3

CRITERIA ASSIGNED TO ROUTINE QC SAMPLES

QC Type	Matrix	Analytes	Nominal Spike Amount	Recovery	RPD	Source
LCS MS MSD	Water	Dalapon 3,5-Dichlorobenzoic acid* 4-Nitrophenol Dicamba Dichloroprop 2,4-D Pentachlorophenol 2,4,5-TP (Silvex) Chloramben* 5-Hydroxydicamba* 2,4,5-T 2,4-DB Dinoseb Bentazon* Picloram* Acifluorfen* DCAA (Surrogate) DCPA* MCPP MCPA	3.5 µg/L each except MCPP and MCPA at 350 µg/L (0.35 µg/mL and 35 µg/mL on column)	Table 12-1 Table 12-1 Table 12-1	-- -- Table 12-1	In-house In-house In-house
	Soil	3,5- Dichlorobenzoic acid* 4-Nitrophenol Dicamba Dichloroprop 2,4-D Pentachlorophenol 2,4,5-TP (Silvex) Chloramben* 5-Hydroxydicamba* 2,4,5-T 2,4-DB Dinoseb	117 µg/kg each except MCPP and MCPA at 11,700 µg/kg (0.35 µg/mL and 35 µg/mL on column)			

Table 12-3

CRITERIA ASSIGNED TO ROUTINE QC SAMPLES

QC Type	Matrix	Analytes	Nominal Spike Amount	Recovery	RPD	Source
		Bentazon* Picloram* Acifluorfen* DCAA (Surrogate) DCPA* MCPP MCPA				
Revision: 4	Status: Final	Method: 8151A	Minor Revision Date: None			

*Compounds not routinely reported.

13.0 SPECIAL PROJECT REQUIREMENTS

13.1 U.S. Army Corps of Engineers (ACE)

13.1.1 Method Quantitation Limit (MQL) is between 3-10 times MDL. The low calibration standard concentration must be ≥3 times MDL. The Method Reporting Limit (MRL) may slide between two times the MDL and project-specific or regulatory requirements. Results below the MRL must be reported "ND." Results between the MQL and MRL must be reported as estimated (J flagged).

13.1.2 If using the grand mean approach for RSD, no single analyte may be greater than 40% RSD.

13.1.3 If using linear regression, the correlation coefficient must be ≥0.995.

13.1.4 Initial Calibration Verification. The initial calibration curve shall be verified as accurate with a standard purchased or prepared from an independent source. This initial calibration verification (ICV) involves the analysis of a standard containing all of the target analytes, typically in the middle of the calibration range, each time the initial calibration is performed. The percent recovery of each target analyte in the ICV is determined from the initial calibration and should be between 70% and 130%.

Note: A small percentage of sporadic marginal failures (50-150% recovery) may be tolerated (i.e., will not trigger reextraction and analysis of the entire batch). The number of target analytes reported for the method will dictate the number of allowable QC failures as given below.

Number Target Analytes	Number Allowed Marginal Failures
05 – 15	1
16 – 30	2
31 – 45	3
46 – 60	4
61 – 75	5
76 – 90	6
91 – 105	7

The marginal failure allowance entails the application of an expanded acceptance criterion. If these QC criteria are not met, a new initial calibration must be performed.

13.1.5 For method blanks, check that no target compounds are found above one-half the required quantitation limit.

13.2 AFCEE - General Requirements
13.2.1 Reporting
13.2.1.2 MDL may not be > 1/2 reporting limit (RL) unless a variance was obtained.
13.2.1.3 A calibration standard at or below the RL must be included.
13.2.1.4 Apply a "U" flag if analyte result <MDL; an "F" flag if MDL ≤ analyte result ≤ RL.
13.2.2 Calibration
13.2.2.1 All initial calibration curves, inorganic or organic, must be **verified with an independent source (vendor) standard** prior to sample analysis unless an independent vendor is unavailable and a variance was obtained.
13.2.2.2 The daily (organic) and continuing (organic and inorganic) calibration verifications are not required to be independent, however, the continuing calibration verification may not be used as the LCS (volatiles, mercury, any test without a preparation).
13.2.3 Batching
13.2.3.1 An AFCEE analytical batch is the environmental samples that are similar in composition (matrix) and are prepared at the same time and with the same **lot of reagents.**
13.2.3.2 A batch is not to exceed 20 samples. **The sample duplicate, MS, MSD are considered an environmental sample, therefore, the actual number of unique samples is less than 20.**
13.2.4 Laboratory Control Sample
13.2.4.1 Laboratory Control Sample for solid must be prepared using **Ottawa sand** unless a variance was obtained.
13.2.4.2 The LCS must be prepared from second source material.
13.2.4.3 The LCS must contain all target analytes unless noted differently under specific method requirements or a variance was obtained.
13.2.4.4 The LCS must be spiked at a concentration ≤ the midpoint of the calibration curve for each analyte unless a variance was obtained.
13.2.5 Matrix Spike/Matrix Spike Duplicate
13.2.5.1 The MS and MSD must be spiked at a concentration ≤ midpoint of the calibration curve for each analyte unless a variance was obtained.
13.2.5.2 AFCEE samples must be used for spiking.
13.2.6 Surrogates (Organic analyses)
13.2.6.1 Whenever one surrogate is outside limits, corrective action must be performed.
13.2.7 When **retention time** is outside windows, perform corrective action.
13.2.8 Method blank must be less than RL unless a variance was obtained.
13.2.9 Holding times for analysis is calculated from the time of extraction completion to the time of completion of all **analytical runs including dilutions, confirmation, reanalyses.**
13.2.10 GC and HPLC Confirmation must be performed on results ≥ RL except for PCB, toxaphene and chlordane and must be quantitative.
13.2.11 AFCEE Project-Specific Method Requirements
13.3.11.1 Retention times must be updated at each initial calibration and each calibration verification unless a variance was obtained.
13.3.11.2 The AFCEE term second source calibration verification is the laboratory term initial calibration verification (ICV), run once after a curve is analyzed. Do not confuse with the AFCEE term ICV (see below).

13.3.11.3 The AFCEE term of initial calibration verification is the same as the laboratory term daily calibration check. It may not be prepared from the same material used to prepare the LCS. Use second source material for the LCS and use calibration material for the AFCEE term ICV and continuing calibration verifications.

14.0 SAMPLE AND WASTE DISPOSAL

14.1 Follow standard procedures for the disposal of all cleaning solutions and solvents.

14.2 Liquid waste with possible low level (trace) contamination can be treated in a hood with silicic acid and KOH solution to decompose any diazald or diazomethane. After sitting in the hood until the ether has evaporated, these solutions can be disposed of as wastewater in the sink in the hood with a 30-minute water flush.

15. 0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Extraction and Concentration of Water Samples for Analysis of Semivolatile Compounds			
Reviewer: G. Hahn		Peer Reviewer: J. Gilmore	
Implementation Date: 5/3/01		Last Annual Review Date: 4/23/01	
Revision: 8	Status: Final	Method: SW-846, 3510C, 3520C, 625	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedure for extraction and concentration of water samples for analysis of Base/Neutral/Acid organic compounds. The SOP applies to both separatory funnel (Method 3510C) and continuous liquid-liquid extraction (3520C). The analysis procedure is in SOP GCMS.19.

2.0 METHOD SUMMARY

2.1 A known volume (Usually 1 liter) of aqueous sample is spiked with surrogate compounds. The pH of the sample is adjusted acidic and it is extracted with methylene chloride. The aqueous portion is then pH adjusted basic and extracted again with methylene chloride. The combined extracts are concentrated.

2.2 Deviations to Method 625

2.2.1 Method 625 describes extraction at basic pH first, but the above practice of extraction at acidic pH has given improved recoveries of phenolic compounds, provides recoveries within method guidelines, and is within the scope of the method.

2.2.2 Phenolic surrogate concentrations are at 120 µg/L instead of method described 100 µg/L.

2.2.3 Water bath temperature stated in method is 60-65 C. This procedure is at 80-90 C nominally with temperature adjusted to give a proper concentration rate.

2.2.4 Method 625 describes the continuous liquid-liquid extraction for 24 hours. An 18-hour extraction has been determined to be sufficient for achieving method recoveries.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 Inspect all glassware, especially separatory funnels for damage (star imperfections or ragged edges). Do not use damaged glassware.

3.3 All work with methylene chloride should be performed in a fume hood.

3.4 Pollution Prevention – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 SW-846, 3rd Ed., Methods 3500B, 3510C, 3520C December 1996.
4.2 Method 625, 40 CFR Part 136 Appendix A.
4.3 Training SOP QA.5, A.23, and A.29.
4.4 Data Review SOP A.25.
4.5 Laboratory Quality Assurance Manual.
4.6 MDL SOP A.18.
4.7 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 Water is ASTM Type II.

5.2 See generic list in the Quality Assurance Manual.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Glassware must be scrupulously clean to avoid contamination of samples. Efforts must be made to minimize sample exposure to nitrile and latex gloves worn by laboratory staff as they are a potential source of phthalate esters being introduced into samples.

6.2 Temperatures exceeding those specified in this SOP or allowing the extract to go to dryness during concentration may cause QC failures that will result in reanalysis and possibly costly resampling.

6.3 When washing glassware after extraction, do not combine glassware from high- or medium-level extracts with standard level glassware. The dishwasher can spread the contamination. Wash high- or medium-level glassware by hand, separately from standard level glassware or throw it away if it will not come clean.

7.0 INSTRUMENTATION AND EQUIPMENT

2-L Separatory funnel with PTFE stopcock or liquid-liquid extractor with ground glass joints and 500-mL round bottom flasks
80-mm Drying funnel
Glass wool (Fisher or equivalent)
2-mL Glass vials with screw cap PTFE-lined tops
Kuderna-Danish (K-D) Apparatus:
- Concentrator tube, 10-mL graduated
- 500-mL Evaporative flask (K569261-03 or equivalent)
- Snyder Column, 3-ball macro (K-503000-0121 or equivalent)

Contrad NF or equivalent
Sparkleen or equivalent
8-oz. Jars
Gas-tight syringe or Class A pipettes
Narrow- range ($\leq$ 4 pH units) pH Test paper, Wide-range pH Test Paper Universal (Whatman 2C13991 or equivalent)
1-L Graduated cylinders
Turbovap (calibrated to 1% tolerance)
N vap
Aluminum foil
NOCHROMIX
Boiling chips, solvent extracted (10/40 mesh, Fisher or equivalent)

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard, Reagents, and Consumables

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Acetone, Ultra resi analyzed	J.T. Baker or equivalent	Neat
Hexane, Ultra resi analyzed	J.T. Baker or equivalent	Neat
Methylene Chloride ($MeCl_2$), Ultra resi analyzed	J.T. Baker or equivalent	Neat
Methanol (MeOH), Ultra resi analyzed	J.T. Baker or equivalent	Neat
Na_2SO_4	J.T. Baker or equivalent	Neat
NaOH	Fisher or equivalent	Neat
H_2SO_4	J.T. Baker or equivalent	Neat
Nitrobenzene-d5	Aldrich 26, 975-1 or equivalent	Neat, 98% pure
2-Fluorobiphenyl	Aldrich 1C, 274-1 or equivalent	Neat, 98% pure
2-Fluorophenol	Aldrich F1, 280-5 or equivalent	Neat, 98% pure
2, 4, 6-Tribromophenol	Aldrich 13, 771-5 or equivalent	Neat, 98% pure
Phenol-d6	Aldrich 17606-0 or equivalent	Neat, 98% pure
p-Terphenyl-d14	Cambridge DLM382 or equivalent	Neat, 98% pure
BN Matrix Spike	Restek #31084 or equivalent	5000 µg/mL
AP Matrix Spike	Restek #310711 or equivalent	10,000 µg/mL
ASTM Type II Water	In-House System or equivalent	Neat
SV Calibration Mix #1	Resek or equivalent (2nd source)	2000 µg/mL each compound of Table 8-1.1
SV Calibration Mix #2	Resek or equivalent (2nd source)	2000 µg/mL each compound of Table 8-1.1
SV Calibration Mix #3	Resek or equivalent (2nd source)	2000 µg/mL each compound of Table 8-1.1
SV Calibration Mix #4	Resek or equivalent (2nd source)	2000 µg/mL each compound of Table 8-1.1
SV Calibration Mix #5	Resek or equivalent (2nd source)	2000 µg/mL each compound of Table 8-1.1
SV Calibration Mix #7	Resek or equivalent (2nd source)	2000 µg/mL each compound of Table 8-1.1
3,3-Dichlorobenzidine Stock	Resek or equivalent (2nd source)	2000 µg/mL
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Table 8-1.1

COMPONENTS OF SV CALIBRATION MIXES

SV Calibration Mix 1	SV Calibration Mix 2	SV Calibration Mix 3
Benzyl alcohol	Benzoic acid	bis(2-Chloroethoxy)methane
4-Chloroaniline	4-Chloro-3-methylphenol	bis(2-Chloroethyl)ether
2-Nitroaniline	2-Chlorophenol	bis(2-Chloroisopropyl)ether
3-Nitroaniline	2,4-Dichlorophenol	bis(2-Ethylhexyl)ether
4-Nitroaniline	2,4-Dimethylphenol	4-Bromophenyl phenyl ether
	2,4-Dinitrophenol	Butyl benzyl phthalate
	2-Methyl-4,6-dinitrophenol	2-Chloronaphthalene
	2-Methylphenol	4-Chlorophenyl phenyl ether
	4-Methylphenol	Dimethyl phthalate
	2-nitrophenol	di-n-Butyl phthalate
	4-Nitrophenol	di-n-Octyl phthalate
	Pentachlorophenol	N-nitrosodimethylamine
	Phenol	N-nitroso-di-n-propylamine
	2,4,5-Trichlorophenol	N-nitrosodiphenylamine
	2,4,6-Trichlorophenol	
SV Calibration Mix 4	**SV Calibration Mix 5**	**SV Calibration Mix 7**
Carbazole	Acenaphthene	1,2-Dichlorobenzene
Dibenzofuran	Acenaphthylene	1,3-Dichlorobenzene
Diethyl phthalate	Anthracene	1,4-Dichlorobenzene
2,4-Dinitrotoluene	Benzo(a)anthracene	
2,6-Dinitrotoluene	Benzo(a)pyrene	
Hexachlorobenzene	Benzo(a)fluoranthene	
Hexachlorobutadiene	Benzo(k)fluoranthene	
Hexachlorocyclopentadiene	Benzo(ghi)perylene	
Hexachloroethane	Chrysene	
Isophorone	Dibenzo(a,h)anthracene	
2-Methylnaphthalene	Fluoranthene	
Nitrobenzene	Fluorene	
1,2,4-Trichlorobenzene	Indeno(1,2,3-cd)pyrene	
	Naphthalene	
	Phenanthrene	
	Pyrene	

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8.2 Standard Solution Preparation

8.2.1 Preparation of each individual surrogate stock solution is arbitrary provided the final concentration of "SW-846 BNA Surrogate" is prepared to the concentration specified. If the recipe noted in Table 8-2 is changed, all calculations must be recorded and peer reviewed in the Standard Preparation Logbook.

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Added to ____	Final Concentration	Storage Expiration
Soap	Contrad NF	2 mL to 100 mL ASTM Type II water	2% v/v	Indefinite
10 N NaOH	NaOH	400 g to 1000 mL ASTM Type II water	10 N	1 year
1:1 H_2SO_4	H_2SO_4	500 mL H_2SO_4 concentrate to 500 mL ASTM Type II water	1:1 v/v	1 year
Nitrobenzene-d5 Stock	Nitrobenzene-d5	1.000 g into 250 mL MeOH	4.00 mg/mL	2-6 C for 1 year
p-Terphenyl-d14 Stock	p-Terphenyl-d14	0.250 g into 250 mL $MeCl_2$*	1.00 mg/mL	2-6 C for 1 year
Phenol-d6 Stock	Phenol-d6	1.000 g into 250 mL MeOH	4.00 mg/mL	2-6 C for 1 year
2,4,6-Tribromophenol Stock	2,4,6-Tribromophenol	1.000 g into 250 mL MeOH	4.00 mg/mL	2-6 C for 1 year
2-Fluorophenol Stock	2-Fluorophenol	2.00 g into 250 mL MeOH	8.00 mg/mL	2-6 C for 1 year
2-Fluorobiphenyl Stock	2-Fluorobiphenyl	1.000 g into 250 mL MeOH	4.00 mg/mL	2-6 C for 1 year
SW-846 BNA Surrogate	Nitrobenzene-d5 Stock	2.5 mL plus	50 µg/mL	2-6 C for 3 months
	p-Terphenyl-d14 Stock	10 mL plus	50 µg/mL	
	2-Fluorobiphenyl Stock	2.5 mL plus	50 µg/mL	
	Phenol-d6 Stock	3 mL plus	60 µg/mL	
	2, 4, 6-Tribromophenol Stock	3 mL plus	60 µg/mL	
	2-Fluorophenol	1.5 mL plus All diluted to 200 mL with methanol	60 µg/mL	

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Added to ____	Final Concentration	Storage Expiration
SW-846, BNA Spike	BN Matrix Spike AP Matrix Spike	300 µL plus 150 µL to 50 mL methanol	30 µg/mL 30 µg/mL	2-6 C for 3 months
AFCEE Spike A	SV Calibration Mix #1 SV Calibration Mix #2 SV Calibration Mix #3 SV Calibration Mix #5 SV Calibration Mix #7	300 µL 300 µL 300 µL 300 µL 300 µL Added in 20-mL volumetric flask and brought to volume with methanol	30 µg/mL for all components	2-6 C for 3 months
AFCEE Spike B	SV Calibration Mix #4 3,3-Dichlorobenzidine	300 µL 300 µL Added in 20-mL volumetric flask and brought to volume with methanol	30 µg/mL for all components	2-6 C for 3 months

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*Unable to dissolve in MeOH.

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Water	Standard	7 days from collection	40 from extraction	½ gallon or 1-L Glass Amber 2-6 C
	NYSDEC	5 days VTSR		

Revision: 8	Status: Final	Method: SW-846, 3510C, 3520C, 625	Minor Revision Date: None

10.0 PROCEDURE

10.1 Glassware Preparation

10.1.1 All glassware shall be manually washed in hot soapy tap water using Sparkleen or equivalent, or automatically washed using Contrad NF.

10.1.2 At a minimum, manually washed glassware shall be rinsed three times with tap water, followed by 3 times with ASTM Type II water.

10.1.3 If glassware still appears dirty, it shall be subjected to sonication, or cleaned with NOCHROMIX. Then it shall be rerun through the above cleaning procedure.

10.2 Preventing Contamination

10.2.1 Before use, rinse all glassware with acetone followed by methylene chloride.

10.2.2 Dry sodium sulfate (Na_2SO_4) (recommended 120 C, overnight or longer). Rinse with methylene chloride before use.

10.3 Stock Solution Preparation

10.3.1 Prepare stock standard surrogate solutions in methanol or methylene chloride and using volumetric flasks and an analytical balance.

10.4 Sample Extraction Procedure

Each sample setup consists of a separatory funnel or liquid-liquid extraction set, drying funnel, and a 16-oz. jar or equivalent. Set up the Waters Preparation Logbook to include the following information:

- Extraction date.
- Analyst signature.
- Work order number.
- Sample identification.
- Sample volume.
- Sample pH.
- Final volume of base/neutral extract after concentration.
- Final volume of acid phenol extract after concentration.
- A witness must oversee the addition of all surrogates and spikes to sample and sign in the logbook that this was done.
- Record the solvent(s) used in the extraction logbook.
- Date of concentration and analyst signature.

10.4.1 Sample Extraction by Separatory Funnel (Method 3510C)

10.4.1.1 To prepare the drying funnels, add Whatman 54 filter paper and approximately 50 g of dried sodium sulfate (dried 120 C, overnight or longer) to an 80-mm glass funnel.

10.4.1.2 The funnel is rinsed with methylene chloride, and the rinse solvent discarded.

10.4.1.3 If 1 L or less of sample is provided, measure the volume by pouring into a 1-L graduated cylinder. Record the volume to the nearest 5 mL in the extraction logbook as "volume extracted." Transfer the sample to a 2-L separatory funnel. If sample volume is less than 800 mL, add enough ASTM Type II water to bring the volume of sample plus water to 1 L in the separatory funnel.

10.4.1.4 If more than 1 L of sample is provided, measure 1 L into a 1-L graduated cylinder. Record the volume of the sample to the nearest 5 mL. Transfer sample into separatory funnel. Always use a clean graduated cylinder for each sample.

10.4.1.5 Measure pH using wide-range pH indicator strips. Record. Adjust pH to 2 using 1:1 sulfuric acid. **Do not overacidify**. Verify with narrow range pH indicator strips. Record pH adjustment and lot numbers of acid. (Note: In general, blanks, spikes, and LCSs will require less acid than samples because of lack of buffer capacity.)
10.4.1.6 Add 1.0 mL of SW-846 BNA surrogate solution to each sample and QC (method blank, MS, MSD, LCS) sample using a syringe or volumetric pipette. See Table 12-1 for addition of other spiking solutions. Swirl the separatory funnel to mix.
10.4.1.7 Rinse the sample bottle or the graduated cylinder with 10-20 mL of the extraction solvent and add to the separatory funnel.
10.4.1.8 If requested by client or if insufficient quantity exists for the preparation of MS/MSD, add surrogate and spike to second 1-L volume of ASTM Type II water. The sample is designated as a Laboratory Control Sample duplicate (LCSD). (Note: Do not prepare LCSD for AFCEE projects unless specifically instructed to do so.)
10.4.1.9 Add 60 mL methylene chloride to samples in separatory funnels.
10.4.1.10 Shake for two minutes with frequent venting. Caution: A vapor build-up will occur.
10.4.1.11 Let stand 10 minutes.
10.4.1.12 If an emulsion forms of greater than one-third of the total methylene chloride portion, transfer the entire emulsion to 40-mL centrifuge tubes. Centrifuge until the emulsion breaks, and decant the water layer back to the separatory funnel.
10.4.1.13 Collect the methylene chloride layer from the separatory funnel and centrifuge tubes, if used, into the jar through the drying funnel. Return the aqueous portion to the separatory funnel. The drying funnel is rinsed with clean methylene chloride after the collection of this first and last fraction.
10.4.1.14 Repeat from Section 10.4.1.8 for two additional times.
10.4.1.15 Adjust the pH to >11 of the aqueous portion using 10 N sodium hydroxide. Test using narrow range pH indicator paper.
10.4.1.16 Repeat the procedure of sections 10.4.1.8 to 10.4.1.13 using the same drying funnel and jar.
10.4.1.17 Cover the jar with foil and label. This is the BNA extract.

10.4.2 Sample Extraction Procedure – Continuous Liquid-Liquid (Method 3520C)
10.4.2.1 Add (approximately 500 mL) methylene chloride to the bottom of the extractor to a depth of at least one inch (1") above the bottom of side arm.
10.4.2.2 If 1 L or less of sample is provided, measure the volume by pouring into a 1-L graduated cylinder. Record the volume to the nearest 5 mL in the extraction logbook as "volume extracted."
10.4.2.3 If more than 1 L of sample is provided, measure 1L into a 1-L graduated cylinder. Record the volume of the sample to the nearest 5 mL.
10.4.2.4 Prepare quality control samples (matrix spike, matrix spike duplicate, method blank, and LCS as described in the Table 12-1. Check the initial pH of all samples with wide range pH paper and record in the logbook. (Add 1.0 mL of SW-846 BNA surrogate to each sample, blank, matrix spike, matrix spike duplicate, and LCS.) **Spiking is done in the graduated cylinder or bottle and must be witnessed.**
10.4.2.5 Adjust pH to 2 with 1:1 sulfuric acid. **Do not over acidify.** Blanks, LCS, and spikes will require less acid than samples. Verify with narrow range pH paper.
10.4.2.6 Transfer to a labeled extraction chamber. Take care that the sample does not spill over the side arm into the distilling flask. If it does, repeat the sample preparation. (Note: A layer of glass wool may be placed between the methylene chloride and sample if the sample contains a large amount of suspended solids that may precipitate.)
10.4.2.7 Rinse the bottle or graduated cylinder with 10-20 mL of extraction solvent and add to extractor.

10.4.2.8 Attach condenser, turn on heating mantle, voltage regulator, and extract for a minimum of 18 hours. Maintain methlyene chloride drip rate at 10-15 mL per minute. Turn water on to an appropriate pressure.
10.4.2.9 Allow to cool.
10.4.2.10 Attach another distilling flask and add approximately 250 mL of methylene chloride to the chamber. Adjust the aqueous phase pH to >11 using 10 N sodium hydroxide. Extract for 18 hours.
10.4.2.11 Turn the condenser water off. Prior to concentration, extracts must be poured through drying funnel containing sodium sulfate.
10.4.2.12 Enter the sample preparation end date and complete the analyst review section of the data review checklist.

10.5 Concentration Procedure
10.5.1 Kuderna-Danish Apparatus
10.5.1.1 Boiling chips are pre-rinsed with methylene chloride.
10.5.1.2 Assemble a Kuderna-Danish (K-D) apparatus by attaching a 10-mL concentrator tube containing a single boiling chip to a 500-mL evaporative flask.
10.5.1.3 Quantitatively transfer the extracts from the distilling flasks to the K-D; rinsing the jar with 10-20 mL methylene chloride. Use a 80-mm funnel with Whatman 54 filter paper and 15-20 g of dried sodium sulfate.
10.5.1.4 Attach a 3-ball Snyder column and pre-wet by adding 1-2 mL methylene chloride. Place on a hot water (80-90 C) bath and gently shake until bubbles appear. Adjust water temperature and vertical position to complete concentration in 15-20 minutes.
10.5.1.5 Concentrate to a volume of <10 mL. Remove from steambath. Allow it to settle and cool (at least 10 minutes) to room temperature. At no time should the extract be allowed to go to dryness throughout the concentration procedure. If it does, reextract the sample.
10.5.1.6 If during concentration, solvent vapor stops escaping from the Snyder column before the final volume is reached, this indicates that the extract contains large concentrations of high-boiling constituents. Concentration should be stopped and the extract brought to the nearest multiple of 5 mL final volume. Final volume should be noted accordingly in the Waters Preparation logbook.
10.5.1.7 Dry the outside of the joint between the concentrator tube and the K-D if condensation has formed. Remove concentrator tube, and provided large concentrations of high boiling constituents is not a problem, the sample can be further reduced.
10.5.1.8 Place concentrator tube in N-Evap water bath at 40 C. Using a gentle stream of nitrogen, blow the extract down to 1.0 mL. If necessary, adjust final volume to 1.0 mL with methylene chloride. (Note: The measurement of final volume is critical to the accuracy of the analysis.)
10.5.1.9 Place the BNA extracts in 2-mL vials with PFTE-lined caps and mark the meniscus. The marking ensures that the extract in the vial represents the final volume transferred and will indicate that no loss has occurred in storage. The vials are labeled, final volume recorded in the waters preparation notebook.
10.5.1.10 Complete the batch in Khemia, fill out the concentration portion of the checklist and arrange for a peer review of the data. Place a copy of the extraction batch and checklist in the notebook in the instrument lab.
10.5.1.11 The entire extraction batch is placed in a storage box in the appropriate secure storage area at <6 C.

10.5.2 Turbovap Concentration
10.5.2.1 Quantitatively transfer the sample from the jar to Zymark Turbovap tube as volume permits. Use a 80-mm funnel with Whatman 54 filter paper and 15-20 g of dried sodium sulfate.

10.5.2.2 Place the tube in the Turbovap, set at 40 C (acceptable working range is from 38 C to 45 C) with a gentle stream of nitrogen. Concentrate to 1.0 mL (or nearest 5 mL if evaporation/concentration cannot be brought down to 1 mL).

10.5.2.3 Record the final volume in the notebook. Transfer the concentrated extracs to a 2-mL vial with PTFE-lined cap. Mark the meniscus. Label the vial. (Note: The measurement of final volume is critical to accuracy of the analysis.)

10.5.2.4 Complete the batch in Khemia, fill out the concentration portion of the checklist, and arrange for a peer review of the data. Place a copy of the extraction batch and checklist in the notebook in the instrument lab.

10.5.2.5 Place the entire extraction batch, in a single storage box, into the appropriate secure storage area at <6 C.

Instrument Maintenance

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment Instrument	Symptom	Operation	Frequency
Steambath	Not heating	Check water level, replace heating coils	As necessary
Fumehood	Not drawing	Notify Supervisor or Lab Manager	As necessary
Revision: 8	Status: Final	Method: SW-846, 3510C, 3520C, 625	Minor Revision Date: None

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 Issue a corrective action form for out-of-control events.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

12.1 Refer to the Quality Control table below and the following sections for corrective actions.

12.2 One in 20 samples or fewer are to be spiked and duplicate spiked. If not done in duplicate, analyst must verify and document that insufficient sample exists for a duplicate spike. File a corrective action report if insufficient samples exists.

Routine Quality Control Samples

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions
BNA Surrogate	Each blank, sample and spikes	1.0 mL of SW-846 BNA surrogate added to the separatory funnel of each sample, blank and QC sample prior to pH adjustment.
BNA Spike Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1 MS/MSD pair per 20 or fewer samples	1.0 mL of SW-846 BNA spike solution to 1- L sample.

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions
LCS (Representative List)	1 per extraction batch	1.0 mL of SW-846 BNA spike solution added to 1 L with ASTM Type II water.
LCS (Full List A)	1 per extraction batch	1.0 mL AFCEE Spike A added to 1 L of ASTM Type II water.
LCS (Full List B)	1 per extraction batch	1.0 mL AFCEE Spike B added to 1 L of ASTM Type II water.
LCS (Full List AB)	1 per extraction batch	1.0 mL each of AFCEE Spikes A and B added to 1 L of ASTM Type II water.
Method Blank	1 per 20 or fewer samples	1 L of ASTM Type II water processed as a sample.
Revision: 8 Status: Final	Method: SW-846, 3510C, 3520C, 625	Minor Revision Date: None

13.0 SPECIAL PROJECT REQUIREMENTS

13.1 Buffalo Color

13.1.1 Due to high levels of aniline in the sample, extract this sample in a separate batch. Do not combine the BNA and AP extracts. Use 1.0 mL of spike and surrogate to compensate for the 2.0 mL final volume.

13.2 AFCEE

13.2.1 Batching

13.2.1.1 An AFCEE analytical batch is the environmental samples that are similar in composition (matrix) and are prepared at the same time and with the same **lot of reagents.**

13.2.1.2 A batch is not to exceed 20 samples. **The sample duplicate, MS, MSD are considered an environmental sample, therefore, the actual number of unique samples is less than 20.**

13.2.2 Laboratory Control Sample

13.2.2.1 Two LCS, each consisting of a subset of compounds, are prepared and analyzed to avoid chemical interactions between compounds present in a single full list LCS. The two aqueous LCS together must contain all target analytes unless noted differently under specific method requirements or a variance was obtained. LCS A shall contain all target analytes from SV Calibration Mixtures 1-3, 5, and 7 and LCS B shall contain all target analytes from SV Calibration Mixture 4 and 3,3-dichlorobenzidine.

13.2.2.2 The LCS must be spiked at a concentration ≤ midpoint of the calibration curve for each analyte unless a variance was obtained.

13.3.3 Matrix Spike/Matrix Spike Duplicate

13.3.3.1 The MS and MSD must be spiked at a concentration ≤ midpoint of the calibration curve for each analyte unless a variance was obtained.

13.3.3.2 AFCEE samples must be used for spiking.

14.0 SAMPLE DISPOSAL

14.1 See SOP A.10, Waste Disposal.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Pesticide/PCB Extraction and Concentration of Soil Samples by Method 3550B			
Reviewer: B. Kruszka		Peer Reviewer: J. Gilmore	
Implementation Date: 10/11/99		Last Annual Review Date: 4/17/01	
Revision: 5	Status: Final	Method: SW-846, 3550B	Minor Revision Date: 6/7/01

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the sample preparation procedure for extraction and concentration of organochlorine pesticides and PCB in soil and solid waste samples. Samples may be prepared for pesticide analysis only SOP GC.72 (Method 8081A), PCB analysis only SOP GC.73 (Method 8082) or both pesticide and PCB analysis GC.14 (Method 8081).

2.0 METHOD SUMMARY

2.1 Samples are mixed with sodium sulfate (drying agent), surrogate spiking solution is added, sonicated three times with solvent and the solvent decanted. The extract is concentrated and solvent exchanged to hexane using a Kuderna-Danish apparatus on a steam bath.

2.2 Alternative PCB spiking information is provided should PCBs be the only target analytes. This PCB spiking information can be used to supplement the normal Pesticide Spike if requested.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E Inc., Analytical Services Center Chemical Hygiene Plan, located in the QA Library Island shelf 2.

3.2 All glassware should be inspected for flaws or ragged edges before use. Do not use damaged glassware.

3.1 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Method 3550B, (Rev. 2, 12/96), SW-846 Update III, June 1997.
4.2 Analysis SOPs GC.14, GC.72, GC.73.
4.3 Use checklist C-048 for analyst and peer review.
4.4 Training SOP QA.5, A.23, and A.29.
4.5 Data Review SOP A.25.
4.6 Laboratory Quality Assurance Manual (L-001).
4.7 MDL SOP A.18.
4.8 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 **Batch** – A group of samples that behave similarly with respect to the sampling or testing procedures being employed which are processed as a unit. If the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

5.2 Method Blank (MB) – An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is used to document contamination resulting from the analytical process.

5.3 Matrix – The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

5.4 Matrix Spike (MS) – An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

5.5 DCB, TCMX – Decachlorobiphenyl and tetrachlorometaxylene, respectively; all are method surrogates.

5.6 Matrix Spike Duplicate /MSD – An intra-laboratory split sample spiked with identical concentrations of analytes. They are used to document the precision and bias of a method in a given sample matrix.

5.7 Laboratory Control Sample/LCS – A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1.1 Phthalates may cause false positives. Plasticware may not be used. Latex gloves must not contact inside of glassware.

7.0 INSTRUMENTATION AND EQUIPMENT

Ultrasonic Disruptor with ¾" Titanium Horn
Sonabox
400-mL Thick-walled beakers
100-mm Drying funnel
Glass wool (Fisher or equivalent)
Filter paper (Whatman 41 or equivalent)
Kuderna-Danish Apparatus (K-D)
- Concentrator tube, 10-mL graduated
- 500-mL Evaporation flask (Kontes K-507001-500 or equivalent)
- Snyder column, micro (569261-031 or equivalent)
- Snyder column, 3-ball macro (Kontes K-503000-0121 or equivalent)

Boiling chips, 10/40 mesh, Fisher or equivalent
Water bath heated with concentric ring cover
Balance, top-loading (readable to .001g or better)
15-mL Glass vials with screw cap PTFE-lined tops
Benchtop centrifuge (Fisher or equivalent)
Spatula, stainless steel
1.0-mL pipettes or syringe
16-oz. Jars
Stainless steel spoonula
Sparkleen (or equivalent)
Contrad NF
NOCHROMIX
Zymark Turbovap
Turbovap tubes

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard, Reagents, and Consumables

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration (µg/mL)	
Methylene chloride ($MeCl_2$)	J.T. Baker Ultra Resi-analyzed or equivalent	Neat	
Acetone	J.T. Baker Ultra Resi-analyzed or equivalent	Neat	
Hexane	J.T. Baker Ultra Resi-analyzed or equivalent	Neat	
Sodium sulfate, Na_2SO_4 (anhydrous)	J.T. Baker or equivalent	Neat, stored at 120 C	
ASTM Type II water	In-house Laboratory System	Neat	
Pesticide Matrix Spike Mix	Restek or equivalent	alpha-BHC	200
		beta-BHC	200
		gamma-BHC	200
		delta-BHC	200
		Heptachlor	200
		Aldrin	200
		Heptachlor epoxide	200
		alpha-chlordane	200
		gamma-chlordane	200
		Endosulfan I	200
		4,4-DDE	200
		Dieldrin	200
		Endrin	200
		Endosulfan II	200
		4,4-DDD	200
		Endrin aldehyde	200
		4,4-DDT	200
		Endosulfan sulfate	200
		Methoxychlor	200
Pesticide Surrogate Mix	Restek or equivalent	200 µg/mL TCMX and DCB	
Aroclor 1016/1260	Restek or equivalent	1000 µg/mL each	
Toxaphene	Restek or equivalent	1000 µg/mL	
Methanol	J.T. Baker Ultra Resi-analyzed or equivalent	Neat	
Methoxychlor	Restek or equivalent	1000 µg/mL	
Sand	Ottawa sand or equivalent	NA	

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8.2 Standard Solution Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	_____ Amount of Stock Diluted to _____	Final Concentration
AR1660 PCB Spike	Aroclor 1016/1260	250 µL to 50 mL with acetone	5 µg/mL
8081A Pestcide/ PCB Surrogate	Pesticide Surrogate Mix	100 µL Pesticide Surrogate Mix to 100 mL with acetone	0.2 µg/mL TCMX and DCB
8081A Pesticide Spike	Pesticide Matrix Spike Mix	50 µL Pesticide Matrix Spike Mix plus 90 µL methoxychlor diluted to 100 mL with methanol	0.1 µg/mL for all compounds except methoxychlor at 1.0 µg/mL
Toxaphene Spike	Toxaphene 1000 µg/mL	200 µL diluted with acetone to 50 mL	4 µg/mL
10% HCl	HCl	10 mL to 100 mL ASTM Type II water	10 % (v/v)
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9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Martrix	Client/ Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Solid	Standard	14-day collection	40 days from extraction	4-, 8-, or 16-oz. glass jar. Store at 2-6 C.
Solid	NYSDEC	5 days from receipt (VSTR)	40 days from extraction	4-, 8-, or 16-oz. glass jar. Store at 2-6 C.
Revision: 5	Status: Final	Method: SW-846, 3550B	Minor Revision Date: 6/7/01	

10.0 PROCEDURE

10.1 Glassware Preparation

10.1.1 All glassware shall be immediately rinsed after use with the last solvent used, manually washed in hot soapy tap water using Sparkleen, or automatically washed using Contrad NF.

10.1.2 At a minimum, manually washed glassware shall be tap water rinsed, followed by 3 rinses with ASTM Type II water.

10.1.3 If glassware still appears dirty, it shall be subjected to sonication, or cleaning with NOCHROMIX. Then it shall be rerun through the above cleaning procedure.

10.1.4 When washing glassware after extraction, do not combine glassware from high or medium level extracts with standard level glassware. The dishwasher can spread the contamination. Wash glassware by hand separately from standard level glassware or throw it away if it will not come clean.

10.2 Preventing Contamination

10.2.1 Before use, all glassware is solvent rinsed with acetone, followed by methylene chloride.

10.2.2 Sodium sulfate is dried (recommended 120 C, overnight or longer).

10.2.3 Rinse dried sodium sulfate with methylene chloride before use.

10.3 Sample Setup

10.3.1 A batch of samples consists of a method blank, matrix spike (MS), matrix spike duplicate (MSD), and laboratory control sample (LCS) for every 20 or fewer samples. A laboratory control sample duplicate shall be used if insufficient sample is provided for an MSD. If both Pesticide and PCB extraction are required, allow three extra extraction setups for separate LCS, MS, and MSD.

10.3.2 Each sample set up consists of one 400-mL beaker, one drying funnel, one 16-oz. jar, and one spoonula. Set up the soil preparation logbook as follows.

10.3.2.1 Extraction date.

10.3.2.2 Analyst name.

10.3.2.3 Method/test (SOP numbers with revision number).

10.3.2.4 Job number.

10.3.2.5 Sample number.

10.3.2.6 Sample weight.

10.3.2.7 Solvent used.

10.3.2.8 Exchange solvent.

10.3.2.9 Final volume.

10.3.2.10 Date of concentration and analyst initials.

10.3.2.11 Reference to balance calibration information and batch number.

10.4 Extraction

10.4.1 Weigh 25-35 g to the nearest 0.1 g of well-homogenized soil into a 400-mL beaker. Use sand for Method Blank and LCS. Record the weight to the nearest 0.1 g. Add 20-60 g sodium sulfate and mix with a spoonula until the sample is free-flowing. Add more sodium sulfate if necessary.

10.4.2 Add 1.0 mL of 8081A Pesticide Surrogate/PCB Surrogate Solution to each sample and QC sample (blank, MS, MSD, LCS) using a 1.0-mL syringe or volumetric pipette.

10.4.3 Pesticide Spiking Instructions:

10.4.3.1 If pesticide extraction is assigned to the sample do the following, otherwise proceed to PCB Spiking Instructions:

10.4.3.2 Add 1.0 mL of 8081A Pesticide Spike Solution to the pesticide matrix spike, pesticide matrix spike duplicate, and pesticide laboratory control sample (see Table 12-1).

10.4.4 PCB Spiking Instructions:

10.4.4.1 If PCB extraction is assigned to the sample a PCB MS, MSD, and LCS must be prepared. DO NOT spike the sample aliquot with both pesticide and PCB spikes.

10.4.4.2 Add 1.0 mL AR1660 PCB Spike to the PCB matrix spike, PCB matrix spike duplicate and PCB laboratory control sample (see Table 12-1).

10.4.3 Prepare a drying funnel by adding a plug of glass wool and approximately 15-20 g of sodium sulfate to the drying funnel. Rinse the funnel with methylene chloride and discard the rinse solvent.

10.4.4 Add 100 mL of 1:1 methylene chloride: acetone to each sample.

10.4.5 Place the bottom surface of the sonicator tip approximately 1/2 inch below the surface of the solvent but above the sediment layer. Do not use the microtip probe.

10.4.6 Sonicate for 3 minutes at an output of 10, the mode switch on Pulse and the duty cycle at 50%.

10.4.7 Decant the extract through the rinsed drying funnel into a labeled clean 16-oz. jar. Rinse down the drying funnel at a minimum after the first and final sonications.
10.4.8 Repeat the extraction two more times with (2) 100 mL volumes of 1:1 methylene chloride: acetone. Combine extracts.
10.4.9 Cover the 16-oz. jar with foil.
10.4.10 Clean the horn of the sonicator between samples rinsing with ASTM Type II water. Wipe the horn and rinse with acetone, then soapy water, methylene chloride, and (Sparkleen or Alconox).
10.4.11 Rinse with ASTM Type II water and wipe the horn. Rinse with acetone, then methylene chloride.
10.4.12 Enter Prep End Date into LIMS after batch is fully sonicated.

10.5 K-D Concentration Procedure
10.5.1 Assemble a Kuderna-Danish (K-D) apparatus by attaching a 10-mL concentrator tube containing a boiling chip to a 500-mL evaporative flask.
10.5.2 Quantitatively transfer the extract to the K-D apparatus, rinsing the container with methylene chloride to complete the transfer.
10.5.3 Attach a 3-ball Snyder column and pre-wet with methylene chloride. Place on a hot water bath (80-90 C) so that the concentrator tube is partially immersed in the hot water and the lower rounded surface of the evaporative flask is bathed with hot water vapor. Expose as much surface area of the flask to the vapor as possible.
10.5.4 Adjust the vertical position of the apparatus and the water temperature to complete the concentration in 10-15 minutes.
10.5.5 Concentrate to an approximate volume of 4 mL. Allow the extract to cool and drain for at least 10 minutes. At no time should the extract be allowed to go to dryness throughout the concentration procedure. Should the extract go to dryness, restart the extraction from the beginning if sample is available. Otherwise, clearly note in extraction log and file a Corrective Action Form notifying the Project Manager and Supervisor.

10.6 K-D Solvent Exchange
10.6.1 After cooling, momentarily remove Snyder Column and add approximately 50 mL of hexane and a fresh boiling chip. Replace column and continue concentration.
10.6.2 Place the apparatus on the steam bath to complete the concentration in 5 to 10 minutes.
10.6.3 When the apparent volume of liquid reaches approximately 4 mL, remove the K-D apparatus from the bath and allow the K-D apparatus to drain and cool for approximately 10 min (or longer). Adjust the extract volume to 10 mL with hexane.
10.6.4 If during concentration solvent vapor stops escaping from Snyder column before the final volume is reached, the extract contains large concentrations of higher boiling constituents. Concentration should be stopped and the extract brought to the next higher 5-mL level. Final volume must be noted in the soils preparation logbook.
10.6.5 If a significant amount of sediment appears in the concentrated extract, it should be centrifuged for 5 minutes. Decant the solvent into a 10-mL concentration tube and bring up to 10 mL final volume with hexane. Note this step in the logbook.
10.6.6 Transfer approximately 2 mL of extract into two, 1-mL screwtop vials with PFTE caps.
10.6.7 Enter Concentration End Date into LIMS after all samples are complete.
10.6.8 Have batch peer reviewed in LIMS and logbook.

10.7 Turbovap Concentration Procedure (alternative to K-D procedure)
10.7.1 Quantitatively transfer sample from jar to Zymark Turbovap tube rinsing with 20-30 mL methylene chloride to complete the transfer.

10.7.2 Place in Turbovap, set at 38 C to 45 C with a gentle stream of nitrogen. Concentrate to 1 mL (or nearest 5 mL if evaporation/concentration can not be brought down to 1 mL).
10.7.3 Enter Concentration End Date into LIMS after all samples are complete.
10.7.4 Have batch peer reviewed in LIMS and logbook.

10.8 Turbovap Solvent Exchange Procedure
10.8.1 Add a minimum of 10 mL hexane to sample in Turbovap tube.
10.8.2 Place in Turbovap, set at 38 C to 45 C with a gentle stream of nitrogen. Concentrate to 1 mL (or nearest 5 mL if evaporation/concentration cannot be brought down to 1 mL).
10.8.3 Bring to 10 mL with hexane. Record the final volume in the notebook.
10.8.4 Add approximately 1g activated copper to one of the 1-mL vials and vortex for 15 minutes. If no reaction, remove from vortex, if there is a reaction (dark or cloudy precipitate) vortex for an additional 15 minutes.
10.8.4.1 If sample is for pesticide only, stop and deliver entire batch of extracts to the instrument lab.
10.8.4.2 If samples are for PCB only, then add 250 µL of sulfuric acid to vial and vortex for 1 minute.
10.8.4.3 If samples are for pesticide and PCB, transfer one-half of the copper-treated extract into an additional screwtop vial and add 250 µL of sulfuric acid to the additional vial only. Vortex for 1 minute.
10.8.4.4 Enter Concentration End Date into LIMS after all samples are complete.
10.8.4.5 Have batch peer reviewed in LIMS and logbook.
10.8.4.6 Deliver the entire batch of extracts to the instrument lab.

10.9 Copper Technique – For removal of sulfur from sample extracts.
10.9.1 Sulfur removal is required for all sample extracts that contain sulfur.
10.9.2 Add approximately 1-2 g of clean copper powder to the extract in the 2-mL vial. Mix the copper and extract for approximately 15-30 minutes on a mechanical shaker.
10.9.3 Prior to analysis, separate the extract from the copper powder by drawing off the extract with a disposable pipette, and transfer the extract to a clean vial. If the copper appears bright, samples delivered for analysis. If the copper changed color, repeat the sulfur removal procedures as necessary (activity performed by GC Analyst).

Instrument Maintenance

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Sonicator	Not disrupting sample	Check power/see Senior Scientist before adjusting	As necessary
Water Bath	Not heating	Replace coils	As necessary
Revision: 5	Status: Final	Method: SW-846, 3550B	Minor Revision Date: 6/7/01

11.0 DATA REDUCTION/EVALUATION/REPORTING

NA

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions
Method Blank (MB)	One per 20 or fewer samples prepared in a batch	Approximately 30 g of sand.
Pesticide Matrix Spike (MS)	One per 20 or fewer samples prepared in a batch	Add 1.0 mL of 8081A Pesticide Spike Solution to 30 g sample. Resulting concentration 3.33 µg/kg each compound except methoxychlor at 33.3 µg/kg.
Pesticide Matrix Spike Duplicate (MSD)	With every matrix spike for pesticide	Prepare as the matrix spike.
Pesticide Laboratory Control Sample (LCS)	One per 20 or fewer samples prepared in a batch	Add 1.0 mL of a Pesticide Spike Solution to 30 g sand. Resulting concentration 3.33 µg/kg each compound except methoxychlor at 33.3 µg/kg.
Surrogate	To each sample and QC sample (blank, MS, MSD, LCS)	Add 1.0 mL of 8081A Pesticide/PCB Surrogate Solution. Resulting concentration DCB, TCMX each 6.67 µg/kg.
PCB Matrix Spike (MS)	One per 20 or fewer samples in a batch requiring PCB QC	1.0 mL AR1660 PCB Spike to 30 g sample. Resulting concentration 167 µg/kg each.
PCB Matrix Spike Duplicate (MSD)	With every PCB MS	Same as Matrix Spike.
PCB Laboratory Control Sample (LCS)	One per 20 or fewer samples in a batch for PCB	1.0 mL AR1660 PCB Spike to 30 g sand. Resulting concentration 167 µg/kg each.

Revision: 5	Status: Final	Method: SW-846, 3550B	Minor Revision Date: 6/7/01

13.0 SPECIAL PROJECTS REQUIREMENTS

13.1 Griffiss

13.1.1 When analyzing pesticide and PCB both pesticide MS/MSD and PCB MS/MSD must be extracted.

13.2 AFCEE - General Requirements

13.2.1 Batching

13.2.1.1 An AFCEE analytical batch is the environmental samples that are similar in composition (matrix) and are prepared at the same time and with the same **lot of reagents.**

13.2.1.2 A batch is not to exceed 20 samples. **The sample duplicate, MS, MSD are considered an environmental sample, therefore, the actual number of unique samples is less than 20.**

13.2.2 Laboratory Control Sample

13.2.2.1 Laboratory Control Sample for solid must be prepared using **Ottawa sand** unless a variance was obtained.

13.2.2.2 The LCS must be prepared from second source materials

13.2.2.3 The LCS must contain all single component target analytes (see Table 12-1) and toxaphene (in a second LCS) unless noted differently under specific method requirements or a variance was obtained (see Table 13-1).

13.2.2.4 The LCS must be spiked at a concentration ≤ midpoint of the calibration curve for each analyte unless a variance was obtained.

13.2.3 Matrix Spike/Matrix Spike Duplicate

13.2.3.1 The MS and MSD must contain all target analytes (see Table 12-1) and toxaphene in a second MS/MSD pair (see Table 13-1) and spiked at a concentration ≤ midpoint of the calibration curve for each analyte unless a variance was obtained.

13.2.3.2 AFCEE samples must be used for spiking.

13.2.3.3 If insufficient sample exists for MS/MSD, prepare a duplicate LCS.

Table 13-1

ADDITIONAL AFCEE QC SAMPLES

QC Type	Frequency	Preparation Instructions
LCS Toxaphene	1 per 20 or fewer AFCEE-defined environmental samples (see Section 13.2.1).	1.0 mL of toxaphene spike (see Table 8-2) added to 30 g sand. Resulting concentration 133 µg/kg toxaphene.
MS Toxaphene	1 per 20 or fewer AFCEE-defined environmental samples (see Section 13.2.1).	1.0 mL of toxaphene spike (see Table 8-2) added to 30 g sample. Resulting concentration 133 µg/kg toxaphene.
MSD Toxaphene	1 per 20 or fewer AFCEE-defined environmental samples (see Section 13.2.1).	Prepare same manner as MS.

Revision: 5	Status: Final	Method: SW-846, 3550B	Minor Revision Date: 6/7/01

13.3 U.S. Army Corps of Engineers (ACE)

13.3.1 Method Quantitation Limit (MQL) is between 3-10 times MDL. The low calibration standard concentration must be ≥3 times MDL. The Method Reporting Limit (MRL) may slide between two times the MDL and project-specific or regulatory requirements. Results below the MRL must be reported "ND." Results between the MQL and MRL must be reported as estimated (J flagged).

13.4 HAZWRAP, NJDEP

13.4.1 Internal chain-of-custody forms must be used for all extracts.

14.0 SAMPLE DISPOSAL

14.1 See SOP A.10 for Waste Disposal.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Extraction of Chlorinated Phenoxy Acid Herbicides from Water Samples			
Reviewer: J. Gilmore		Peer Reviewer: None	
Implementation Date: 7/9/99		Last Annual Review Date: 9/14/00	
Revision: 2	Status: Final	Method: SW-846,8151A	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the extraction of chlorinated phenoxy acid herbicides from water samples and Toxicity Characteristic Leachate Procedure (TCLP) extracts.

2.0 METHOD SUMMARY

2.1 Chlorinated phenoxy acids and their esters are extracted from the acidified water sample with ethyl ether. The acids are converted to methyl esters. The methyl esters are determined by gas chromatography. This SOP details the sample preparation procedure up to the esterification step. The esterification procedure can be found in SOP GC.60.

3.0 HEALTH AND SAFETY

3.1 Diethyl Ether is highly flammable, always use in a fume hood. Do not allow vapors to come into contact with open flames, sparks or hot surfaces. Do not allow diethyl ether to enter laboratory drains.

3.2 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.3 See SW-846 Method 8151A, Section 5.11.5 on peroxides.

3.4 Inspect all glassware before use for star cracks, breaks, rough edges. ***Do not*** use damaged glassware.

3.5 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 SW-846, Method 8151A, 3rd Edition, Rev. 1, December 1996.
4.2 SOP GC.60 – Analysis of Soil or Water Samples for Chlorinated Herbicides by Method 8151A.
4.3 Training SOP QA.5, A.23, and A.29.
4.4 Data Review SOP A.25.
4.5 Laboratory Quality Assurance Manual.
4.6 MDL SOP A.18.
4.7 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 **PTFE** – Polytetrafluoroethylene.
5.2 **Ether** – Diethyl ether.
5.3 See the generic list in the quality assurance manual.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Organic acids and phenol may interfere with the methylation process.

6.2 The herbicides being strong organic acids react with alkaline substances and, therefore, all glass-ware must be scrupulously clean and acid sulfuric acid rinsed before use.

6.3 The final drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2-hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

7.0 INSTRUMENTATION AND EQUIPMENT

2-L Separatory funnel with PTFE stopcock
88-mm Drying funnel
Acidified glass wool (Fisher or equivalent)

Kuderna-Danish Apparatus (K-D) includes:
Concentrator tube, 10-mL graduated
500-mL Evaporation flask (Kontes K-57001-500 or equivalent)
Snyder Column, micro (569261-031 or equivalent)
Snyder column, 3 ball macro (Kontes K-503000-0121 or equivalent)
Boiling chips, solvent extracted (10/40 mesh Fisher or equivalent)
Water bath heated with concentric ring cover capable of temperature control of +/- 5°C
2-mL Glass Vials with screw cap PTFE-lined tops
pH Test paper, Fisher narrow-range (≤4 pH units) or equivalent.
8-oz Jar
100-µL Wiretrol® pipettes or gas-tight syringe
1-L, Graduated cylinders
Culture tubes with Teflon®-lined screw caps

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard, Reagents, and Consumables.

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Chlorinated Herbicide Acid Mix 8151 Spiking Solution	Accustandard or equivalent	MCPP/MCPA at 10 mg/mL others at 0.1 mg/mL
5-Hydroxydicamba	Chemservice or equivalent	100 µg/mL
2, 4-D Acid	Supelco	5000 µg/mL
Silvex Acid	Supelco	5000 µg/mL
2,4-Dichlorophenyl Acetic Acid (DCAA)	Chemservice or equivalent	2000 µg/mL
Sulfuric Acid (H_2SO_4)	J.T. Baker or equivalent	Concentrated
Sodium Sulfate (Na_2SO_4)	J.T. Baker or equivalent	Neat

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Salt (NaCl)	J.T. Baker or equivalent	Neat
Sodium Hydroxide (NaOH)	J.T. Baker or equivalent	Neat, reagent grade
Diethyl Ether Ultra Resi Analyzed	J.T. Baker or equivalent	Neat
Isooctane	J.T. Baker or equivalent	Neat, reagent grade
Methanol (MeOH), HPLC grade	J.T. Baker or equivalent	Neat
Sodium Phosphate (NaH_2PO_4) Reagent Grade	Fisher or equivalent	Neat
Revision: 2	Status: Final	Method: SW-846,8151A	Minor Revision Date: None

8.2 Standard Solution Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	Standard Preparation	Final Concentration
Acidified Na_2SO_4	Na_2SO_4 H_2SO_4	1. Add 100 g Na_2SO_4 (anhydrous) to beaker. 2. Add enough diethyl ether to cover the Na_2SO_4. 3. Add 1.0 mL concentrated H_2SO_4. 4. Vacuum filter, using Buchner funnel and # 1 Whatman filter paper until dry. 5. Air dried 24 hours in a fume hood. 6. Check the pH of a slurry comprised of 5.0 mL water and 1 g Na_2SO_4. The pH must be < 2.	NA
37% KOH	KOH	Dissolve 37 g potassium hydroxide pellets in ASTM Type II water and dilute to 100 mL.	37% (w/v)
8151 Herbicide Spike	Underivatized 8151 Chlorinated Herbicide Mix and 5-Hydroxydicamba	350 µL underivatized chlorinated herbicide mix and 350 µL 5-Hydroxydicamba into 10 mL acetone.	3.5 µg/mL, except for MCPP and MCPA (350 µg/mL)
6N NaOH	NaOH	24.0 g NaOH in 100 mL of ASTM Type II water.	6N
Phosphate Buffer	NaH_2PO_4	12 g NaH_2PO_4 in 1 L of ASTM Type II water. Adjust pH to 2.5 with phosphoric acid.	0.1 M
25% H_2SO_4	H_2SO_4	25 mL concentrated H_2SO_4 added 75 mL of ASTM Type II water.	25%
2% H_2SO_4	H_2SO_4	2 mL concentrated H_2SO_4 added to 100 mL ASTM Type II water	2% v/v

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	Standard Preparation	Final Concentration
8151 Surrogate	DCAA	125 µL to 50 mL acetone	5 µg/mL
TCLP Herbicide Surrogate	DCAA	2.0 mL to 50 mL acetone	80 µg/mL
TCLP Herbicide Spike	2,4-D Acid Silvex Acid	1.0 mL 2,4-D plus 100 µL Silvex to 50 mL acetone	100 µg/mL 2,4-D 10 µg/mL Silvex.

Revision: 2	Status: Final	Method: SW-846,8151A	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/ Project	Field Collection to TCLP Extraction (Days)	TCLP Extraction to Preparative Extraction (Days)	Preparative Extraction to Analysis (Days)	Container Type and Preservative
Water	Standard	14	7	40	16-oz. Glass amber 2-6 C
Water	NYSDEC (1)	5 from verified time of sample receipt	7	40	16-oz. Glass amber 2-6 C

Revision: 2	Status: Final	Method: SW-846,8151A	Minor Revision Date: None

10.0 PROCEDURE

10.1 Glassware Preparation

10.1.1 Follow SOP A.31, Glassware Cleaning. When washing glassware after extraction, do not combine glassware from high- or medium-level extracts with standard level glassware. The dishwasher can spread the contamination. Wash glassware by hand separately from standard level glassware or throw it away if it will not come clean.

10.1.2 At a minimum, manually washed glassware must be tap water rinsed, followed by 3 rinses with ASTM Type II water.

10.1.3 All extraction and concentration glassware must be rinsed with 2% (v/v) sulfuric acid followed by ASTM Type II water immediately prior to use.

10.2 Separatory Funnel Extraction

10.2.1 Measure 1 L of water sample and transfer to a 2-L separatory funnel. (For TCLP extracts, add 100-mL extract and add 900 mL water.) Record exact sample volume used.

10.2.2 Add 1.0 mL of 8151 Herbicide Spike solution to two aliquots of sample chosen for MS/MSD. If doing TCLP, use 1.0 mL of TCLP Herbicide Spike in place of 8151 Herbicide Spike. If both TCLP and regular herbicide extractions are being done, both a MS, MSD and LCS are required for each.
10.2.3 Add 1.0 mL of 8151 Herbicide Spike solution to 1L ASTM Type II water to produce an LCS. If doing TCLP, use 1.0 mL of TCLP Herbicide Spike in place of 8151 Herbicide Spike.
10.2.4 Add 1.0 mL 8151 surrogate solution to each sample and QC sample. Add 1.0 mL TCLP surrogate if analyzing TCLP extracts.
10.2.5 Add 250 g of sodium chloride and shake until dissolved.

10.3 Hydrolosis of Herbicide Esters for Extraction
10.3.1 Add 17 mL of 6N sodium hydroxide, shake sample and check the pH with narrow-range pH paper. Add additional sodium hydroxide if pH is less than 12.
10.3.2 Let sample stand for a minimum of one hour, shaking periodically.
10.3.3 Add 60 mL of methylene chloride and shake vigorously for two minutes, venting periodically.
10.3.4 Allow sample to sit for 10 minutes. If emulsion is greater than 1/3 volume of solvent layer, transfer emulsion and centrifuge.
10.3.5 Discard methylene chloride layer.
10.3.6 Repeat steps 10.3.3 through 10.3.5 two additional times.

10.4 Extraction
10.4.1 Add 17 mL of cold (4°C) 25% sulfuric acid and check pH with narrow-range pH paper. Add additional sodium sulfate if pH is greater than 2.
10.4.2 Add 120 mL diethyl ether and shake vigorously for two minutes, venting periodically. Allow the sample to sit for 10 minutes. Break emulsions.
10.4.3 Collect diethyl ether into 500-mL Erlenmeyer flasks containing approximately 10 g acidified sodium sulfate. Vigorously shake periodically.
10.4.4 Repeat extraction of the water sample twice with 60-mL aliquots of diethyl ether.
10.4.5 Verify that the acidified sodium sulfate is free flowing by swirling the flask. If not free flowing, add additional acidified sodium sulfate until free flowing crystals are visible upon swirling the flask.
10.4.6 Let extract remain in contact with sodium sulfate for a minimum of 2 hours. Overnight is acceptable. This step is critical to ensure good recoveries. Moisture must be removed by sodium sulfate. Make certain there are some free flowing crystals of sodium sulfate always present in the flask.

10.5 Concentration
10.5.1 Put extract into a labeled K-D apparatus with 10-mL concentrator tube.
10.5.2 Attach a 3-Ball Snyder column. Pre-wet the column by adding 1 mL ether through the top. Place the apparatus on a hot (60-65°C) water bath so that the concentrator tube is partially immersed in the hot water and the lower surface of the flask is bathed in water vapor. Adjust the vertical position of the apparatus and the water temperature to complete the concentration in 15-20 minutes.
10.5.3 When the apparent volume reaches 1 mL, remove the K-D apparatus from the bath. Allow the sample to cool at least 10 minutes. Rinse flask with 1-2 mL ether. Remove concentration extraction tube.
10.5.4 Add fresh boiling chip to concentrator tube and attach micro Snyder column. Replace on steambath. Carefully reduce apparent volume to <1.0 mL. Remove from steambath and cool.
10.5.5 Rinse down sides of concentrator tube with 100-200 μL of ether.
10.5.6 Add 1.0 mL isooctane and 0.5 mL methanol. Bring to final volume of 4 mL with diethylether. Transfer to a culture tube and give to GC for derivatization. See GC.60 for esterification step.
10.5.7 Store extracted samples in the refrigerator until ready for derivatization.

11.0 DATA REDUCTION/EVALUATION/REPORTING

Target Compounds and Reporting Limits

See SOP GC.60 for target compounds and quantitation limits.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

12.1 Unless for a client with other specific requirements, prepare an MS/MSD for every 20 or fewer samples in a batch.

12.2 If sufficient water sample is not available for extraction, Sample Management must be asked to check if there is additional sample volume available in Custody.

12.2.1 If only 1 L is available, do not prepare an MS/MSD. Prepare two spiked blanks. Document the non-availability of additional sample volumes (after consulting Sample Management).

12.2.2 If only 2 L are available, prepare the sample using one liter, and prepare an MS/MSD by splitting the other liter equally. Document volume shortage of sample.

Routine Quality Control Samples

Table 12-1
ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions
Method Blank	1 per batch or 1 per 20 or fewer samples	1 L of ASTM Type II water processed as a sample.
Matrix Spike	1 per 20 or fewer samples	1.0 mL of 8151 Herbicide Spike solution added to 1 L of a water sample. For TCLP extracts, add 1 mL of the TCLP Herbicide Spike solution to the diluted extract.
Matrix Spike Duplicate	1 per 20 or fewer samples	Same as matrix spike.
Laboratory Control Sample (Spiked Blank)	1 per batch	1 L of ASTM Type II water spike as a matrix spike.
Surrogate in Samples	Each sample, method blank, MS, MSD, and LCS.	1 mL of 8151 Surrogate containing dichlorophenylacetic acid at 5 µg/mL added to each sample. For TCLP extracts: 1 mL of TCLP Herbicide Surrogate containing dichlorophenylacetic acid at 80 µg/mL added to the diluted extract.

Revision: 2	Status: Final	Method: SW-846,8151A	Minor Revision Date: None

13.0 SPECIAL PROJECT REQUIREMENTS

13.1 AFCEE - General Requirements

13.1.1 Batching

13.1.1.1 An AFCEE analytical batch is the environmental samples that are similar in composition (matrix) and are prepared at the same time and with the same **lot of reagents.**

13.1.2.2 A batch is not to exceed 20 samples. **The sample duplicate, MS, MSD are considered an environmental sample, therefore, the actual number of unique samples is less than 20.**

13.1.2 Laboratory Control Sample

13.1.2.1 The LCS must be prepared from second source material.

13.1.2.2 The LCS must contain all target analytes unless noted differently under specific method requirements or a variance was obtained.

13.1.2.3 The LCS must be spiked at a concentration ≤ the midpoint of the calibration curve for each analyte unless a variance was obtained.

13.1.3 Matrix Spike/Matrix Spike Duplicate

13.1.3.1 The MS and MSD must be spiked at a concentration ≤ midpoint of the calibration curve for each analyte unless a variance was obtained.

13.1.3.2 AFCEE samples must be used for spiking.

13.2 U.S. Army Corps of Engineers (ACE)

13.2.1 Method Quantitation Limit (MQL) is between 3-10 times MDL. The low calibration standard concentration must be ≥3 times MDL. The Method Reporting Limit (MRL) may slide between two times the MDL and project specific or regulatory requirements. The MRL may be higher or lower than the MQL. Results below the MQL must be reported as estimated (J flagged).

13.2.2 Prepare matrix QC on ACE sample. Spike with all analytes.

14.0 SAMPLE DISPOSAL

14.1 Follow Waste Disposal SOP A.10.

14.2 Broken glassware may be salvaged by the glass blower. Broken glassware is stored on the counter next to the dishwasher.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Extraction of Soil and Solid Waste for Chlorinated Phenoxy Acid Herbicides			
Reviewer: R. Ground		Peer Reviewer: None	
Implementation Date: 8/5/99		Last Annual Review Date: 9/19/00	
Revision: 3	Status: Final	Method: 8151A	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to soil and solid waste samples and provides for the hydrolysis of herbicides, solvent washing to remove other organics, acidification, extraction, and concentration. This procedure can be used to prepare samples for the determination of herbicide esters and acids and salts. Laboratory procedure is for the analysis of acids and salts unless esters are required by the project. This SOP details the sample preparation procedure up to the esterification step. The esterification step is described in SOP GC.60.

2.0 METHOD SUMMARY

2.1 A known volume of sample is acidified and extracted via sonication using acetone and methylene chloride. The aqueous portion is acidified and extracted into ether and concentrated prior to derivitization. See SOP GC.60 for the derivitization step.

3.0 HEALTH AND SAFETY

3.1 Diethyl ether is highly flammable, always use in a fume hood. Do not allow vapors to come into contact with open flames, sparks, or hot surfaces. Do not dry acidified sodium sulfate in an oven until all ether has been evaporated by air drying in a fume hood. Do not allow diethyl ether to enter laboratory drains.

3.2 All employees should protect themselves at a minimum with safety glasses, protective gloves and a lab coat. For more information see the E & E, Inc., Analytical Services Center, Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.3 See SW-846 Method 8151A, Section 5.11.5 on peroxides.

3.4 Inspect all glassware before use for star cracks, breaks, rough edges. ***Do not*** use damaged glassware.

3.5 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Method 8151A, Revision 1, (12/96), SW-846, 3rd ed., Update III, June 1997.
4.2 Data Review SOP A.25.
4.3 Training SOP QA.5, A.23, and A.29.
4.4 Laboratory Quality Assurance Manual.
4.5 MDL SOP A.18.
4.6 Corrective Action Procedure, QA.1.
4.7 SOP GC.60 – Analysis of Soil or Water Samples for Chlorinated Herbicides by Method 8151A.
4.8 Form C-47, Data Review Checklist.

5.0 DEFINITIONS/ACRONYMS

5.1 **PTFE** – Poly Tetrafluoroethylene.

5.2 **Ether** – Diethyl ether.

5.3 See the generic list in the Quality Assurance Manual.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Organic acids and phenol may interfere with the methylation process.

6.2 The herbicides being strong organic acids react with alkaline substances and, therefore, all glass-ware must be scrupulously clean and sulfuric acid rinsed before use.

6.3 The final drying stop is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2-hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

7.0 INSTRUMENTATION AND EQUIPMENT

Ultrasonic disruptor with ¾" titanium horn
Sonabox
400-mL thick-walled beakers
80-mm Drying funnel
Kuderna-Danish (K-D) apparatus:
- Concentrator tube, 10-mL graduated
- 500-mL Evaporation flask (Kontes K-57001-500 or equivalent)
- Snyder Column, micro (569261-031 or equivalent)
- Snyder Column, 3-ball macro (Kontes K-503000-0121 or equivalent)

Glass boiling chips, solvent extracted (10/40 mesh Fisher or equivalent)
Water bath heated with concentric ring cover capable of temperature control of +/-5 C
Balance, top-loading (readable to 0.001 g or better)
2-mL Glass vials with screw cap PTFE-lined tops
Bench top centrifuge (Fisher or equivalent)
Spatula, stainless steel
100-µL Wiretrol™ pipettes
1000-mL Erlenmeyer flask (or equivalent)
500-mL Separatory funnel
Glass rod
pH Test paper (Fisher narrow-range [<4 pH units] or equivalent)
16-oz. jars
Buchner funnel
Vacuum flask
Whatman #1 filter paper or equivalent

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard, Reagents, and Consumables

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Hydrochloric acid (HCl)	Fisher or equivalent	Concentrated, reagent grade
Sulfuric acid (H_2SO_4)	Fisher or equivalent	Concentrated, reagent grade
Sodium sulfate (Na_2SO_4)	Mallinckrodt or equivalent	Neat, reagent grade
Potassium hydroxide (KOH)	Fisher or equivalent	Neat, reagent grade
Diethyl ether, anhydrous	J.T. Baker or equivalent With BHT Preservative	Ultra resi analyzed or equivalent
Sand	Pool Mart or equivalent	NA
2,4-Dichlorophenylacetic acid (DCAA)	Chem Service or equivalent	2000 µg/mL
8151 Herbicide Acid mix	Accustandard or equivalent	MCPP/MCPA at 10 mg/mL, Others at 0.1 mg/mL
5-Hydroxydicamba	Chem Service or equivalent	100 µg/mL
Acetone, pesticide grade	J.T. Baker or equivalent	Neat
Methylene chloride ($MeCl_2$) Pesticide grade	J.T. Baker or equivalent	Neat
Isooctane, pesticide grade	J.T. Baker or equivalent	Neat
Methanol, HPLC grade	J.T. Baker or equivalent	Neat

Revision: 3	Status: Final	Method: 8151A	Minor Revision Date: None

8.2 Standard Solution Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
Acidified Na_2SO_4	Na_2SO_4 H_2SO_4	1. Add 100 g Na_2SO_4 (anhydrous) to beaker. 2. Add enough diethyl ether to cover the Na_2SO_4. 3. Add 1.0 mL concentrated H_2SO_4. 4. Vacuum filter through Buchner funnel using Whatman #1 filter paper. 5. Check the pH of a slurry comprised of 5.0 mL ASTM Type II water and 1 g Na_2SO_4. The pH must be < 2.	NA

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
37% KOH solution	KOH	In a 100-mL volumetric flask, dissolve 37 g potassium hydroxide pellets in ASTM Type II water and dilute to 100-mL.	37% (w/v)
Sand		Baked 120° C for 16 hours	NA
2% H_2SO_4	Sulfuric Acid (H_2SO_4)	2-mL to 100 mL ASTM Type II water	2% (v/v)
25% H_2SO_4	Sulfuric Acid (H_2SO_4)	25-mL to 100 mL ASTM Type II water	25% (v/v)
8151 Herbicide Spike	8151 Herbicide Mix underivitized and 5-Hydroxydicamba	350 µL each to 10-mL acetone	MCPP/MCPA at 350 µg/mL, others at 3.5 µg/mL
8151 Surrogate Solution	DCAA	125 µL to 50-mL acetone	5 µg/mL

Revision: 3	Status: Final	Method: 8151A	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Solid	Standard	14 collection	NA	2-6 C, glass
	NYSDEC (1)	5 VTSR	NA	2-6 C, glass

Revision: 3	Status: Final	Method: 8151A	Minor Revision Date: None

10.0 PROCEDURE

10.1 Glassware Preparation

10.1.1 See Glassware Cleaning Procedure, SOP A.31.

10.1.2 When washing glassware after extraction, do not combine glassware from high- or-medium level extracts with standard level glassware. The dishwasher can spread the contamination. Wash glassware by hand separately from standard level glassware or throw it away if it will not come clean.

10.2 Extraction

10.2.1 Add 25 to 35 g (weighed to the nearest 0.1 g) of well mixed soil to 400-mL thick wall beakers. Adjust pH to 2.0 using narrow-range (0 - 2.5 pH paper) with concentrated HCl and thoroughly mix contents with glass stirring rod. Add 1.0 mL 8151 surrogate to all samples and blanks and 1.0 mL 8151 herbicide spike to LCS, MS/MSD.

10.2.2 Add approximately 30 g acidified Na_2SO_4 to sample and mix until it is free flowing.

10.2.3 Add 100 mL 1:1 (v/v) methylene chloride/acetone to beaker and sonicate for 3 minutes at 10 output and 50% pulse.

10.2.4 Decant solvent into 500-mL Erlenmeyer flasks containing approximately 10 g of acidified Na_2SO_4.

10.2.5 Sonicate 2 times more with 100 mL methylene chloride. (Note: 1:1 $MeCl_2$/acetone as used in 10.2.3). Decant each time combining extracts in the designated Erlenmeyer flask.

10.2.6 Clean the horn of the sonicator between samples rinsing with ASTM Type II water. Wipe the horn and rinse with acetone, then methylene chloride.

10.2.7 Verify that the acidified Na_2SO_4 is free flowing by swirling the flask. If not free flowing, add additional acidified Na_2SO_4 until free-flowing crystals are visible upon swirling the flask.

10.2.8 Let the extract set in contact with the sodium sulfate for a minimum of 2 hours.

10.2.9 Quantitatively transfer the extract to a K-D apparatus. Add boiling chip and macro Snyder column.

10.2.10 Concentrate on steambath to approximately 5 mL.

10.2.11 Remove from steambath, cool to room temperature.

10.2.12 **If the herbicide esters, acids, and salts are to be analyzed,** proceed with Sections 10.3 and 10.4. **If only the herbicide acids and salts** are to be analyzed, go directly to Section 10.5. Consult with the section supervisor first to determine which procedure is applicable.

10.3 Hydrolysis of Herbicides; Removal of Extraneous Organics

10.3.1 To a 1000-mL Erlenmeyer flask add 5 mL 37% KOH, 30 mL ASTM Type II water and three glass boiling beads.

10.3.2 Add soil extract.

10.3.3 Attach a 3-ball Snyder column to the 1000-mL Erlenmeyer flask and place directly on a 60-65 C steambath. Continue heating until methylene chloride has evaporated (approximately 2 hours).

10.3.4 After the remaining water solution has cooled, quantitatively transfer it to a 500-mL separatory funnel using a 5-10 mL Type II water wash .

10.3.5 Add 100 mL methylene chloride to the separatory funnel. Shake for 2 minutes and allow to stand for 10 minutes.

10.3.6 Discard methylene chloride layer (bottom)

10.3.7 Repeat steps 10.3.5 and 10.3.6 twice more using 100 mL methylene chloride. The hydrolyzed herbicides remain soluble in the aqueous phase.

10.4 Extraction of Hydrolyzed Herbicides from Aqueous Phase

10.4.1 Add 10.0 mL 25% cold (2-5 C) sulfuric acid. Check the pH using narrow-range pH paper; it should be < 2. Add more acid if necessary.

10.4.2 Add 100-mL ether to the separatory funnel. Shake for 2 minutes; allow to stand 10 minutes.

10.4.3 Drain the water into a 1000-mL Erlenmeyer. Drain ether into a clean Erlenmeyer flask.

10.4.4 Return the water to the separatory funnel and repeat steps 10.4.2 and 10.4.3 twice more using 40 mL ether.

10.4.5 Combine the ether extracts and add acidified sodium sulfate until free flowing. Stopper the flask. Let stand for a minimum of 2 hours (overnight is acceptable). This step is critical to ensure good recoveries. Moisture must be removed by sodium sulfate. Make certain there are always some free flowing crystals of sodium sulfate present in the flask.

10.5 Concentration

10.5.1 If esters, acids, and salts are to be determined, pour the ether through a funnel plugged with acid washed glass wool that has been pre-rinsed with diethyl ether into a 500-mL K-D flask equipped with a 10-mL concentrator tube. Rinse the Erlenmeyer twice with a total of 20 - 30 mL ether. **If only herbicide acids are to be determined,** pour 5 mL extract into 500-mL K-D flask equipped with a 10-mL concentrator tube. Add 50 mL hexane.

10.5.2 Attach a 3-ball Snyder column. Pre-wet the column by adding 1 mL ether through the top. Place the apparatus on a hot (60-65 C) water bath so that the concentrator tube is partially immersed in the hot water and the lower surface of the flask is bathed in water vapor. Adjust the vertical position of the apparatus and the water temperature so concentration is complete in 15-20 minutes.

10.5.3 When the apparent volume reaches 2 mL, remove the K-D apparatus from the bath and allow sample to condense and cool at least 10 minutes.

10.5.4 Remove the 3-ball Synder. (If sediment is evident, pour concentrate into centrifuge tube and centrifuge until sediment is separated from extract. Pour extract into a clean concentrator tube and add a fresh boiling chip.) Rinse the flask into the concentrator tube with a small amount of ether. Remove concentrator tube and add a fresh boiling chip and micro Snyder column apparatus to concentrate.

10.5.5 Return to steambath. Carefully reduce volume to approximately 1.0 mL. Remove from steambath to cool.

10.5.6 Rinse down the sides of the concentrator tube with approximately 200 μL of ether.

10.5.7 Add 1.0 mL isooctane 0.5 mL methanol. Bring to final volume of 4.0 mL with diethyl ether. Transfer to a culture tube and give to GC for derivitization. See SOP GC.60 for esterification step.

10.5.8 Store extracted samples in refrigerator until ready for derivitization.

Instrument Conditions and Maintenance

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Sonicator	Pitted horn	Replace horn	As needed
Sonicator	Low out put	Check voltage, retune	As needed
Revision: 3	Status: Final	Method: 8151A	Minor Revision Date: None

11.0 DATA REDUCTION/EVALUATION/REPORTING

See GC.60 for target compounds and quantitation limits.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Routine Quality Control Samples

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions
Method Blank	1 per 20 or fewer samples	30 g of sand processed as a sample
Matrix Spike	1 per 20 or fewer samples	1 mL of 8151 herbicide spike solution added to 30 g of sample (117 µg/kg, 11700 for MCPA, MCPP).
Matrix Spike Duplicate	1 per 20 or fewer samples	Prepared as the matrix spike on another portion of the same sample
Laboratory Control Sample (Spiked Blank)	1 per batch of 20 or fewer samples	1 mL of 8151 herbicide spike solution added to 30 g of sand and processed as a sample (117 µg/kg, 11700 for MCPA, MCPP).
Surrogate	Each sample, method blank, MS/MSD, and LCS.	1 mL of 8151 surrogate solution added to each (Concentration: 167 µg/kg).

Revision: 3	Status: Final	Method: 8151A	Minor Revision Date: None

13.0 SPECIAL PROJECT REQUIREMENTS

13.1 AFCEE - General Requirements

13.1.1 Batching

13.1.1.1 An AFCEE analytical batch is the environmental samples that are similar in composition (matrix) and are prepared at the same time and with the same **lot of reagents.**

13.1.2.2 A batch is not to exceed 20 samples. **The sample duplicate, MS, MSD are considered an environmental sample, therefore, the actual number of unique samples is less than 20.**

13.1.2 Laboratory Control Sample

13.1.2.1 Laboratory Control Sample for solid must be prepared using **Ottawa sand** unless a variance was obtained.

13.1.2.2 The LCS must be prepared from second source material.

13.1.2.3 The LCS must contain all target analytes unless noted differently under specific method requirements or a variance was obtained.

13.1.2.4 The LCS must be spiked at a concentration ≤ the midpoint of the calibration curve for each analyte unless a variance was obtained.

13.1.3 Matrix Spike/Matrix Spike Duplicate.

13.1.3.1 The MS and MSD must be spiked at a concentration ≤ midpoint of the calibration curve for each analyte unless a variance was obtained.

13.1.3.2 AFCEE samples must be used for spiking.

14.0 SAMPLE DISPOSAL

14.1 As per Waste Disposal SOP (A.10).

14.2 Broken glassware may be able to be salvaged by the glass blower. Broken glassware is stored on the counter next to the dishwasher.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Extraction and Concentration of Pesticides and PCB from Water Samples			
Reviewer: B. Kruszka		Peer Reviewer: L. DePalma	
Implementation Date: 8/11/00		Last Annual Review Date: 6/8/01	
Revision: 6	Status: Final	Method: 3510C, 3520C	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedure for extraction and concentration of organochlorine pesticides and PCBs in water samples.

2.0 METHOD SUMMARY

2.1 Water samples of a known volume to which surrogate and spiking solutions have been added are extracted with methylene chloride in a separatory funnel or continuous Liquid-Liquid Extractor. The resulting extract is concentrated and solvent exchanged to hexane in a Kuderna-Danish apparatus.

2.2 Instructions are provided for preparation of substitute quality control samples should only PCBs be the required analytes or should additional PCB QC be required by the client.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 All glassware, especially separatory funnels, should be inspected before use. Do not use glassware with star imperfections, cracks or ragged edges.

3.3 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Method 3510C (Rev 3, 12/96), 3520C(Rev 3, 12/96), SW-846, 3rd ed. Update III, June 1997.

4.2 Method 608, 40 CFR, Part 136, Appendix A, (7-1-94 ed.).

4.3 Notebook Policy, SOP A.37.

4.4 Chemical Hygiene Plan (L-009).

4.5 ISO Guide 25, General Requirements for the Competence of Calibration and Testing Laboratories. Third Edition, 1990.

4.6 Waste Disposal Plan, SOP A.10.

4.7 SOP GC.14 for determinative analysis.

4.8 Use checklist number C-049 for analyst and peer review.

4.9 Training SOP QA.5, A.23, and A.29.

4.10 Data Review SOP A.25.

4.11 Laboratory Quality Assurance Manual (L-001).

4.12 MDL SOP A.18.

4.13 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 Batch – A group of samples that behave similarly with respect to the sampling or testing procedures being employed which are processed as a unit. If the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

5.2 Method Blank /MB – An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is used to document contamination resulting from the analytical process.

5.3 Matrix – The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

5.4 Matrix Spike /MS – An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

5.5 DCB and TCMX – Decachlorobiphenyl and tetrachlorometaxylene, respectively, all are method surrogates. 4C3N - 4-chloro-3-nitrobenzotrifluoride - is an alternative surrogate for Methods 3510C, 3520C and 8081.

5.6 Matrix Spike Duplicate /MSD – An intralaboratory split sample spiked with identical concentrations of analytes. They are used to document the precision and bias of a method in a given sample matrix.

5.7 Laboratory Control Sample/LCS – A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Phthalates may cause false positives. The use of plastic (except Teflon®) is prohibited. Gloves should not contact the interior of glassware.

6.2 Contaminated glassware is the greatest source of interference. Glassware must be scrupulously clean. Glassware must be free of contamination and residual detergent and acid from the washing process.

6.3 Exact measurement of the final volume is critical to the accuracy of the method.

7.0 INSTRUMENTATION AND EQUIPMENT

- 2-L Separatory funnel with PTFE stopcock
- Glass wool (Fisher or equivalent)
- Kuderna-Danish (K-D) Apparatus:
- Concentrator tube, 10 mL graduated
- 100 µL gas tight syringes
- 500-mL Evaporative flask (Kontes K-57001-500 or equivalent)
- Snyder Column, micro (K569261-03 or equivalent)
- Snyder Column, 3 ball macro
- (K-503000-0121 or equivalent)
- Contrad NF
- Sparkleen or equivalent
- Liquid-Liquid extractor with 500-mL distillation flask
- Water bath heated with concentric ring cover capable of Temperature control of +/- 5 C
- 80-mm Drying funnel
- 15-mL Glass vials with screw cap (PTFE-lined)
- pH Test paper, Universal (Whatman 2C13991 or equivalent)
- 8-oz jars
- 1-L Graduated cylinders
- Aluminum foil
- Boiling chips, solvent extracted (10/40 mesh Fisher or Equivalent)
- NOCHROMIX
- Heating mantle and voltage regulator
- Zymark Turbovap
- Turbovap Concentration Tubes

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard, Reagents, and Consumables

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Sulfuric acid (H_2SO_4)	JT Baker ACS reagent grade or equivalent	Concentrated
Sodium hydroxide, (NaOH)	JT Baker ACS reagent grade or equivalent	Neat
Nitric acid, (HNO_3)	JT Baker ACS reagent grade or equivalent	Concentrated
Methylene chloride (CH_2Cl_2)	JT Baker Ultra Resi-analyzed or equivalent	Neat
Hexane	JT Baker Ultra Resi-analyzed or equivalent	Neat
Acetone	JT Baker Ultra Resi-analyzed or equivalent	Neat
Methanol	JT Baker Ultra Resi-analyzed or equivalent	Neat, store at 120 C
Sodium sulfate (Na_2SO_4), anhydrous	JT Baker ACS Reagent Grade or equivalent	alpha-BHC 200 beta-BHC 200 gamma-BHC 200 delta-BHC 200 Heptachlor 200 Aldrin 200 Heptachlor epoxide 200 alpha-chlordane 200 gamma-chlordane 200 Endosulfan I 200 4,4-DDE 200 Dieldrin 200 Endrin 200 Endosulfan II 200 4,4-DDD 200 Endrin aldehyde 200 4,4-DDT 200 Endosulfan sulfate 200 Methoxychlor 200

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Pesticide Matrix Spike Mix	Restek or equivalent	Toxaphene 200
Toxaphene	Chem Service or equivalent	1000 µg/mL
Methoxychlor	Restek or equivalent	1000 µg/mL
Pesticide Surrogate Mix	Restek or equivalent	200 µg/mL TCMX and DCB
Aroclor 1016/1260 (AR 1660)	Restek or equivalent	1000 µg/mL each
ASTM Type II water	ASC Water Purity System	NA
NOCHROMIX	Fisher or equivalent	NA

Revision: 6	Status: Final	Method: 3510C, 3520C	Minor Revision Date: None

8.2 Standard Solution Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
1:1 (v/v) H_2SO_4	H_2SO_4 concentrated	50 mL added to 50 mL ASTM Type II water **(Caution: Heat evolves)**	1:1 (v/v)
Na_2SO_4, dried	Na_2SO_4	Dried and stored at 120 C in oven	N/A
10N NaOH	NaOH	40 g dissolved in 100 mL with ASTM Type II water	10 N
10% HCl	HCl	10 mL to 100 mL ASTM Type II water	10 % (v/v)
AR 1660 PCB Spike	Aroclor 1016/1260	250 µL to 50 mL with acetone	5 µg/mL each
8081A Pesticide/ PCB Surrogate	Pesticide Surrogate Mix	100 µL Pesticide Surrogate Mix to 100 mL with acetone	0.2 µg/mL TCMX and DCB
8081A Pesticide Spike	Pesticide Matrix Spike Mix	50 µL pesticide spike mix plus 90 µL methoxychlor to 100 mL with methanol	0.1 µg/mL for all compounds except methoxychlor at 1.0 µg/mL
Toxaphene Spike	Toxaphene 1000 µg/mL	200 µL diluted with acetone to 50 mL	4 µg/mL

Revision: 6	Status: Final	Method: 3510C, 3520C	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Water	Standard	7 days from collection	40 days from extraction	1-L Amber @ 2-6 C
Water	NYSDEC	5 days from receipt	40 days from extraction	1-L Amber @ 2-6 C

Revision: 6	Status: Final	Method: 3510C, 3520C	Minor Revision Date: None

10.0 PROCEDURE

10.1 Glassware Preparation

10.1.1 All glassware shall be immediately rinsed with the last solvent used, manually washed in hot soapy tap water using Sparkleen or automatically washed using 2% Contrad NF.

10.1.2 At a minimum, manually washed glassware shall be rinsed once with tap water and three times with ASTM Type II water.

10.1.3 If glassware still appears dirty, subject it to sonication or cleaning with NOCHROMIX.

10.1.4 When washing glassware after extraction, do not combine glassware from high- or medium-level extracts with standard level glassware. The dishwasher can spread the contamination. Wash glassware by hand separately from standard level glassware or throw it away if it will not come clean.

10.2 Preventing Contamination

10.2.1 Prior to use, solvent rinse all glassware with Acetone followed by methylene chloride.

10.2.2 Dry sodium sulfate @ 120 C or higher overnight or longer.

10.2.3 Rinse dried sodium sulfate with methylene chloride before use.

10.2.4 Always use a clean graduate when measuring multiple samples.

10.3 Sample Set-Up

10.3.1 A batch of samples consists of a method blank, matrix spike (MS), matrix spike duplicate (MSD) and laboratory control sample (LCS) for every 20 or fewer samples. A laboratory control sample duplicate shall be used if insufficient sample is provided for MSD.

10.3.2 If both pesticide and PCB are required, allow three additional extraction setups for separate LCS, MS and MSD.

10.3.2 Each sample set up consists of one 2-L separatory funnel, one drying funnel, one 8-oz. jar. Set up the water preparation logbook as follows.

10.3.2.1 Extraction date.

10.3.2.2 Analyst name.

10.3.2.3 Method/test (SOP numbers with revision number).

10.3.2.4 Work order number.

10.3.2.5 Sample number.

10.3.2.6 Sample volume.

10.3.2.7 Solvent used.

10.3.2.8 Exchange solvent.
10.3.2.9 Final volume.
10.3.2.10 Date of concentration and analyst initials.

10.4 Sample Extraction by Separatory Funnel (Method 3510C)
10.4.1 Each sample setup consists of a separatory funnel, a drying funnel and an 8-oz jar.
10.4.2 Add a plug of glass wool and approximately 50 g of sodium sulfate to the drying funnel. Rinse the funnel with methylene chloride and discard the rinse solvent.
10.4.3 If 1 L or less of sample is provided, measure the volume by pouring into a 1-L graduated cylinder. Record the volume to the nearest 5 mL in the extraction logbook as "volume extracted." Transfer the sample to a 2-L separatory funnel. If sample volume is less than 800 mL, add enough ASTM Type II water to bring the volume of sample plus water to 1 L in the separatory funnel.
10.4.4 If more than 1 L of sample is provided, measure 1 L into a 1-L graduated cylinder and record the volume of the sample to the nearest 5 mL. Transfer sample into separatory funnel. Record the volume in the extraction logbook as "volume extracted". Always use a clean graduated cylinder for each sample.
10.4.5 Measure pH using wide-range pH indicator strips. Record. Adjust pH to between 5 and 9 with 10N NaOH and/or (1:1) sulfuric acid solution if necessary. Record pH adjustment and lot numbers for acids and bases.
10.4.6 Add 1.0 mL of pesticide/PCB surrogate solution to each sample and QC (method blank, MS, MSD, LCS) sample using a 1.0-mL syringe or volumetric pipette. See Table 12-1 for addition of other spiking solutions. Swirl the separatory funnel to mix.
10.4.7 Rinse both the sample bottle and the graduated cylinder with 10-20 mL of the extraction solvent and add to the separatory funnel.
10.4.8 If requested by client or if insufficient quantity exists for the preparation of MS/MSD, add surrogate and spike to 1 L of ASTM Type II water. The sample is designated as a Laboratory Control Sample duplicate (LCSD).
10.4.9 Pesticide Spiking Instructions
10.4.9.1 If pesticide extraction is assigned to the sample do the following, otherwise proceed to PCB Spiking Instructions:
10.4.9.2 Add 1.0 mL of 8081A pesticide spike solution to the pesticide matrix spike, pesticide matrix spike duplicate and pesticide laboratory control sample (see Table 12-1).

10.4.10 PCB Spiking Instructions
10.4.10.1 If PCB extraction is assigned to the sample a PCB MS, MSD, and LCS must be prepared. DO NOT spike the sample aliquot with both pesticide and PCB spikes.
10.4.10.2 Add 1.0 mL of AR 1660 PCB spike to the PCB matrix spike, PCB matrix spike duplicate and PCB laboratory control sample (see Table 12-1).
10.4.11 Add 60-mL methylene chloride to the separatory funnel.
10.4.12 Shake vigorously for 2 minutes with frequent venting.
10.4.13 Allow the methylene chloride (lower) layer to separate from the water phase for a minimum of 10 minutes.
10.4.14 If an emulsion interface between layers is greater than 1/3 the size of the methylene chloride solvent layer, centrifuge or use other mechanical means until the emulsion breaks. Decant the water layer back to the separatory funnel.
10.4.15 Collect the methylene chloride layer into the jar through the drying funnel. The funnel is then rinsed thoroughly with clean methylene chloride.
10.4.16 Repeat extraction two more times with (2) 60-mL volumes of methylene chloride. Combine extracts.

10.4.17 Cover the jar with foil and label. Concentrate using one of the two procedures below.
10.4.18 Record the extraction method used in the notebook.

10.5 Continuous Liquid-Liquid Extraction (Method 3520C)

10.5.1 Add (approximately 500 mL) methylene chloride to the bottom of the extractor to a depth of at least one-inch (1") above the bottom side arm.
10.5.2 If less than 1-L of sample is provided, measure the volume by pouring into a 1-L graduated cylinder. Record the volume in the extraction logbook as "volume extracted."
10.5.3 If more than 1 L of sample is provided, measure 1 L by pouring into a 1-L graduated cylinder. Record the volume of the sample to the nearest 5 mL
10.5.4 Measure pH using wide-range pH indicator strips. Record. Adjust pH to between 5 and 9 with 10N NaOH and/or (1:1) sulfuric acid solution if necessary. Record pH and lot numbers for acid and base.
10.5.5 Prepare quality control samples (matrix spike, matrix spike duplicate, method blank, and LCS as described in Table 12-1. Add spiking solutions where required to the sample **in the graduated cylinder.**
10.5.5.1 If PCB extraction is assigned to the sample a PCB MS, MSD, and LCS must be prepared. DO NOT spike the sample aliquot with both Pesticide and PCB spikes.
10.5.5.2 If requested by client or if insufficient quantity exists for the preparation of MS/MSD, add surrogate and spike to 1 L of ASTM Type II water. The sample is designated as a Laboratory Control Sample duplicate (LCSD).
10.5.6 Transfer to a labeled extraction chamber. Take care that the sample does not spill over the side arm into the distilling flask. (Note: A layer of glass wool may be placed between the methylene chloride and sample if the sample contains a large amount of suspended solids that may precipitate.)
10.5.7 Rinse both bottle and graduated cylinder with 10-20 mL of extraction solvent and add to extractor.
10.5.8 Attach condenser, turn on cooling water, heating mantle, voltage regulator, and extract for a minimum of 18 hours. Maintain methylene chloride drip rate at 10-15 mL per minute. Adjust voltage as necessary.
10.5.9 Turn off heating mantle and voltage regulator. Tip extractor to empty most of the methylene chloride into the distilling flask.
10.5.10 Add glass wool and approximately 50 g of sulfuric acid to the drying funnel. Rinse the funnel with methylene chloride and discard the rinse solvent.
10.5.11 Collect the methylene chloride extract into a jar or directly into the K-D apparatus by pouring through the drying funnel. Rinse the funnel thoroughly with clean methylene chloride and drain into K-D.
10.5.12 Concentrate as described below.
10.5.13 Record the extraction method used in the notebook.

10.6 Concentration Procedure

10.6.1 Assemble a Kuderna-Danish (K-D) apparatus by attaching a 10-mL concentrator tube containing a single boiling chip to a 500-mL evaporative flask.
10.6.2 Quantitatively transfer the extract to the K-D apparatus, rinsing the container with 20-30 mL methylene chloride to complete the transfer.
10.6.3 Attach a 3-ball Snyder column and pre-wet with methylene chloride. Place on a hot water bath (80-90 C) so that the concentrator tube is partially immersed in the hot water and the lower rounded surface of the evaporative flask is bathed with hot water vapor.
10.6.4 Adjust the vertical position of the apparatus and the water temperature to complete the concentration in 10-20 minutes. Concentrate to approximately 4 mL.
10.6.5 Allow the extract to cool and drain for at least 10 minutes. At no time should the extract be allowed to go to dryness throughout the concentration procedure. Should the extract go to dryness, restart the ex-

traction from the beginning if sample is available. Otherwise, clearly note in extraction log and file a Corrective Action Form notifying the Project Manager and Supervisor.

10.7 K-D Solvent Exchange

10.7.1 After cooling, momentarily remove the Snyder column and add approximately 50 mL of hexane, a fresh boiling chip and reattach the Snyder column.

10.7.2 Place the apparatus on the steam bath to complete the concentration in 5 to 10 minutes.

10.7.3 When the apparent volume of liquid reaches approximately 4 mL, remove the K-D apparatus from the bath and allow the K-D apparatus to drain and cool for approximately 10 min (or longer).

10.7.4 If during concentration, solvent vapor stops escaping from Snyder column before the final volume is reached, the extract contains large concentrations of higher boiling constituents. Concentration should be stopped and the extract brought to the next higher 5-mL level. Final volume must be noted in the water preparation logbook.

10.7.5 Remove the Snyder column and rinse the flask and its lower joint with 1-2 mL of hexane into the concentrator tube.

10.7.6 If a large amount of sediment appears in the concentrated extract, it should be centrifuged for 5 minutes. Decant the extract into a 10-mL concentration tube and bring up to 10 mL final volume with hexane. Note final volume in the logbook.

10.7.7 Transfer approximately 2 mL of extract into two 1-mL screwtop vials with PTFE caps.

10.8. Turbovap Concentration Procedure (alternative to K-D procedure)

10.8.1 Quantitatively transfer sample from jar to Zymark Turbovap tube rinsing with 20-30 mL methylene chloride to complete the transfer.

10.8.2 Place in Turbovap, set at 38 C to 45 C with a gentle stream of nitrogen. Concentrate to 1 mL (or nearest 5 mL if evaporation/concentration can not be brought down to 1 mL).

10.9 Turbovap Solvent Exchange Procedure

10.9.1 Add a minimum of 10 mL hexane to sample in Turbovap tube.

10.9.2 Place in Turbovap, set at 38 C to 45 C with a gentle stream of nitrogen. Concentrate to 1 mL (or nearest 5 mL if evaporation/concentration can not be brought down to 1 mL).

10.9.3 Bring to 10 mL with hexane. Record the final volume in the notebook.

10.9.4 Add approximately 1 g activated copper to one of the 1-mL vials and vortex for 15 minutes. If no reaction, remove from vortex; if there is a reaction (dark or cloudy precipitate), vortex for an additional 15 minutes.

Instrument Maintenance

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Heating Mantle	Not heating	Check power/replace	As necessary
Water Bath	Not heating	Replace coils	As necessary

Revision: 6	Status: Final	Method: 3510C, 3520C	Minor Revision Date: None

11.0 DATA REDUCTION/EVALUATION/REPORTING

NA

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions
Method Blank	1 per 20 or fewer samples prepared in a batch	1000 mL ASTM Type II water processed as a sample
Surrogate	To each sample and QC sample (Blank, MS, MSD, LCS)	1.0 mL of 8081A pesticide/PCB surrogate solution. (Resulting concentration DCB, TCMX each 0.2 µg/L.)
Pesticide Matrix Spike (MS)	1 per 20 or fewer samples prepared in a batch	1.0 mL of a pesticide spike solution added to 1 L of sample. (Resulting concentration 0.1 µg/L for all compounds except methyoxyclor at 1.0 µg/L.)
Pesticide Matrix Spike Duplicate (MSD)	With each matrix spike for pesticide	Prepare as the matrix spike
Laboratory Control Sample (LCS)	1 per 20 or fewer samples prepared in a batch	2.0 1.0 mL of 8081A pesticide spiking solution added to 1 L of ASTM Type II water. (Resulting concentration 0.1 µg/L for all compounds except methyoxyclor at 1.0 µg/L.)
PCB Matrix Spike (MS)	1 per 20 or fewer samples in a batch requiring PCB QC	1.0 mL of AR 1660 PCB spike to 1 L of water sample

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions
PCB Matrix Spike Duplicate (MSD)	With each PCB MS	Same as matrix spike
PCB- Laboratory Control Sample (LCS)	1 per 20 or fewer samples in a batch requiring PCB QC	1.0 mL of AR 1660 PCB spike to 1 L ASTM Type II water. (Resulting concentration 5 µg/L each.)

Revision: 6	Status: Final	Method: 3510C, 3520C	Minor Revision Date: None

13.0 SPECIAL PROJECTS REQUIREMENTS

13.1 Griffiss

13.1.1 When analyzing pesticide and PCB, both pesticide MS/MSD and PCB MS/MSD must be extracted.

13.2 AFCEE - General Requirements

13.2.1 Batching

13.2.1.1 An AFCEE analytical batch is the environmental samples that are similar in composition (matrix) and are prepared at the same time and with the same **lot of reagents.**

13.2.1.2 A batch is not to exceed 20 samples. **The sample duplicate, MS, MSD are considered an environmental sample, therefore, the actual number of unique samples is less than 20.**

13.2.2 Laboratory Control Sample

13.2.2.1 Laboratory Control Sample for solid must be prepared using **Ottawa sand** unless a variance was obtained.

13.2.2.2 The LCS must be prepared from second source materials.

13.2.2.3 The pesticide LCS must contain all single component target analytes (Table 12-1) and toxaphene (in a second LCS) unless noted differently under specific method requirements or a variance was obtained (see Table 13-1).

13.2.2.4 The LCS must be spiked at a concentration ≤ midpoint of the calibration curve for each analyte unless a variance was obtained.

13.2.3 Matrix Spike/Matrix Spike Duplicate

13.2.3.1 The MS and MSD must contain all target analytes (Table 12-1) and toxaphene in a second MS/MSD pair (Table 13-1) and spiked at a concentration ≤ midpoint of the calibration curve for each analyte unless a variance was obtained.

13.2.3.2.1 AFCEE samples must be used for spiking.

13.2.3.2.2 If insufficient sample exists for MS/MSD, prepare a duplicate LCS.

Table 13-1

ADDITIONAL AFCEE QC SAMPLES

QC Type	Frequency	Preparation Instructions
LCS Toxaphene	1 per 20 or fewer AFCEE-defined environmental samples (see Section 13.2.1)	1.0 mL of toxaphene spike (Table 8-2) added to 1L ASTM Type II water. (Resulting concentration 4 µg/L toxaphene.)
MS Toxaphene	1 per 20 or fewer AFCEE-defined environmental samples (see Section 13.2.1)	1.0 mL of toxaphene spike added to 1-L sample. (Resulting concentration 4 µg/L toxaphene.)
MSD Toxaphene	1 per 20 or fewer AFCEE-defined environmental samples (see Section 13.2.1)	Prepare same manner as MS.

Revision: 6	Status: Final	Method: 3510C, 3520C	Minor Revision Date: None

13.3 USACE

13.3.1 The pesticide LCS must contain all single component target analytes.

13.3.2 The LCS is prepared by adding 1.0 mL of AFCEE pesticide spike to each applicable matrix.

13.4 HAZWRAP, NJDEP

13.4.1 Internal chain of custody forms must be used for all extracts.

14.0 SAMPLE DISPOSAL

14.1 Dispose of all wastes per the Waste Disposal Plan SOP A.10.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Digestion of Soil Samples By Method 3050B Using Block Digestion			
Reviewer: J. Warf		Peer Reviewer: F. Lindauer	
Implementation Date: 5/17/01		Last Annual Review Date: 5/16/01	
Revision: 1	Status: Final	Method: 3050B	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the digestion procedure for preparation of soils/sludges for analysis by ICP by SW-846 Method 3050B using a block digestor.

2.0 METHOD SUMMARY

2.1 A representative 0.5-1.0 gram sample is digested with additions of acid and peroxide for ICP.

2.2 This method will solubilize and hold in solution only minimal concentrations of barium in the presence of free sulfate. For the analysis of barium in samples having varying and unknown concentrations of sulfate, analysis should be completed as soon as possible after sample preparation.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and lab coat. See the E & E, Inc., Analytical Service Center (ASC) Chemical Hygiene Plan (CHP) located in the QA/QC library island, shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Method 3050B, Rev. 2, 12/96, SW836 3rd Ed., Update III, June 1997.
4.2 Training SOP QA.5, A.23, and A.29.
4.3 Data Review SOP A.25.
4.4 Laboratory Quality Assurance Manual.
4.5 MDL SOP A.18.
4.6 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS AND ACRONYMS

5.1 See the standard list in the Quality Assurance Manual (QAM).

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 If any part of the bottom of the *Digi*tube goes dry during the digestion process, the sample must be reprepared and redigested.

6.2 **Oils and petroleum products** need special preparation. **See Supervisor**.

7.0 INSTRUMENTATION AND EQUIPMENT

Class A pipettes
Ribbed *Digi*bloc watch glasses
Balance capable of weighing to ± 0.01g
*Digi*bloc 3000 by SCP Science
Wiretrol® pipettes or syringes
Glass beads, ≤1-mm diameter or Teflon® boiling chips
Certified thermometers
*Digi*tubes – 50-mL polypropylene tubes, Class A tolerant, with screw caps

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standards, Reagents, and Consumables

Table 8-1

SUMMARY OF STANDARDS AND REAGENTS

Description	Source	Concentration
Ag Reference Stock	VHG Laboratories or equivalent	1000 µg/mL
Hydrogen peroxide (H_2O_2), metals grade	Baker or equivalent	30%
MS1/LCS A	SCP Science or equivalent	Ca , K, Mg, Na @ 1000 µg/mL Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn @ 100 µg/mL
MS2/LCS B	SCP Science or equivalent	Sn, Zr, Ti @ 100 µg/mL
Metals Grade HNO_3	Baker Instra-analyzed or equivalent	Concentrated
Metals Grade HCl	Baker Instra-analyzed or equivalent	Concentrated
Revision: 1	Status: Final	Method: 3050B	Minor Revision Date: None

8.2 Standard Solution Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	Preparation Instructions	Final Concentration
1:1 Nitric Acid	HNO_3	500 mL ASTM Type II water into 1000-mL flask and add 500 mL concentrated nitric acid slowly.	1:1 Nitric acid
MS3/LCS Ag	Ag Reference Stock	In a 100-mL volumetric flask containing ASTM Type II water and 6 mL concentrated HNO_3, transfer 0.5 mL of Ag Reference Stock. Dilute to volume with ASTM Type II water. Store in amber bottle. Prepare monthly.	Ag @ 5 µg/mL
Revision: 1	Status: Final	Method: 3050B	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Container Type and Preservation
Solid	Standard	180	Plastic or glass, refrigerate 2-6 C
Solid	NYSDEC	180	Plastic or glass, refrigerate 2-6 C
Revision: 1	Status: Final	Method: 3050B	Minor Revision Date: None

10.0 PROCEDURE

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Balance	Out of calibration	Check with Class I weights	Daily
*Digi*Bloc 3000	Not heating	Check electrical connection. Check to see if bloc is on. Monitor with certified thermometer 95 ±5 C.	Daily
Revision: 1	Status: Final	Method: 3050B	Minor Revision Date: None

10.1 Enter sample information into the digestion logbook and Khemia as samples are prepared, including texture and artifacts for solid sample.

10.2 Mix the sample thoroughly with a spatula to achieve homogeneity. Weigh 0.5 g to 1.0 g of wet sample to the nearest 0.01 g and transfer to a labeled D*igi*tube. A larger sample size should be used for samples with low percent solids as long as digestion is complete.

10.3 Prepare QC samples according to Table 12-1.

10.4 Add 5 mL of 1:1 HNO_3 and cover with a ribbed *Digi* watch glass. Place in D*igi*bloc and heat sample to 95 ±5 C. Monitor each block with a thermometer designated to each block. Record block temperature. Reflux for 10-15 minutes without boiling.

10.5 Allow the sample to cool, add 2.5 mL of concentrated nitric acid, replace the ribbed watch glass. If brown fumes are generated, and indicating oxidation of the sample by nitric acid, repeat this step by adding additional 2.5 mL aliquots of concentrated nitric acid until no brown fumes are given off.

10.6 Place sample in a D*igi*bloc at least 95 ±5 C and allow to reflux for 30 minutes to ensure complete oxidation.

10.7 With the ribbed watch glass on, allow solution to evaporate to 5 mL without boiling. Maintain a covering of solution over the bottom (or heat of at least 95 ±5 C without boiling for 2 hours).

10.8 Cool the sample and rinse inside walls of digitube with 2 mL of ASTM Type II water.

10.9 Add 1 mL of 30% hydrogen peroxide. Cover the sample with a ribbed watch glass and place in *Digi*tube to start the peroxide reaction. Do not allow solution to bubble vigorously as sample loss may occur.

10.10 Continue to add 30% hydrogen peroxide in 1-mL aliquots with gentle warming until effervescence is minimal or until unchanged. Note: Do not add more than a total of 5 mL 30% hydrogen peroxide.
10.11 Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate until the volume is reduced to approximately 5 mL or heat at 95 ±5 C without boiling for 2 hours. Maintain a covering of solution over the bottom of the vessel at all times.
10.12 Add 5 mL of concentrated hydrochloric acid. Cover with a ribbed watch glass.
10.13 Reflux an additional 15 minutes at 95 ±5 C. Cool.
10.14 Dilute with ASTM Type II water to a final volume of 50 mL. Put a screw cap on it, place tubes from one batch in one box. Identify the box with digestion batch number and deliver the entire batch (box) to the analysis area. Relinquish custody of the batch to an approved analyst.
10.15 Note: If Sb, Ba, Pb, and Ag show historical low recoveries, Section 10.15 may be used to improve the solubilities and recoveries of these analytes when necessary, as an alternative to the above procedure.
10.15.1 Add 1.25 mL concentrated nitric acid and 5 mL of concentrated hydrochloric acid to a 0.5 g to 1.0 g of sample (wet weight). Cover with a ribbed watch glass and place in heating bloc and reflux for 15 minutes.
10.15.2 Filter the digestate through Whatman #41 filter paper (or equivalent) into a 50-mL *Digi*tube. Wash the filter paper, while still in funnel, with no more than 2.5 mL of hot (95 ±5 C) ASTM Type II water. Collect washings in the same 50-mL *Digi*tube.
10.15.3 Remove the filter paper and residue from the funnel and place them back into the appropriate 50-mL *Digi*tube. Add 2.5 mL of concentrated hydrochloric acid and place back in the *Digibloc*. Heat at least 95 C (but not boiling) until the filter paper dissolves. Remove the beaker from the heating source and wash the cover and sides with ASTM Type II water, filter the residue and collect the filtrate in the same 50-mL *Digi*tube. Allow filtrate to cool then dilute to volume. Note: If precipitation occurs in the flask upon cooling, do not dilute to volume. Add 5 mL of concentrated hydrochloric acid to dissolve the precipitate. After precipitate is dissolved, dilute to volume with ASTM Type II water.
10.15.4 Label the tube, put a screw cap on it, place tubes from one batch in one box. Identify the box with digestion batch number and deliver the entire batch (box) to the analysis area. Relinquish custody of the batch to an approved analyst.

11.0 DATA REDUCTION/EVALUATION/REPORTING

N/A

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1.1

QUALITY CONTROL SAMPLES - ROUTINE

QC Type	Frequency	Preparation Instructions
Preparation blank	1 per preparation batch (20 samples or less)	0.5 g glass beads or Teflon® boiling chips processed as sample, including the addition of 10 mL H_2O_2

Table 12-1.1

QUALITY CONTROL SAMPLES - ROUTINE

QC Type	Frequency	Preparation Instructions
LCS	1 per preparation batch of 20 samples or less	Add 0.5 mL each of MS1/LCS A , MS2/LCS B, and MS3/LCS Ag stocks to 0.5 g glass beads or Teflon® boiling chips and process. Resulting concentrations are: Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn, Zr @ 100 mg/kg Ca, Mg, K, Na @ 1000 mg/kg Ag @ 50 µg/L (5 mg/kg)
Matrix Spike/ Matrix Spike Duplicate	1 set per batch of 20 or fewer samples.	Add 0.5 mL each of MS1/LCS A, MS2/LCS B, and MS3/LCS Ag into 0.5 g of sample and process. Resulting concentrations: Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn, Zr @ 100 mg/kg Ca, Mg, K, Na @ 1000 mg/kg Ag @ 5 mg/kg
LCS-Solid Purchased	1 per preparation batch if specified by client. Replaces LCS above when specified.	0.5 g purchased solid standard processed.
Revision: 1	Status: Final	Method: 3050B | Minor Revision Date: None

Table 12-1.2

QUALITY CONTROL SAMPLES – AFCEE

QC Type	Frequency	Preparation Instructions
Preparation blank	1 per preparation batch (20 samples or less)	0.5 g glass or Teflon® boiling chips processed as sample, including the addition of 10 mL H_2O_2
Laboratory Control Sample (LCS)	1 per preparation batch of 20 samples or less	0.25 mL each of MS1/LCS A, MS2/LCS B, and 0.5 mL of MS3/LCS Ag stocks into 0.5 g glass beads or Teflon® boiling chips and processed as sample. Resulting concentrations: Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Mo, Mn, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn, Zr @ 50 mg/kg Ca, Mg, K, Na @ 500 mg/kg Ag @ 5 mg/kg

Table 12-1.2

QUALITY CONTROL SAMPLES – AFCEE

QC Type	Frequency	Preparation Instructions
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 set per preparation batch of 20 samples or less	0.25 mL each of MS1/LCS A, MS2/LCS B, and 0.5 mL of MS3/LCS Ag stocks into 0.5 g sample and processed. Resulting concentrations: Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn, Zr @ 50 mg/kg Ca, Mg, K, Na @ 500 mg/kg Ag @ 5 mg/kg

Revision: 1	Status: Final	Method: 3050B	Minor Revision Date: None

13.0 SPECIAL PROJECT REQUIREMENTS

13.1 ACE

13.1.1 Prepare LCS using glass beads or Teflon® chips. Perform matrix QC on ACE sample.

13.2 AFCEE

13.2.1 LCS is LCS stock spiked onto glass beads of ≤ 1-mm diameter or Teflon® boiling chips.
13.2.1.1 Spike at ≤ midpoint of high calibration standard.
13.2.2 MS/MSD on AFCEE sample.
13.2.2.1 MS and MSD spiked at ≤ midpoint of high calibration standard.
13.2.3 A batch is 20 environmental samples. These 20 samples *include* the MS/MSD.
13.2.4 The sample weight must be adjusted to compensate for percent moisture prior to digestion.
13.2.5 Use internal COC form F-026 for digestates.

13.3 North Carolina DENR

13.3.1 North Carolina regulations require a duplicate every 10 samples.
13.3.2 All spiking volumes must be 1 mL.

13.4 NJDEP

13.5.1 The digestion of solid samples containing concentrations of silver >50 mg/kg (approximately 500 µg/L in digestate) should be treated such that a smaller portion of solid sample is digested to bring the raw value in the digestate to below 500 µg/L before conversion. The digestion of tin from solid samples should be prepared using aliquots <1 g when determined sample concentrations exceed 1% (10,000 mg/kg).

14.0 SAMPLE DISPOSAL

14.1 Acid waste container is located in metals preparation area.
14.2 Samples and digestate are returned to sample management after use.
14.3 For detailed waste disposal see SOP A.10, Waste Disposal.

15.0 EXAMPLE FORMS

N/A

END OF SOP

TITLE: Preparation for Mercury in Soil Samples by Method 7471A Using Block Digestion			
Reviewer: Pat Rosiek		Peer Reviewer: F. Lindauer	
Implementation Date: 3/29/01		Last Annual Review Date: 3/19/2001	
Revision: 1	Status: Final	Method: SW-846, 7471A	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP directs procedures for the preparation of solid samples for the subsequent analysis of mercury by manual cold vapor atomic absorption using SW-846 procedures.

2.0 METHOD SUMMARY

2.1 A portion of soil is digested with concentrated sulfuric and nitric acids and potassium permanganate. Mercury in the digestate is reduced to elemental mercury with stannous chloride (SOP Metals.67) and measured by cold vapor atomic absorption spectrophotometry.

2.2 This procedure utilizes a block digestion system that allows use of lesser sample and reagent volumes.

2.3 Deviation

2.3.1 A 0.2 µg/L standard replaces the 2 µg/L standard described in the method. This allows one curve to be used for all mercury analyses and provides a standard at the reporting level.

3.0 HEALTH AND SAFETY

3.1 All laboratory personnel should be wearing laboratory coats, gloves, and safety glasses. Refer to the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in QA/QC library island, shelf 2.

3.2 Samples known to contain mercury should only be handled in a fume hood.

3.3 Pollution Prevention – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Method 7471A, (Rev 1, September 1994), SW846 Update III, June 1997.
4.2 *Digi*Bloc 3000 Digestion System Operation Manual, 1999.
4.3 Training SOP QA.5, A.23, and A.29.
4.4 Data Review SOP A.25.
4.5 Laboratory Quality Assurance Manual.
4.6 MDL SOP A.18.
4.7 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 MDL – Method Detection Limit as determined by 40 CFR part 136, Appendix B. Methods detection limits must be determined yearly for both water and soil matrices. See SOP A.18 for procedure.

5.2 PQL – Practical Quantitation Limit is the concentration above the MDL which can be reasonably obtained and is used as the limit of reporting.

5.3 CAL BLK (CCB) – Continuing Calibration Blank.

5.4 **CCV** – Continuing Calibration Verification. This is a check sample approximately midpoint from the calibration standards.
5.5 **MBS** – Method Blank (Soil).
5.6 **ICV** – Initial Calibration Verification, independent of calibration standards verifies calibration up to first CCV analyzed.
5.7 **ICB** – Initial Calibration Blank.
5.8 **LCS, LCSS** – Laboratory Control Sample Soil.
5.9 **MS** – A matrix spike is a predigestion spiked sample prepared with batch.
5.10 **MSD** – A matrix spike duplicate prepared with batch.
5.11 **DUP** – A duplicate sample prepared with batch.
5.12 **LCSD – A** Laboratory Control Sample Duplicate.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Certain organic compounds will absorb at the wavelength, causing interferences.
6.2 Elemental chlorine native to the samples or resulting from oxidation of chloride will cause a positive interference at 253.7 nm.

7.0 INSTRUMENTATION AND EQUIPMENT

Wiretrol® disposable pipettes
Class A pipettes
Syringes
*Digi*Bloc 3000 by SCP Science to 95 C
50-mL polypropylene digitubes with screw caps
Digi watch cover
Balance capable of weighing to $\pm$0.01g
Teflon® beads, $\leq$1-mm diameter
Thermometer

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard, Reagents, and Consumables

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Mercury (Hg) stock calibration standard	Fisher or equivalent	1000 mg/L Hg
Mercury (Hg) stock reference standard	CPI or equivalent (different lot from that used for calibration standards)	1000 mg/L Hg
Sulfuric acid (H_2SO_4) – suitable for Hg analysis	Baker or equivalent	Concentrated
Nitric acid (HNO_3) – suitable for Hg analysis	Baker or equivalent	Concentrated
Potassium permanganate ($KMnO_4$) – reagent grade – suitable for Hg analysis	Baker or equivalent	Neat

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Hydrochloric acid (HCl) – suitable for Hg analysis	Baker or equivalent	Concentrated
Aqua Regia (nitric acid +HCL mix)	Baker or equivalent	Concentrated mix
Sodium chloride (NaCl) – suitable for Hg analysis	Fisher or equivalent	Neat
Hydroxylamine hydrochloride	Fisher or equivalent	Neat
Teflon® beads	Fisher or equivalent	N/A

Revision: 1	Status: Final	Method: SW-846, 7471A	Minor Revision Date: None

Note: Reagents must be "suitable for mercury analysis" grade.

8.2 Standard Solution Preparation

All reagents **must** state that they are "Suitable for Mercury Determination." Stir all solutions with a magnetic stirrer to dissolve and cool.

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Added to ____	Final Concentration
Hg Intermediate Calibration Standard	Hg Stock Calibration Standard	To ASTM Type II water, in a 100-mL volumetric flask, add 1.0 mL of Hg Stock Calibration Standard and add 2 mL of concentrated HNO_3 and dilute to mark. **Prepare every 6 months or when necessary.**	10 µg/mL
Hg Working Calibration Standard	Hg Intermediate Calibration Standard	To ASTM Type II water, in a 100-mL volumetric flask, add 1.0 mL of Hg Intemediate Calibration Standard and add 2 mL of concentrated HNO_3 and dilute to mark. **Prepare daily.**	0.1 µg/mL
Hg Intermediate Reference Standard	Hg Stock Reference Standard	To ASTM Type II water, in a 100-mL volumetric flask, add 1.0 mL Hg Stock Reference Standard and add 2 mL of concentrated HNO_3 and dilute to mark. **Prepare every 6 months or when necessary.**	10 µg/mL
Hg Working Reference Standard	Hg Intermediate Reference Standard	To ASTM Type II water, in a 100-mL volumetric flask, add 1.0 mL of Hg Intermediate Reference Standard and add 2 mL of concentrated HNO_3 and dilute to mark. **Prepare daily.**	0.1 µg/mL

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Added to ____	Final Concentration
ICV	Hg Working Reference Standard	In the digitube – pipet 500 µL of Hg Working Reference Standard. Add 5 mL ASTM Type II water and process like a sample.	1.0 µg/L
Potassium permanganate solution	$KMnO_4$	Add 100 g $KMnO_4$ to 2000 mL ASTM Type II water. **Prepare monthly.**	5% w/v
Sodium chloride and hydroxylamine hydrochloride solution	NaCl hydroxylamine hydrochloride	Add 240 g hydroxylamine hydrochloride and 240 g NaCl to 2000 mL ASTM Type II water. **Prepare monthly**.	12% w/v each
Aqua regia	HCl, concentrated HNO_3, concentrated	Prepare before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3 in a fume hood.	3:1 v/v HCl/HNO_3

Revision: 1	Status: Final	Method: SW-846, 7471A	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Solid	Standard	28	28	8 oz. jar
Solid	NYSDEC (1)	26 from VTSR	26 from VTSR	8 oz. jar

Revision: 1	Status: Final	Method: SW-846, 7471A	Minor Revision Date: None

10.0 PROCEDURE

10.1 Instrument Conditions and Maintenance

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Balance	Out of calibration	Check with Class I weight	Daily
*Digi*Bloc 3000	Not heating	Check electrical connection. Make sure instrument is on. If all else fails, call E & E Equipment Services Center to repair.	As needed

Revision: 1	Status: Final	Method: SW-846, 7471A	Minor Revision Date: None

10.2 Preparation of Solid Samples, Standards, and QC Samples

10.2.1 Weigh approximately 0.1g of sample in triplicate into a polypropylene digitube. Total weight should be approximately 0.3 g. Add 5 mL ASTM Type II water.

10.2.2 Prepare calibration standards according to Table 10-2 and QC samples according to Table 12-1.

10.2.3 Prepare standards daily.

10.2.4 Because the Leeman Mercury Analyzer uses only a portion of the total preparation volume for analysis, it is **IMPERATIVE** that all standards, QC samples, and samples be treated in similar fashion such that the final volume is the same for each.

Table 10-2

CALIBRATION STANDARDS

Volume of 0.1 µg/mL Hg Working Calibration Standard Added to Digestion Tube	Volume (mL) of ASTM Type II Water to add to Reach Approximate Starting Volume of 5 mL	Final Concentration Based on 50 mL (µg/L)	Equivalent Soil Concentration for 0.3 g Sample (mg/kg)
0	5	0 (ICB, CCB)	0
100 µL	5	0.2	0.033
250 µL	5	0.5	0.083
500 µL	5	1.0	0.167
2.5 **mL**	2.5	5.0 (CCV)	0.833
5 **mL**	0	10.0	1.67

Revision: 1	Status: Final	Method: SW-846, 7471A	Minor Revision Date: None

10.2.5 Add 2.5 mL Aqua Regia.

10.2.6 Put samples in *Digi*Bloc at 95 C ±2 C for 2 minutes. The *Digi*Bloc must be in a fume hood.

10.2.7 Remove samples after 2 minutes, then cool.

10.2.8 After samples have cooled, add 25 mL of ASTM Type II water and add 7.5 mL potassium permanganate solution.

10.2.9 Heat in *Digi*Bloc for 30 minutes with *Digi*Bloc watch covers.

10.2.10 After 30 minutes, remove samples from *Digi*Bloc, let cool, and put screw cap on.

10.3 Analysis

10.3.1 Immediately before analysis, reduce potassium permanganate by adding 3.0 mL sodium chloride and hydroxylamine hydrochloride solution and swirling.

10.3.2 Bring to full volume of 50 mL in tube with ASTM Type II water.

10.3.3 Proceed to analysis SOP Metals.67.

11.0 DATA REDUCTION/EVALUATION/REPORTING

NA

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions
Initial Calibration Verification (ICV)	One per calibration	See Table 8-2.
Initial Calibration Blank (ICB)	One per calibration after ICV	See Table 10-2.
Continuing Calibration Verification (CCV)	After ICB, every 10 analyses, and at end of analysis	See Table 10-2.
Continuing Calibration Blank (CCB)	After each CCV	See Table 10-2.
Method Blank Soil (MB) (S)	1 per batch of 20 or fewer samples	Use approximately 0.3 g Teflon® beads. Add 5 mL ASTM Type II water and process.
Laboratory Control Sample Solid (LCSS)	1 per batch of 20 or fewer samples	Add 1000 µL of the Hg 0.1 µg/mL Working Reference Standard to approximately 0.3 g of Teflon® beads. Add 5 mL ASTM Type II water and process. (Concentration of solution is 2 µg/L, based on 0.3 g is 0.333 mg/kg.)
Matrix Spike (MS)	1 per 20 samples (or less)	Spiked Sample. Prepare a spiked sample by adding 1000 µL of the Hg 0.1 µg/mL Working Reference Standard into the sample (2 µg Hg added). Added spike is equivalent to 0.333 mg/kg in a 0.3 g sample.
Matrix Spike Duplicate (MSD)	With every MS	Spiked Sample. Prepare a spiked sample by adding 1000 µL of the Hg 0.1 µg/mL Working Reference Standard into the sample (2 µg Hg added). Added spike is equivalent to 0.333 mg/kg in a 0.3 g sample.
Duplicate (DUP)	If client specified	Duplicate Sample. Prepare exactly as original sample.

Revision: 1	Status: Final	Method: SW-846, 7471A	Minor Revision Date: None

13.0 SPECIAL PROJECT REQUIREMENTS

Client-Specific Quality Control Requirements

13.1 Jacobs Otis

13.1.1 LCS and LCSD required.

13.1.2 Internal COC required.

13.3 AFCEE

13.3.1 See Table 7.2.23-3 of the AFCEE QAPP version 3.0, March 1998, pages 7-148 to 7-150 incorporated by reference.

13.3.2 Perform a MS/MSD per 20 Air Force samples per matrix. Spike recovery must be within 77% - 120%; RPD $\leq$ 25.

13.3.3 Perform a LCS per analytical batch (independent of ICV).

13.3.4 For each new or unusual matrix perform a 1:4 serial dilution. Percent difference must be $\pm$ 10% of undiluted sample result.

13.3.4.1 If the test fails, perform recovery test. Recovery of added spike must be 85% - 115%. If this test also fails, perform the method of standard addition.

13.3.4.2 Use glass beads $\leq$ -1 mm diameter as the solid matrix to prepare solid LCS and solid PBS.

13.3.4.3 A batch is 20 environmental samples. These 20 samples *include* the MS/MSD or MS/MD.

13.3.5 Internal COC required.

14.0 SAMPLE AND WASTE DISPOSAL

14.1 For more information see Waste Disposal SOP A.10.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Digestion of Waters by Method 3010A Using Block Digestion for ICP Analysis			
Reviewer: J. Warf		Peer Reviewer: F. Lindauer	
Implementation Date: 5/17/01		Last Annual Review Date: 5/16/01	
Revision: 1	Status: Final	Method: 3010A	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP details the preparation of aqueous samples including TCLP and similar leach test extracts for analysis of total metals by ICP using a block digestor.

2.0 METHOD SUMMARY

2.1 Samples are digested by adding a mixture of nitric acid to the sample in a disposable screw top polypropylene tube that is refluxed in a heated graphite block. The digestate is reduced to a low volume. For ICP analysis, hydrochloric acid is added to the sample, then refluxed again. Samples are then brought up to a volume.

2.2 This method will solubilize and hold in solution only minimal concentrations of barium in the presence of free sulfate. For the analysis of barium in samples having varying and unknown concentrations of sulfate, analysis should be completed as soon as possible after sample preparation.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Methods 3010A (ICP), SW-846 Update III, June 1997.
4.2 Training SOP QA.5, A.23, and A.29.
4.3 Data Review SOP A.25.
4.4 Laboratory Quality Assurance Manual (L-001).
4.5 MDL SOP A.18.
4.6 Corrective Action Procedure, QA.1

5.0 DEFINITIONS/ACRONYMS

5.1 **LCS** - Laboratory Control Sample.
5.2 For more information see the generic list in the Quality Assurance Manual.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 If any part of the bottom of the tube goes dry during the digestion process, the sample must be reprepared and redigested.

6.2 **Waters containing oils and petroleum products** need special preparation techniques. See Supervisor.

7.0 INSTRUMENTATION AND EQUIPMENT

*Digi*Bloc 3000 by SCP Science
Certified thermometers
ASTM Type II water
*Digi*Bloc ribbed watch glasses
Class A pipettes
Wiretrol® pipette or syringes
*Digi*tubes – 50-mL polypropylene tubes, Class A tolerant, with screw caps
Whatman filter paper #41, Fisher or equivalent

8.0 REAGENTS AND MATERIALS

8.1 **See Table 8-1, Summary of Standard, Reagents, and Consumables**

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Ag Reference Stock	VHG Laboratories or equivalent	1000 µg/mL
MS1/LCS A	SCP Science or equivalent	Ca , K, Mg, Na @ 1000 µg/mL Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn @ 100 µg/mL
MS2/LCS B	SCP Science or equivalent	Sn, Zr, Ti @ 100 µg/mL
Metals Grade HNO_3	Baker Instra-analyzed or equivalent	Concentrated
Metals Grade HCl	Baker Instra-analyzed or equivalent	Concentrated
TCLP Spike	SCP Science or equivalent	Ba @ 5000 µg/mL; As, Pb, Se @ 500 µg/mL; Ag, Cr @ 100 µg/mL

Revision: 1	Status: Final	Method: 3010A	Minor Revision Date: None

8.2 Standard Solution Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
1:1 Hydrochloric Acid	HCl	500 mL ASTM Type II water into 1000 mL flask and add 500 mL concentrated HCl slowly	1:1 HCl
MS3/LCS Ag	Ag Reference Stock	In a 100-mL volumetric flask containing ASTM Type II water and 6 mL concentrated HNO_3, transfer 0.5 mL of Ag Reference Stock. Dilute to volume with ASTM Type II water. Store in amber bottle. Prepare monthly.	Ag @ 5 µg/mL
Revision: 1	Status: Final	Method: 3010A	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Water	Standard	6 months	6 months	125 mL Poly, < 2 pH HNO_3
	NYSDEC (1)	6 months	6 months	125 mL Poly, < 2 pH HNO_3
Revision: 1	Status: Final	Method: 3010A		Minor Revision Date: None

10.0 PROCEDURE

10.1 Shake the sample container to ensure a homogenous mixture. Measure 50 mL of sample using the calibrated line on the *Digi*tube. Set up and prepare the appropriate QC samples as per Table 12-1.

10.2 Enter sample information into digestion logbook and LIMS (Khemia) as samples are prepared.

10.3 Add 1.5 mL of concentrated HNO_3. Place the tubes in a block digestor without caps or watch glasses. Monitor each *Digi*bloc used with a thermometer. Maintain the temperature of the solution between 95 $\pm$5 C. Record block temperature. Make sure sample does not go to dryness.

10.4 After 1½ hours, remove from block, cool. Add an additional 1.5 mL of concentrated nitric acid and continue heating.

10.5 Continue the digestion, with the addition of nitric acid if necessary until complete (no change in color of digestate upon continued refluxing). If a sample goes to dryness, discard and reprepare the sample.

10.6 Cool the sample, add 5 mL 1:1 hydrochloric acid, reflux for 15 minutes, then cool.

10.7 Dilute with ASTM Type II water to a final volume of 50 mL. Put a screw cap on it, place tubes from one batch in one box. Identify the box with digestion batch number and deliver the entire batch (box) to the analysis area. Relinquish custody of the batch to an approved analyst.

Instrument Conditions and Maintenance

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
*Digi*Bloc 3000	Not heating.	Monitor and record 95 ±5 C with certified thermometer. Check electrical connection. Check to see if block is on.	Daily

Revision: 1	Status: Final	Method: 3010A	Minor Revision Date: None

11.0 DATA REDUCTION/EVALUATION/REPORTING

N/A

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Routine Quality Control Samples

Table 12-1.1

QUALITY CONTROL SAMPLES - ROUTINE

QC Type	Frequency	Preparation Instructions
Preparation Blank	1 per preparation batch of 20 samples or less.	50 mL ASTM Type II water processed as a sample.
LCS	1 per preparation batch of 20 samples or less.	Add 0.5 mL each of MS1/LCS A, MS2/LCS B, and MS3/LCS Ag stocks to 50 mL ASTM Type II water and process. Resulting concentrations are: Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn, Zr @ 1000 µg/L; Ca, Mg, K, Na @ 10,000 µg/L; Ag @ 50 µg/L
Matrix Spike/ Matrix Spike Duplicate	1 set per batch of 20 or fewer samples.	Add 0.5 mL each of MS1/LCS A, MS2/LCS B, and MS3/LCS Ag into 50-mL of sample and process. Resulting concentrations: Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn, Zr @ 1000 µg/L; Ca, Mg, K, Na @ 10,000 µg/L; Ag @ 50 µg/L

Revision: 1	Status: Final	Method: 3010A	Minor Revision Date: None

Table 12-1.2

QUALITY CONTROL SAMPLES – AFCEE

QC Type	Frequency	Preparation Instructions
Preparation Blank	1 per preparation batch of 20 samples or less.	50 mL ASTM Type II water processed as a sample.
Laboratory Control Sample (LCS)	1 per preparation batch of 20 samples or less	0.25 mL each of MS1/LCS A, MS2/LCS B, and 0.5 mL of MS3/LCS Ag stocks into 50 mL ASTM Type II water and processed as sample. Resulting concentrations: Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn, Zr @ 500 µg/L; Ca, Mg, K, Na @ 5000 µg/L; Ag @ 50 µg/L
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 set per preparation batch of 20 samples or less	0.25 mL each of MS1/LCS A, MS2/LCS B, and 0.5 mL of MS3/LCS Ag stocks into 50 mL sample and processed. Resulting concentrations: Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn, Zr @ 500 µg/L; Ca, Mg, K, Na @ 5000 µg/L; Ag @ 50 µg/L

Revision: 1	Status: Final	Method: 3010A	Minor Revision Date: None

Table 12-1.3

QUALITY CONTROL SAMPLES - TCLP

QC Type	Frequency	Preparation Instructions
Preparation Blank	1 per preparation batch of 20 samples or less	50 mL ASTM Type II water processed as a sample.
TCLP Matrix Spike/Matrix Spike Duplicate	1 per preparation batch of 20 TCLP samples or less	0.5 mL of TCLP spike to 50 mL of selected TCLP sample digestate. Resulting concentrations: Ba @ 50,000 µg/L; As, Pb, Se @ 5000 µg/L; Ag, Cr @ 1000 µg/L
TCLP LCS	1 per preparation batch of 20 samples or fewer	0.5 mL of TCLP spike to 50 mL of ASTM Type II water. Resulting concentrations: Ba @ 50,000 µg/L; As, Pb, Se @ 5000 µg/L; Ag, Cr @ 1000 µg/L

Revision: 1	Status: Final	Method: 3010A	Minor Revision Date: None

13.0 SPECIAL PROJECT REQUIREMENTS

13.1 AFCEE

13.1.1 LCS in ASTM Type II water for aqueous sample.

13.1.1.1 Spike at ≤ midpoint of high calibration standard.

13.1.2 MS/MSD on AFCEE Sample.

13.1.2.1 MS and MSD spiked at ≤ midpoint of high calibration standard.

13.1.2.2 A batch is 20 environmental samples. These 20 samples *include* the MS/MSD or MS/MD.

13.1.3 Use internal COC from F-026 for digestates.

13.2 U.S. Army Corps of Engineers

13.2.1 For USACE water samples: Do not filter any samples unless cleared with the laboratory project manager. If one sample requires filtration, then filter all samples for the project in the batch. Filter all QC samples. If all samples are filtered, indicate this in the digestion logbook. An alternate procedure is to let the sample settle by gravity. Since samples are not transferred from a beaker and brought to volume in a volumetric flask in this block digestion procedure, filtering is not anticipated.

13.2.2 Perform matrix QC on ACE sample.

13.3 North Carolina DENR

13.3.1 North Carolina regulations require a duplicate every 10 samples.

13.3.2 All spiking volumes must be 1 mL.

13.4 NJDEP

13.4.1 This method is suitable for preparation of aqueous samples containing silver concentrations up to 100 µg/L. For the analysis of water samples containing higher concentrations of silver, succeeding smaller volume, well mixed aliquots must be prepared until the analysis solution contains <100 µg/L silver.

14.0 SAMPLE DISPOSAL

14.1 A drum for acid waste is located in the metals preparation and metals analysis areas.

14.2 For more information see Waste Disposal SOP, A.10.

15.0 EXAMPLE FORMS

N/A

END OF SOP

13.0 SPECIAL PROJECT REQUIREMENTS

Client-Specific Quality Control Requirements

13.1 Jacobs; Otis

13.1.1 Requires LCS/LCSD.

13.1.2 Internal COC required.

13.2 AFCEE

13.2.1 See Table 7.2.23.2 and 7.2.23.3 of the AFCEE QAPP version 3.0, March 1998, pages 7-147 to 7-150 incorporated by reference.

13.2.2 Perform a MS/MSD per 20 Air Force samples per matrix. Spike recovery must be within 77-120%; RPD $\leq$ 15 (AFCEE QAPP 3.0).

13.2.3 Perform an LCS per analytical batch (independent of ICV).

13.2.4 For each new or unusual matrix perform a 1:4 serial dilution. Percent difference must be $\pm$ 10% of undiluted sample result.

13.2.4.1 If the test fails, perform recovery test. Recovery of added spike must be 85-115%. If this test also fails, perform the method of standard addition.

13.2.4.2 A batch is 20 environmental samples. These 20 samples *include* the MS/MSD or MS/MD.

13.2.5 Internal COC required.

13.3 IDOT

13.3.1 TCLPs or SPLPs use 10 µL of extract and dilute to 25 mL mark.

14.0 SAMPLE DISPOSAL

14.1 For more information, see Waste Disposal SOP, A.10.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Preparation for Mercury in Water Samples by Method 7470A or 245.1 Using Block Digestion			
Reviewer: F. Lindauer		Peer Reviewer: P. Rosiek	
Implementation Date: 3/29/01		Last Annual Review Date: 3/15/2001	
Revision: 1	Status: Final	Method: SW-846 7470A, 245.1; TCLP, SPLP; CAL WET	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP directs procedure for the preparation of water samples for mercury (Hg) by manual cold vapor atomic absorption using SW-846 procedures.

1.2 The digestion procedures for Methods 7470A and 245.1 are identical and this procedure may be used for either method designation. The former SOP for Method 245.1 is incorporated into Metals.70.

1.3 This procedure is used for extracts from the TCLP, SPLP, and California Waste Extraction (CAL WET) tests.

2.0 METHOD SUMMARY

2.1 Sample along with concentrated sulfuric acid (H_2SO_4) and concentrated nitric acid (HNO_3) are placed in a polypropylene disposable tube. Potassium permanganate ($KMnO_4$) sufficient to maintain color for 15 minutes, is added and the sample heated 2 hours at 95 C. Mercury is reduced to elemental mercury with stannous chloride (SOP Metals.67), and measured by cold vapor atomic absorption spectrophotometry.

2.2 This procedure utilizes a block digestion system that allows use of lesser sample and reagent volumes. The relative proportions are identical to Method 7470A.

2.3 Deviation: A 0.2 µg/L standard replaces the 2 µg/L standard described in the method. This allows one curve to be used for all mercury analyses and provides a standard at the reporting level.

3.0 HEALTH AND SAFETY

3.1 All laboratory employees should protect themselves at a minimum with safety glasses, protective glove, and a lab coat. For more information see the E & E, Inc., Analytical Services Center, Chemical Hygiene Plan located in the QA library island, shelf 2.

3.2 Mercury vapors may be generated. Use Method 7470A recommended traps or vents.

3.3 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Method 7470A (Rev.1 9/94), SW-846 Update III, June 1997.

4.2 Method 245.1, Rev.3.0, EMMC Version, Methods for the Determination of Metals in Environmental Samples – Supplement I, EPA-600/R-94/111, May 1994.

4.3 *Digi*Bloc 3000 Digestion System Operation Manual, 1999.

4.4 Training SOP QA.5, A.23, and A.29.

4.5 Data Review SOP A.25.

4.6 Laboratory Quality Assurance Manual.

4.7 MDL SOP A.18.

4.8 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 MDL - Method Detection Limit as determined by 40 CFR part 136, Appendix B. Methods detection limits must be determined yearly for both water and soil matrices. See SOP A.18 for procedure.

5.2 PQL - Practical Quantitation Limit is the concentration above the MDL which can be reasonably obtained and is used as the limit of reporting .

5.3 CAL BLK (CCB) - Continuing Calibration Blank.

5.4 CCV (IPC) Continuing Calibration Verification (Method 245.1 term: IPC, Instrument Performance Check). This is a check sample approximately midpoint of the calibration standards.

5.5 MBW (LRB) - Method Blank (Water). (Method 245.1 term: LRB, Laboratory Reagent Blank.)

5.6 ICV - Initial Calibration Verification, independent of the calibration solutions, verifies calibration up to the first CCV analyzed.

5.7 ICB - Initial Calibration Blank.

5.8 LCS, LCSW (LFB) – Laboratory Control Sample Water as needed by client request. (Method 245.1 term: LFB, Laboratory Fortified Blank.)

5.9 MS (LFM) – A matrix spike is a predigestion spiked sample prepared with batch. (Method 245.1 term: LFM, Laboratory Fortified Matrix.)

5.10 MSD – A matrix spiked duplicate prepared with batch.

5.11 DUP – Duplicate sample prepared with batch.

5.12 QCS – Quality Control Sample in Method 245.1 (same as ICV except at midrange).

5.13 LCSD – A Laboratory Control Sample Duplicate.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Certain organic compounds will absorb at the wavelength, causing interferences.

6.2 Elemental chlorine native to the samples or resulting from oxidation of chloride will cause a positive interference at 253.7 nm.

7.0 INSTRUMENTATION AND EQUIPMENT

Wiretrol® disposable pipettes
Class A pipettes
Syringes
*Digi*Bloc 3000 by SCP Science to 95 C
50-mL polypropylene digitubes with screw cap (Digitubes)
Digi watch covers
Thermometer

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard, Reagents, and Consumables.

Table 8-1

SUMMARY OF STANDARDS, REAGENTS, AND CONSUMABLES

Description	Source	Concentration
Mercury (Hg) stock calibration standard	Fisher or equivalent	1000 mg/L Hg
Mercury (Hg) stock reference standard	CPI or equivalent (different lot from that used for calibration standards)	1000 mg/L Hg

Table 8-1

SUMMARY OF STANDARDS, REAGENTS, AND CONSUMABLES

Description	Source	Concentration
Sulfuric acid (H_2SO_4) – suitable for Hg analysis	Baker or equivalent	Concentrated
Nitric acid (HNO_3)– suitable for Hg analysis	Baker or equivalent	Concentrated
Potassium permanganate ($KMnO_4$) reagent grade – suitable for Hg analysis	Baker or equivalent	Neat
Potassium persulfate ($K_2S_2O_8$) – reagent grade – suitable for Hg analysis	Baker or equivalent	Neat
Sodium chloride (NaCl) – suitable for Hg analysis	Fisher or equivalent	Neat
Hydroxylamine hydrochloride	Fisher or equivalent	Neat

Revision: 1	Status: Final	Method: SW-846 7470A, 245.1; TCLP, SPLP; CAL WET	Minor Revision Date: None

Note: Reagents must be "suitable for mercury analysis" grade.

8.2 Standard Solution Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Added to ____	Final Concentration
Hg Intermediate Calibration Standard	Hg Stock Calibration Standard	To ASTM Type II water, in a 100-mL volumetric flask, add 1.0 mL of Hg Stock Calibration Standard and add 2 mL of concentrated HNO_3 and dilute to mark. **Prepare every 6 months or when necessary.**	10 µg/mL
Hg Working Calibration Standard	Hg Intermediate Calibration Standard	Using a syringe or Class A pipette, to ASTM Type II water, in a 100-mL volumetric flask, add 500 µL of Hg Intermediate Calibration Standard and add 2 mL of concentrated HNO_3 and dilute to mark. **Prepare daily.**	0.05 µg/mL
Hg Intermediate Reference Standard	Hg Stock Reference Standard	To ASTM Type II water, in a 100-mL volumetric flask, add 1.0 mL of Hg Stock Reference Standard and add 2 mL of concentrated HNO_3 and dilute to mark. **Prepare every 6 months or when necessary.**	10 µg/mL
Hg Working Reference Standard	Hg Intermediate Reference Standard	Using a syringe or Class A pipette to ASTM Type II water, in a 100-mL volumetric flask, add 500 µL of Hg Intermediate Reference Standard and add 2 mL of concentrated HNO_3 and dilute to mark. **Prepare daily.**	0.05 µg/mL

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Added to ____	Final Concentration
LCSW (LCS) and ICV	Hg Working Reference Standard	To 25 mL ASTM Type II water – in the digitube – pipette 500 µL of Hg Working Reference Standard. **Prepare daily.**	1.0 µg/L
Potassium permanganate solution	$KMnO_4$	Add 100 g $KMnO_4$ to 2000 mL ASTM Type II water. **Prepare monthly.**	5% w/v
Potassium persulfate solution	$K_2S_2O_8$	Add 25g $K_2S_2O_8$ to ASTM Type II water make up to 500 mL. **Prepare monthly.**	5% w/v
Sodium chloride and hydroxylamine hydrochloride solution	NaCl hydroxylamine hydrochloride	Add 240g hydroxylamine hydrochloride and 240g NaCl to 2000 mL ASTM Type II water. **Prepare monthly**.	12% w/v each

Revision: 1	Status: Final	Method: SW-846 7470A, 245.1; TCLP, SPLP; CAL WET	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Water	Standard	28	28	1L Poly, pH <2 with HNO_3
Water	NYSDEC	26 from VTSR	26 from VTSR	1L Poly, pH <2 with HNO_3

Revision: 1	Status: Final	Method: SW-846 7470A, 245.1; TCLP, SPLP; CAL WET	Minor Revision Date: None

10.0 PROCEDURE

10.1 Instrument Conditions and Maintenance

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
*Digi*Bloc 3000	Not heating	Check electrical connection. Make sure instrument is on.	As needed

Revision: 1	Status: Final	Method: SW-846 7470A, 245.1; TCLP, SPLP; CAL WET	Minor Revision Date: None

10.2 Preparation of Samples, Standards, and QC Samples

10.2.1 Shake vigorously the 1-L polyethylene bottle immediately before pouring 25 mL of sample.

10.2.2 Measure out 25 mL of sample and prepare 25 mL of each calibration standard (see Table 10-2) and the associated QC samples (see Table 12-1) and transfer to a clean, polypropylene digi tube.

10.2.3 Prepare standards daily. Use 1.0 mL of sample for TCLP, SPLP, and CAL WET and dilute to 25 mL with ASTM Type II water

10.2.4 Because the Leeman mercury analyzer uses only a portion of the total preparation volume for analysis, it is **IMPERATIVE** that all standards, QC samples, and samples be treated in similar fashion such that the final volume is the same for each.

Table 10-2

CALIBRATION STANDARDS

Volume of 0.05 µg/mL Hg Working Calibration Standard added to Digestion Tube	Volume (mL) of ASTM Type II Water to add	Standard Concentration based on 25 mL (µg/L)
0	Bring to 25-mL mark	0 (ICB, CCB, MB)
100 µL	Bring to 25-mL mark	0.2
250 µL	Bring to 25-mL mark	0.5
500 µL	Bring to 25-mL mark	1.0
2.5 mL	Bring to 25-mL mark	5.0 (CCV)
5 mL	Bring to 25-mL mark	10.0

Revision: 1	Status: Final	Method: SW-846 7470A, 245.1; TCLP, SPLP; CAL WET	Minor Revision Date: None

10.2.3 Add 1.25 mL concentrated H_2SO_4 and 0.625 mL concentrated HNO_3.

10.2.4 Add 3.75 mL of the 5% w/v potassium permanganate solution and mix (up to 3 aliquots to maintain color for 15 minutes). The same amount of permanganate must be used for all samples and standards.

10.2.5 Add 2.0 mL potassium persulfate solution.

10.2.6 Heat in *Digi*Bloc 95 C (± 2 C) for 2 hours with digi watch cover on.

10.2.7 Allow to cool, remove digi watch covers and put screw cap on until ready for analysis.

10.2.8 Make certain all Digitubes are at the same solution level.

10.3 Analysis

10.3.1 Immediately before each standard, QC and sample is analyzed, add 1.5 mL of sodium chloride and hydroxylamine hydrochloride solution and swirl to reduce potassium permanganate.

10.3.2 Proceed to analysis SOP Metals.67.

11.0 DATA REDUCTION/EVALUATION/REPORTING

NA

12.0 QUALITY CONTROL

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions
ICV Initial Calibration Verification	One per calibration	See Table 8-2.
ICB Initial Calibration Blank	One per calibration, after ICV.	See Table 10-2.
CCV Continuing Calibration Verification	After ICB, every 10 analyses and at end of analysis	See Table 10-2.
CCB Continuing Calibration Blank	After each CCV.	See Table 10-2.
MB(Water) Method Blank Water	One per 20 samples or less	See Table 10-2.
Duplicate (DUP)	One per 20 samples (245.1)	Duplicate sample. Prepare a second aliquot of a sample. NOTE: Do not use a field blank for a duplicate.
Matrix Spike (MS)	One per 20 samples or less	Spiked sample. Prepare a spiked sample by adding 500 µL of the Hg 0.05 µg/mL Working Reference Standard into 25-mL sample (0.025 µg Hg added). Added spike is equivalent to 1 µg/L (0.025 mg/L for TCLP, SPLP, CAL WET extracts). Note: Do not use a field blank as a matrix spike.
Matrix Spike Duplicate (MSD)	With every MS	Spiked sample. Prepare a spiked sample by adding 500 µL of the Hg 0.05 µg/mL Working Reference Standard into the 25-mL sample (0.025 µg Hg added). Added spike is equivalent to 1 µg/L (0.025 mg/L for TCLP, SPLP, CAL WET extracts). Note: Do not use a field blank as a matrix spike.
LCSW (LCS) Laboratory Control Sample Water	If specified by client	See Table 8-2.

TITLE: Mercury Analysis			
Reviewer: T. Seymore		Peer Reviewer: None	
Implementation Date: 11/10/99		Last Annual Review Date: 12/4/00	
Revision: 0	Status: Final	Method: SW-846, CLP ILM04.1	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP outlines the procedure for analyzing mercury by the Leeman Mercury Analyzer.

2.0 METHOD SUMMARY

2.1 A digestate is first prepared by the SOP Metals.70 (7470A, 245.1), Metals.71 (7471A), Metals.21 (CLP water), Metals.60 (CLP soil), Metals.66 (NIOSH 6009), or Metals.69 (wipes). Mercury is reduced to elemental mercury by the addition of stannous chloride and measured by cold vapor atomic absorption spectrophotometry.

3.0 HEALTH AND SAFETY

3.1 All laboratory personnel should wear laboratory coats, gloves, and safety glasses with side shields. Refer to the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in QA/QC library island, shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 SW-846 7470A Water Method.
4.2 SW846 7471 Soil Method.
4.3 USEPA Statement of Work for Inorganic Analysis ILM04.1 Water Method.
4.4 USEPA Statement of Work for Inorganic Analysis ILM04.1 Soil Method.
4.5 Method 245.1, Rev. 3.0, EMMC Version, Methods for the Determination of Metals in Environmental Samples, Supplement I, EPA-600/R-94/111, May 1994.
4.6 Mercury Method 6009, NIOSH Manual of Analytical Methods, 4th edition, 8/15/94.
4.7 Mercury One Electronic Notebook.
4.8 AP/PS 200II Mercury Analysis Systems Manual.
4.9 Training SOP QA.5, A.23, and A.29.
4.10 Data Review SOP A.25.
4.11 Laboratory Quality Assurance Manual.
4.12 MDL SOP A.18.
4.13 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 MDL – Method Detection Limit as determined by 40 CFR part 136, Appendix B. Methods detection limits must be determined yearly for both water and soil matrices. See SOP A.18 for procedure.

5.2 PQL – Practical Quantitation Limit is the concentration above the MDL that can be reasonably obtained and is used as the limit of reporting.

5.3 CAL BLK (CCB) – Continuing Calibration Blank.

5.4 CCV (IPC) – Continuing Calibration Verification (Method 245.1 term: Instrument Performance Check). This is a check sample approximately midpoint from the calibration standards.

5.5 PBS, PBW (LRB) – Preparation Blank Soil, Preparation Blank Water, (Method 245.1 term: Laboratory Reagent Blank).

5.6 QCS – Quality Control Sample in Method 245.1, independent of calibration standards, same as ICV except at midrange.

5.7 ICB – Initial Calibration Blank.

5.8 LCSW (LFB), LCSS – Laboratory Control Sample Water (Method 245.1 term: Laboratory Fortified Blank), Laboratory Control Sample Soil.

5.9 MS (LFM) – A matrix spike is a predigestion spiked sample prepared with batch (Method 245.1 term: Laboratory Fortified Matrix).

5.10 DUP – A duplicate sample prepared with batch.

5.11 LCSD – A Laboratory Control Sample Duplicate.

5.12 CRA – CRDL standard (CLP) used to verify linearity near CRDL.

5.13 ICV – Initial Calibration Verification, independent of calibration standards.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Certain organic compounds will absorb at the wave length causing interferences.

6.2 Elemental chlorine from oxidation or chloride will cause a positive interference.

7.0 INSTRUMENTATION AND EQUIPMENT

Leeman PS200II Mercury Analyzer
*Digi*Block 3000
Digitubes
Class A pipettes or syringes
Teflon® tubes
Laboratory balance
Other laboratory glassware

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard, Reagents, and Consumables.

Table 8-1

SUMMARY OF STANDARDS AND REAGENTS

Description	Source	Concentration
Sodium chloride salt (NaCl) – suitable for Hg analysis	Baker or equivalent	Neat
Stannous chloride ($SnCl_2$) – suitable for Hg analysis	Baker or equivalent	Neat
Hydroxylamine HCl - suitable for Hg analysis	Fisher or equivalent	Neat

Revision: 0	Status: Final	Method: SW-846, CLP ILM04.1	Minor Revision Date: None

All other reagents and standards are listed in the appropriate preparation SOP – Metals.70 (7470A; 245.1), Metals.71 (7471A), Metals.21 (CLP water), Metals.60 (CLP soils), Metals.66 (NIOSH 6009), and Metals.69 (wipes).

8.2 PREPARATION OF REAGENTS

8.2.1 All reagents must state that they are "Suitable for Mercury Determination." Stir all solutions with a magnetic stirrer to dissolve and cool. See Table 8-2, Standard and Reagent Preparation.

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Added to ____	Final Concentration
$KMnO_4$	Saturated $KMnO^4$	Add 100 g $KMnO_4$ to 2000 mL ASTM Type II water. **Prepare monthly.**	Saturated
NaCl and Hydroxylamine Hydrochloride	NaCl and Hydroxylamine Hydrochloride for mercury	Add 240 g hydroxylamine hydrochloride and 240 g NaCl to 2000 mL ASTM Type II water. **Prepare monthly**.	12% each
$SnCl_2$	Stannous chloride solution	Prepare a sufficient quantity to allow 5 mL of solution for each determination. Example--for 20 determinations: 10.0 g stannous chloride and 2 mL 0.5N H_2SO_4 into 100 mL ASTM Type II water. **Prepare daily.**	10%

Revision: 0	Status: Final	Method: SW-846, CLP ILM04.1	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Solid	Standard (7471A)	28	28	8-oz. glass
Water	Standard (7470A, 245.1)	28	28	1-L poly, HNO_3, <2 pH
Water	NYSDEC; USEPA CLP	26 VTSR	26 VTSR	1-L poly, HNO_3, <2 pH
Soil	NYSDEC; USEPA CLP	26 VTSR	26 VTSR	8-oz. glass

Revision: 0	Status: Final	Method: SW-846, CLP ILM04.1	Minor Revision Date: None

10.0 PROCEDURE

10.1 Start-up of the Leeman PS200II Mercury Analyzer.

10.1.1 Turn on computer.

10.1.2 Turn on Mercury Analyzer.

10.1.3 Go into Leeman Mercury Analysis program.

10.1.4 Turn on gas nitrogen 99.995% purity at 70 psi.

10.1.5 Main menu – type "I" to call up instrument.

10.1.6 Type "O" to call up operation.

10.1.7 Turn on gas.

10.1.8 Turn on rate or pump.

10.1.9 Turn on lamp.

10.1.10 Let warm up for 30 minutes.

10.1.11 Hit F1 that displays the main menu.

10.1.12 Type "P" to call up protocol, select the run protocol that defines the operating parameters of the system.

10.1.13 Once this is done, the data output screen is automatically chosen. The analyst must create a folder where the data will be stored.

10.1.14 Hit F1 main menu.

10.1.15 Type "A" to call up autosampler.

10.1.16 Hit R to create a rack, sample rack editing will allow the analyst to type in sample names, dilution factors, weights, etc.

10.1.17 Hit F1 main menu.

10.1.18 Type "S" to call up set up. In set up the software will know which rack to run and how many of the cups to run. To apply a given sample rack file to a rack position enter either 1 or 2 for the rack number. Then enter the rack name used when creating a rack. Then enter the beginning cup number and ending cup number.

10.1.19 Cup macros – The cup macros are a method of applying various functions to each sample cup after it is run (e.g., percent recovery on a spiked sample during reporting). Note: the cup macro options are displayed on the rack entry page.

10.1.20 Prepare stannous chloride solution ($SnCl_2$) according to the preparation instructions. Add to $SnCl_2$ reservoir and tighten cap.

10.2 Initial Calibration

10.2.1 The preparation of calibration standards is detailed in the appropriate preparation SOP and are summarized below.

10.2.2 Autosampler rack entry for calibration curve. To calibrate the PS200II a series of standards are analyzed and a curve is generated (see Table 10-1). The calibration curve may be accepted by the analyst if it is greater or equal than 0.995. Setting the concentration of the standards is required before running standards for a calibration curve. Hit F1 main menu followed by CS (calibration : standards). Enter the units for the calibration of the standards by hitting "U" and enter text representing the units to be used to report sample concentration. Hit a number from 1 to 6, this allows entry of concentrations for standards 1 through 6.

10.2.3 To load the standards, fill the large sample cups supplied and place them in the 14-cup rack. This rack is reserved for calibration standards and check standards.

Table 10-1

CALIBRATION STANDARDS

Concentration (µg/L)	Equivalent Soil Concentration for 0.2 g Sample (CLP)	Equivalent Soil Concentration for 0.6 g Sample (7471A)
0	0	0
0.2	0.1	0.033
0.5	0.25	0.083
1.0	0.5	0.167
5.0	2.5	0.833
10.0	5.0	1.67

Revision: 0	Status: Final	Method: SW-846, CLP ILM04.1	Minor Revision Date: None

10.2.4 It is important that the calculated value for the 0 standard be less than the MDL in order to obtain ND values for blanks. This is critical in some projects (see Section 13). In other cases as long as the low standard is <0.2 µg/L, the results are acceptable.

10.3 Daily and Continuing Calibration

10.3.1 To ensure that the sample results are valid, it is important to run check standards at frequency specified in this SOP. Setting the concentration and limits of acceptance of the check standards is required before running samples. Hit F1 main menu followed by "C" then "K" (Calibration : Check Standards). Hit a number from 1 to 7, this allows entry of concentrations for check standards 1 through 7. The first check standard is reserved for a blank check standard (i.e., CCB). When entering concentration for check standard, set the +/- limits of acceptance around zero. The other check standards are entered by target concentration and then the +/- limits of acceptance in percentage (see SOP.12 and SOP.57). The values entered on this page are stored in the protocol file. The results of the check standard are printed with a percent recovery calculation. The printout also indicates if the check standard fails to meet its acceptance. A printout of an H or L represents whether the results exceed the limits either high or low. To load the check standards, fill the large sample cups supplied and place them in the 14-cup rack.

CLP Analytical Run Sequence
Blank
Calibration Standards
ICV
ICB
CCV
CCB
CRA
PBW or PBS
LCSW or LCSS – Up to 7 analyses
CCV
CCB
Up to 10 analyses
CCV
CCB

Other Analytical Run Sequences
Blank
Calibration Standards
ICV
ICB
CCV
CCB
PBW or PBS or PB filter or PB gauze
LCSW or LCSS or LCS filter or LCS gauze
Up to 8 analyses
CCV
CCB
Up to 10 analyses
CCV
CCB

10.4 Sample Analysis

10.4.1 To run samples, sample rack files have to be created. A rack file is simply the information of sample IDs and cup macros associated with a physical rack where the samples are loaded. To edit or create a sample rack, hit F1 main menu, "A" then "R" (Autosampler : Rack Entry). Enter the name of the sample rack to edit. Edit the rack by column entry or hit the insert key and enter by individual cells. The sample IDs should be entered with consideration to summary reporting.

10.4.2 To indicate which sample racks for the software to run, enter the Autosampler Setup page. Hit F1 Main Menu A S (Autosampler: Setup) and then enter either 1 or 2 to indicate which rack to name. Enter a rack file name and the first and last sample cup to run.

10.4.3 To load the samples, fill disposable sample cups supplied and place them in the 44-cup rack. Make sure sufficient stannous chloride solution is available in the reservoir (see Section 10.1.17).

10.4.4 The run is started by hitting the F8 sample key while on the Autosampler: Setup Page OR by hitting the F8 sample key twice from any page will initiate running the sample racks.

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10.5 Instrument Conditions and Maintenance

10.5.1 The software offers a simple to use online scheduled maintenance page. To view page, Go To Instrument: Scheduled Maintenance (F1 Menu, I, S). A page displaying all items necessary to keep the instrument well maintained is shown. The schedule must be followed and documented in the Instrument Maintenance Log.

10.5.2 Each scheduled maintenance item has a usage counter, timed usage, or both (N/A indicates that the usage counter or the timed usage is not applicable for that item). If either condition expires for a given item, a maintenance message will alert the user at the top of the status box.

10.5.3 An asterisk will appear next to the item requiring maintenance on the scheduled maintenance screen. To clear the message hit [TAB] or replace, clean, or replenish the item using the hot key for the item on the scheduled maintenance page. To perform the maintenance on a given item simply type the hot key (e.g., type [P] for pump tubing} and follow the directions. Once the directions are followed to completion, the usage counter and timed usage gets updated. The instrument, computer, and printer must be kept clean at all times.

Instrument Conditions and Maintenance

See Table 10-1, Routine Maintenance Procedures.

Table 10-2

ROUTINE MAINTENANCE PROCEDURES

Equipment Instrument	Symptom	Operation	Frequency
Leeman PS200 II	Poor readings	Clean Hg optical cell	As needed or indicated on maintenance screen
Leeman PS200 II	No flow or dirty tubing	Change reductant bottle tubing from bottle to pump tubing	As needed or indicated on maintenance screen
Leeman PS200 II	No reading (no peaks)	Change Hg lamp or adjustment	As needed or indicated on maintenance screen
Leeman PS200 II	High baseline for blanks	Change pump tubing	As needed or indicated on maintenance screen
Leeman PS200 II	No vapor flow	Check nitrogen pressure	As needed or indicated on maintenance screen
Revision: 0	Status: Final	Method: SW-846, CLP ILM04.1	Minor Revision Date: None

11.0 DATA REDUCTION/EVALUATION/REPORTING

Table 11-1

TARGET ANALYTES AND QUANTITATION LIMITS

Compound/ Analyte	Type	PQL			
		Water (Units)	Soil (Units)	Air Tube	Wipe
Hg	T,S,L	0.2 µg/L	0.1 mg/kg (0.2 g sample) 0.033 mg/kg (0.6 g sample)	NA	NA
Hg	T,L	NA	NA	0.05 µg/tube (0.0005 mg/m^3 based on 0.1 m^3 [100 L] air volume)	0.02 µg/wipe
Revision: 0	Status: Final	Method: SW-846, CLP ILM04.1		Minor Revision Date: None	

Key Type:

T = Compound/analyte is target compound routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike.
L = Compound/analyte is routinely used as a LCS spike.
Q = Compound/analyte is used as a surrogate spike.

11.1 Water Samples. Instrument prints results in µg/L.

11.2 Soil Samples. Calculate from the curve (µg/L) to obtain results in mg/kg.

$$\frac{\mu g/L \cdot 0.1L}{g\,(\text{grams of sample weighed})} \cdot df = \mu g/g = mg/kg$$

Example: When 0.3 g soil is used:

$$\frac{1.2\ \mu g/L\,(\text{curve}) \cdot 0.1L}{0.3g} = 0.4\,mg/kg$$

where:

df = Any dilution factor.

11.3 Filter Samples (Metals.66)

If air volume supplied is in m^3, use

$$\frac{mg}{m^3} = \mu g/L\,(curve) \cdot 0.100\,L\,(BOD\,Volume) \cdot Tube \cdot \frac{Tube}{Air\,Volume(m^3)} \cdot df \cdot \frac{1\,mg}{1000\,\mu g}$$

If air volume is supplied in liters, use

$$\frac{mg}{m^3} = \mu g/L\,(curve) \cdot 0.100\,L\,(BOD\,Volume) \cdot Tube \cdot \frac{Tube}{Air\,Volume(L)} \cdot \frac{1000\,L}{m^3} \cdot \frac{1\,mg}{1000\,\mu g} \cdot df$$

If no air volume is supplied, use

$$\frac{mg}{Tube} = \frac{\mu g\,curve}{L} \cdot \frac{0.100\,L\,(BOD\ Volume)}{Tube} \cdot df$$

where:

df = Any dilution factors.

11.4 Wipe Samples (Metals.69)

$$\mu g/wipe = \mu g/L\,(curve) \cdot \frac{0.1\,L}{wipe} \cdot df$$

where:

df = Any dilution factor.

11.5 Matrix Spike Analysis

11.5.1 Calculate the recovery of mercury in the matrix spike and report on the appropriate forms. Calculate the concentrations of the mercury using the above equations used to calculate sample. Calculate the recovery of each mercury as follows:

$$\text{Matrix spike recovery} = \frac{SSR - SR}{SA} \cdot 100$$

where:

SSR = Spike sample result.
SR = Sample result.
SA = Spike added.

11.6 Matrix Spike Duplicate or Duplicate Sample Analysis

11.6.1 Calculate the relative percent difference (RPD) between the sample result(s) and duplicate (or MS and MSD) as follows:

$$RPD = \frac{|S - D|}{1/2(S + D)} \cdot 100$$

where:

S = Sample or MS result.
D = Duplicate or MSD result.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Routine Quality Control Samples

Table 12-1.1

ROUTINE QUALITY CONTROL SAMPLES (7470A, 7471A)

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Initial Calibration	Daily	See preparation SOP	Correlation coefficient ≥0.995	Recalibrate
ICV	Immediately following calibration	See Metals.70 or Metals.71 (Concentration 1.0 µg/L)	90-110% recovery	Recalibrate or redigest batch.
ICB	After ICV	See Metals.70 or Metals.71	< | PQL |	Recalibrate
CCV	Every 10 Analyses	See Metals.70 or Metals.71 (Concentration 5.0 µg/L)	80-120% recovery	Recalibrate and reanalyze last 10 samples.
CCB	After CCV	See Metals.70, or Metals.71 (Concentration 1.0 µg/L)	< | PQL |	Recalibrate. Reanalyze last 10 samples.
PBW **(7470A)**	One per batch of 20 or fewer samples	See Metals.70	< | PQL |	Reanalyze once; if fails, redigest batch.
LCSW **(7470A)**	One per batch of 20 or fewer samples if client specified	See Metals.70 (Concentration 1.0 µg/L)	80-120% recovery	Reanalyze once; if fails, redigest batch.

Table 12-1.1

ROUTINE QUALITY CONTROL SAMPLES (7470A, 7471A)

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Matrix Spike (MS) **(7470A)**	One per 20 or fewer samples	See Metals.70 (Concentration added 1.0 µg/L)	- Project requirements if specified. - Compare against LCS limits for bias.	Project requirements if any, otherwise evaluate results in conjunction with other QC information (e.g., LCS recoveries) to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Matrix Spike Duplicate (MSD) **(7470A)**	With every MS	See Metals.70 (Concentration 1.0 µg/L)	- Project requirements if specified. - Compare against LCS limits for bias.	Project requirements if any, otherwise evaluate results in conjunction with other QC information (e.g., LCS recoveries) to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Duplicate (DUP)	If client specified	Duplicate sample. Prepare exactly as original sample.	Project requirements if specified, otherwise: - 20% for waters - 35% for soils If one or both sample results are <5 times PQL, then duplicate results are acceptable if the difference between the two results is < 2 times PQL.	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
PBS **(7471A)**	One per batch of 20 or fewer samples	See Metals.71	< | PQL |	Reanalyze once; if fails, redigest batch.
LCSS **(7471A)**	One per batch of 20 or fewer samples	See Metals.71	Laboratory limits	Redigest batch

Table 12-1.1

ROUTINE QUALITY CONTROL SAMPLES (7470A, 7471A)

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Matrix Spike (MS) **(7471A)**	One per 20 or fewer samples	See Metals.71 (Concentration 0.167 mg/kg for 0.3 g sample)	- Project requirements if specified. - Compare against LCS limits for bias.	Project requirements if any, otherwise evaluate results in conjunction with other QC information (e.g., LCS recoveries) to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Matrix Spike Duplicate (MSD) **(7471A)**	With every MS	See Metals.71 (Concentration 0.167 mg/kg for 0.3 g sample)	- Project requirements if specified. - Compare against LCS limits for bias.	Project requirements if any, otherwise evaluate results in conjunction with other QC information (e.g., LCS recoveries) to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Duplicate (DUP)	If client specified	Duplicate sample. Prepare exactly as original sample.	Project requirements if specified, otherwise: - 20% for waters - 35% for soils	Provide comment. Flag data.
Revision: 0	Status: Final	Method: 7470A, 7471A		Minor Revision Date: None

Table 12-1.2

ROUTINE QUALITY CONTROL SAMPLES (CLP)

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Initial Calibration	Daily	See preparation SOP	Correlation coefficient $\geq$0.995	Recalibrate
ICV	Immediately following calibration	See Metals.21 or Metals.60 (Concentration 1.0 µg/L)	80-120% recovery	Recalibrate or redigest batch

Table 12-1.2

ROUTINE QUALITY CONTROL SAMPLES (CLP)

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
ICB	After ICV	See Metals.21 or Metals.60	< | PQL |	Recalibrate
CCV	Every 10 Analyses	See Metals.21 or Metals.60 (Concentration 5.0 µg/L)	80-120% recovery	Recalibrate and reanalyze last 10 samples.
CCB	After CCV	See Metals.21 or Metals.60 (Concentration 1.0 µg/L)	< | PQL |	Recalibrate. Reanalyze last 10 samples.
PBW	One per batch of 20 or fewer samples	See Metals.21	< | PQL |	Reanalyze once; if fails, redigest batch.
Matrix Spike (MS) (CLP water)	One per 20 or fewer samples	See Metals.21 (Concentration 1.0 µg/L)	75-125% recovery	Flag "N"
Duplicate (DUP) (CLP water)	One per 20 or fewer samples	Duplicate sample. Prepare exactly as original sample.	±20%	Flag "*"
PBS (CLP soils)	One per batch of 20 or fewer samples	See Metals.60	< | PQL |	Reanalyze once; if fails, redigest batch.
LCSS (CLP soils)	One per batch of 20 or fewer samples	See Metals.60 (Concentration varies with lot)	Within manufacturer's limits	Redigest batch.
Matrix Spike (MS) (CLP soils)	1 per 20 or fewer samples	See Metals.60 (Concentration 0.5 mg/kg for 0.2 g sample)	75-125% recovery	Flag "N"
Duplicate (DUP) (CLP soils)	1 per 20 or fewer samples	Duplicate sample. Prepare exactly as original sample.	20% recovery	Flag "*"
Revision: 0	Status: Final	Method: CLP ILM04.1	Minor Revision Date: None	

Table 12-1.3

ROUTINE QUALITY CONTROL SAMPLES (245.1)

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Initial Calibration	Daily	See preparation SOP.	Correlation coefficient ≥0.995	Recalibrate
IPC (analogous to CCV)	Immediately following calibration and every 10 analyses to end	See Metals.70. Same material and concentration as CCV in other methods. (Concentration 5.0 µg/L)	95-105% first IPC, 90-110% thereafter	Recalibrate and reanalyze last 10 samples.
ICB	After first IPC	See Metals.70.	< | PQL |	Recalibrate, reanalyze last 10 samples.
LFB (analogous to ICV, LCSW)	One per 20 or fewer samples	See Metals.70. Same material and concentration as ICV and LCSW in other methods. (Concentration 1.0 µg/L)	85-115% recovery	Identify cause, resolve. Rerun the batch if applicable.
LRB (analogous to ICV, PBW)	One per batch of 20 or fewer samples	See Metals.70 (same as PBW in other methods).	< PQL or 10% of sample concentration	Reanalyze once; if fails, redigest batch.
QCS	Minimally quarterly	See Metals.70. Mid-range second source. (Concentration 5.0 µg/L)	90-110%	Identify cause, resolve.
CCB	After IPC	See Metals.70.	< | PQL |	Recalibrate. Reanalyze last 10 samples.
LFM	**Two** per 20 or fewer samples	See Metals.70. Same as MS/MSD in other water methods. (Concentration 1.0 µg/L)	70-130% recovery	Check LFB and IPC.
Revision: 0	Status: Final	Method: 245.1	Minor Revision Date: None	

Table 12-1.4

ROUTINE QUALITY CONTROL SAMPLES (SORBENT TUBES) (NIOSH 6009)

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Initial Calibration	Daily	See preparation SOP	Correlation coefficient $\geq$0.995	Recalibrate
ICV	Immediately following calibration	See Metals.66 (Concentration 1.0 µg/L)	80-120% recovery	Recalibrate or redigest batch.
ICB	After ICV	See Metals.66	< | PQL |	Recalibrate
CCV	Every 10 Analyses	See Metals.66 (Concentration 5.0 µg/L)	80-120% recovery	Recalibrate and reanalyze last 10 samples.
CCB	After CCV	See Metals.66 (Concentration 1.0 µg/L)	< | PQL |	Recalibrate. Reanalyze last 10 samples.
PB media	One per batch of 20 or fewer samples	See Metals.66	< | PQL |	Reanalyze once; determine source. If source is lab, redigest the batch if additional sample tubes are available.
LCS	One per batch of 20 or fewer samples	See Metals.66 (Concentration 1.0 µg/L)	80-120% recovery	Reanalyze once; if fails, redigest batch.
Revision: 0	Status: Final	Method: NIOSH 6009		Minor Revision Date: None

Table 12-1.5

ROUTINE QUALITY CONTROL SAMPLES (WIPES)

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Initial Calibration	Daily	See preparation SOP	Correlation coefficient $\geq$0.995	Recalibrate
ICV	Immediately following calibration	See Metals.69 (Concentration 1.0 µg/L)	90-110% recovery	Recalibrate or redigest batch.
ICB	After ICV	See Metals.69	< | PQL |	Recalibrate
CCV	Every 10 Analyses	See Metals.69 (Concentration 5.0 µg/L)	80-120% recovery	Recalibrate and reanalyze last 10 samples.

Table 12-1.5

ROUTINE QUALITY CONTROL SAMPLES (WIPES)

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
PB wipe	One per batch of 20 or fewer samples	See Metals.69	< | PQL |	Reanalyze once; determine source. If source is lab, redigest the batch if additional sample wipes are available.
Revision: 0	Status: Final	Method: 7470A Wipes	Minor Revision Date: None	

Table 12-2

CONTROL CHARTED QUALITY CONTROL SAMPLES

QC Type	Analytes Charted	Matrix Charted
LCSW	Hg	Water
LCSS	Hg	Soil
Revision: 0 Status: Final	Method: SW-846, CLP ILM04.1	Minor Revision Date: None

Table 12-3.1

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES (7470A) METALS.70

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
~~(LCS)~~ ICV	Water	Hg	1.0 µg/L	90-110	NA	Method
CCV	Water	Hg	5.0 µg/L	80-120	NA	Method
MS	Water	Hg	1.0 µg/L	See Table 12-1.1	NA	Laboratory
MSD	Water	Hg	1.0 µg/L	See Table 12-1.1	20	Laboratory
LCS	Water	Hg	1.0 µg/L	80-120	NA	Laboratory
Revision: 0	Status: Final	Method: 7470A		Minor Revision Date: None		

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Table 12-3.2

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES (7471A) METALS.71

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
ICV	Water	Hg	1.0 µg/L	90-110	NA	Method
CCV	Water	Hg	5.0 µg/L	80-120	NA	Method
LCSS	Soil	Hg	1.0 µg/L (0.167 mg/kg)	Laboratory limits	NA	Laboratory
MS	Soil	Hg	0.167 mg/kg (0.3 g sample)	See Table 12-1.1	NA	Laboratory
MSD	Soil	Hg	0.167 mg/kg (0.3 g sample)	See Table 12-1.1	35	Laboratory
Revision: 0	Status: Final	Method: 7471A		Minor Revision Date: None		

Table 12-3.3

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES (CLP WATER) METALS.21

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
ICV (LCS)	Water	Hg	1.0 µg/L	80-120	NA	Method
CCV	Water	Hg	5.0	80-120	NA	Method
MS	Water	Hg	1.0	75-125	NA	Method
DUP	Water	Hg	NA	NA	20	Method
Revision: 0	Status: Final	Method: CLP ILM04.1		Minor Revision Date: None		

Table 12-3.4

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES (CLP SOIL) METALS.60

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
ICV (LCS)	Water	Hg	1.0 µg/L	80-120	NA	Method
CCV	Water	Hg	5.0 µg/L	80-120	NA	Method
LCS	Soil	Hg	Manufacturer	Manufacturer	NA	Method
MS	Soil	Hg	0.5 mg/kg	75-125	NA	Method
DUP	Soil	Hg	NA	NA	20	Method
Revision: 0	Status: Final	Method: CLP ILM04.1		Minor Revision Date: None		

Table 12-3.5

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES (245.1) METALS.70

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
LFB (ICV, LCS)	Water	Hg	1.0 µg/L	85-115	NA	Method
IPC (CCV)	Water	Hg	5.0 µg/L	95-105 first 90-110 subsequent	NA	Method
LFM (MS)	Water	Hg	1.0 µg/L	70-130	NA	Method
DUP	Water	Hg	NA	NA	NA	Method
QCS	Water	Hg	5.0 µg/L	90-110	20	Method
Revision: 0	Status: Final	Method: 245.1			Minor Revision Date: None	

Table 12-3.6

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES (NIOSH 6009) METALS.66

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
ICV	Water	Hg	1.0 µg/L	80-120	NA	Laboratory
CCV	Water	Hg	5.0 µg/L	80-120	NA	Laboratory
LCS	(Sorbent if available)	Hg	1.0 µg/L	80-120	NA	Laboratory
Revision: 0	Status: Final	Method: NIOSH 6009			Minor Revision Date: None	

Table 12-3.7

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES (WIPES) METALS.69

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
ICV	Water	Hg	1.0 µg/L	80-120	NA	Laboratory
CCV	Water	Hg	5.0 µg/L	80-120	NA	Laboratory
LCS	Wipe	Hg	0.5 µg/L	80-120	NA	Laboratory
Revision: 0	Status: Final	Method: 7470A Wipes			Minor Revision Date: None	

13.0 SPECIAL PROJECT REQUIREMENTS

13.1 URS – Plattsburg

13.1.1 Requires matrix duplicate (MD) and matrix spike (MS).

13.2 Griffis

13.2.1 Make certain calculated value for 0 standard is << | MDL |.

14.0 SAMPLE DISPOSAL

14.1 See Waste Disposal SOP A.10.

14.2 Samples or standards exceeding 200 µg/L must not be poured down the drain.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: ICP Metals Analysis by Method 6010B			
Reviewer: G. Matthies		Peer Reviewer: S. Turk	
Implementation Date: 11/3/00		Last Annual Review Date: 11/2/00	
Revision: 10	Status: Final	Method: 6010B	Minor Revision Date: 4/23/01

1.0 SCOPE AND APPLICATION

1.1 This SOP outlines the procedure for analyzing metals by inductively coupled argon plasma (ICP) in water and solids. The method may also be used to calculate hardness of a water sample. The sample is first digested as given in SOP Metals.10 or Metals.11.

2.0 METHOD SUMMARY

2.1 This is the SOP for ICP analysis of water or soils digestate. Prefiltered and acidified aqueous samples need not be digested, but must be matrix matched with the calibration standards.

2.2 Deviation from method. The method Section 8.6.1.3 directs the results of the calibration blank are to agree within three times IDL and to repeat twice, average, with average to be within three standard deviations of background mean. Method detection limits are used in 6010B analyses. Laboratory criteria is such that blank result must be within absolute value of PQL.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Method 6010B (Rev. 2, December 1996) SW-846, Third Edition, Update III, June 1997.
4.2 Method 6010C (Draft Revision 3, January 2000), SW-846, third edition, pre-release.
4.3 Checklist C-010.
4.4 Training SOP QA.5, A.23, and A.29.
4.5 Data Review SOP A.25.
4.6 Laboratory Quality Assurance Manual.
4.7 MDL SOP A.18.
4.8 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS

5.1 **MDL** – Method Detection Limit as determined by 40 CFR part 136, Appendix B. The MDL must be determined yearly for both water and soil matrices. See SOP A.18 for procedure.

5.2 **PQL** – Practical Quantitation Limit is the concentration above the MDL that can be reasonably obtained and is used as the limit of reporting.

5.3 **ICV** – Initial Calibration Verification. A solution independent of the calibration standards within the calibration range of the instrument.

5.4 Analytical Sample – Any determination except calibration verification and calibration blank. An analytical sample includes preparation blanks, LCS, matrix spike, and matrix duplicate.

5.5 IEC Factor – Interelement Correction Factor, determined yearly, corrects for emission line interferences from an interferent element on interfered elements. See SOP Metals.59 for procedure.

5.6 CAL BLK (CCB) – Continuing Calibration Blank.

5.7 CCV – Continuing Calibration Verification. This is a check sample approximately midpoint from the calibration standards.

5.8 ICSAB – Interference Check Solution (Used to verify interelement correction).

5.9 PDS – Post Digestion Spike. Also known as Analytical Post Spike (A). A known amount of spike solution added to a processed sample at the instrument to judge interferences.

5.10 Linear Range – The upper limit of the ICP range. Results above this range require dilution. Determined quarterly by analyzing a solution of known concentration. Results of the analysis are to agree to within +5% of the true value.

5.11 L – Serial dilution.

5.12 IDL – Instrument Detection Limit.

5.13 LCS – Laboratory Control Sample.

5.14 MS – Predigestion spike prepared with batch.

5.15 MSD – MS duplicate.

5.16 DUP – Duplicate sample prepared with batch.

5.17 CCB – Continuing Calibration Blank.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Interference check standard is used to verify the absence of any spectral interference. The elements that commonly give spectral interferences are listed on the instrument's IEC tables and include aluminum, iron, calcium, and magnesium. If the concentration of these interfering elements are present above the linear range than the instrument correction will no longer be valid. The sample should be diluted and re-analyzed. Any elements indicated in the IEC tables, as having either a positive or negative interference with the interfering element also should be reported from the re-analysis at a dilution.

6.2 Sample carry over.

6.3 Keep a copy of the current plasma conditions and background correction points in the service /maintenance log.

7.0 INSTRUMENTATION AND EQUIPMENT

Perkin-Elmer ICP (Optima 3000XL and 3300DV)
Class "A" pipettes used for standard preparation
NIST traceable standards
Class "A" volumetric flasks

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard and Reagents.

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Nitric acid, metals grade	Fisher or equivalent	Concentrated
Hydrochloric acid, metals grade	Fisher or equivalent	Concentrated
ICSA	VHG or equivalent	2,000 µg/mL - Fe 5,000 µg/mL - Al, Ca, Mg
ICSAB-1	VHG or equivalent	50 µg/mL - Be, Co, Cu, Mn 100 µg/mL - Ag, Cd, Ni, Pb, Zn 50 µg/mL - Ba, Cr, V
Argon gas 99.9999%	Rogers Welding	N/A
Nitrogen gas 99.9999%	Rogers Welding	N/A
CMIX#1A	CPI or equivalent	50 mg/L - Be, Cd, Pb, Sr 100 mg/L - As, B, Ba, Co, Cr, Cu, Mn, Mo, Ni, Se, Sn, Ti, Tl, V, Zn, Zr
CMIX#2A	CPI or equivalent	500 mg/L - B, Be, Cd, Sr 1000 mg/L - As, Ba, Cr, Cu, Ni, Pb
CMIX#1B	CPI or equivalent	1000 mg/L - Al, Fe 5000 mg/L - Co, K, Mg, Na
CMIX#2B	CPI or equivalent	1000 mg/L - Co, Mn, Mo, Se, Sn, Ti, Tl, V, Zn, Zr
1000 µg/mL B Calibration Stock	CPI or equivalent	1000 µg/mL
1000 µg/mL Mo Calibration Stock	CPI or equivalent	1000 µg/mL
1000 µg/mL Sn Calibration Stock	CPI or equivalent	1000 µg/mL
1000 µg/mL Sr Calibration Stock	CPI or equivalent	1000 µg/mL
1000 µg/mL Ti Calibration Stock	CPI or equivalent	1000 µg/mL
1000 µg/mL Zr Calibration Stock	CPI or equivalent	1000 µg/mL
1000 µg/mL Ag Calibration Stock	CPI or equivalent	1000 µg/mL
1000 µg/mL As Calibration Stock	CPI or equivalent	1000 µg/mL
1000 µg/mL Pb Calibration Stock	CPI or equivalent	1000 µg/mL
1000 µg/mL Se Calibration Stock	CPI or equivalent	1000 µg/mL
1000 µg/mL Tl Calibration Stock	CPI or equivalent	1000 µg/mL
1000 µg/mL Sb Calibration Stock	CPI or equivalent	1000 µg/mL
1000 µg/mL Ag Reference Stock	High Purity or equivalent	1000 µg/mL
1000 µg/mL B Reference Stock	High Purity or equivalent	1000 µg/mL
1000 µg/mL Cd	High Purity or equivalent	1000 µg/mL
1000 µg/mL Mo	High Purity or equivalent	1000 µg/mL
1000 µg/mL Pb	High Purity or equivalent	1000 µg/mL
1000 µg/mL Sn	High Purity or equivalent	1000 µg/mL

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
1000 µg/mL Sr	High Purity or equivalent	1000 µg/mL
1000 µg/mL Ti	High Purity or equivalent	1000 µg/mL
1000 µg/mL Zr	High Purity or equivalent	1000 µg/mL
10.0 mg/mL Al Calibration Stock	CPI	10,000 µg/mL
10.0 mg/mL Ca Calibration Stock	CPI	10,000 µg/mL
10.0 mg/mL Fe Calibration Stock	CPI	10,000 µg/mL
10.0 mg/mL K Calibration Stock	CPI	10,000 µg/mL
10.0 mg/mL Mg Calibration Stock	CPI	10,000 µg/mL
10.0 mg/mL Na Calibration Stock	CPI	10,000 µg/mL
ICV-1 Stock	High Purity or equivalent	100 µg/mL - Al, As, Ba, Cr, Co, Cu, Fe, Pb, Mn, Ni, Se, Tl, V, Zn 1000 µg/mL - Ca, K, Mg, Na 40 µg/mL - Be 50 µg/mL - Cd
ICV-2 Stock	High Purity or equivalent	200 µg/mL - Sb
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Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Added to ____	Final Concentration
Standard Dilution Water	Metals Grade Acids, HCl and HNO_3	Pipet 60 mL of concentrated HNO_3 and 50 mL of concentrated HCl to a 1-L volumetric flask. Dilute to mark with ASTM Type II water and mix by inversion. Prepare weekly.	6% HNO_3, 5% HCl
EEMIX#1	Individual Stocks 1000 µg/mL Ag 1000 µg/mL Sb	Pipet 5 mL each 1000 µg/mL stock for Ag and Sb into a 50-mL volumetric flask containing 3 mL HNO_3 and 5 mL HCl. Dilute to volume with ASTM Type II water. Prepare monthly.	100 mg/L - Ag, Sb
EEMIX#2	Individual Stock 1000 µg/mL B	Pipet 1 mL 1000 µg/mL B stock into a 100-mL volumetric flask containing 6 mL HNO_3 and 5 mL HCl. Dilute to volume with ASTM Type II water. Prepare monthly.	10 mg/L - B
EEMIX#3	Individual Stock 1000 µg/mL Ag	Pipet 5 mL each 1000 µg/mL Ag stock into a 50-mL volumetric flask containing 3 mL HNO_3 and 5 mL HCl. Dilute to volume with ASTM Type II water. Prepare monthly.	100 mg/L - Ag

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Added to ____	Final Concentration
ICSAB-3	Individual Stocks 1000 µg/mL As 1000 µg/mL Se 1000 µg/mL Sb 1000 µg/mL Tl 1000 µg/mL B 1000 µg/mL Mo 1000 µg/mL Sn 1000 µg/mL Ti 1000 µg/mL Sr 1000 µg/mL Zr	Pipet 10 mL of each 1000 µg/mL stock for As, B, Mo, Sb, Se, Sn, Ti, Tl, and Zr and 5 mL Sr 1,000 µg/L stock into a 100-mL volumetric flask containing 6 mL HNO_3 and 5 mL HCl. Dilute to volume with ASTM Type II water. Prepare every 6 months.	50 µg/mL - Sr; 100 µg/mL - As, B, Mo, Sb, Se, Sn, Ti, Tl, and Zr
ICSAB	ICSA ICSAB-1 ICSAB-3 10.0 Na µg/mL 10.0 K µg/mL	~~Pipet 4 mL ICSA, 1 mL ICSAB-1, and 1 mL ICSAB-3 into a 100-mL volumetric flask containing 6 mL HNO_3 and 5 mL HCl. Dilute to volume with ASTM Type II water. Prepare weekly.~~ Pipet 8 mL ICSA, 2 mL ICSAB-1, and 2 mL ICSAB-3, 5 mL of 10 µg/mL sodium and 5 mL of 10 µg/mL potassium into a 200-mL volumetric flask containing 12 mL HNO_3 and 10 mL HCl. Dilute to volume with ASTM Type II water. Prepare weekly.	500 µg/L - Ba, Be, Co, Cr, Cu, Mn, Sr, V; 1,000 µg/L - Ag, As, B, Cd, Mo, Ni, Pb, Sb, Se, Sn, Ti, Tl, Zn, Zr; 250,000 µg/L - Na, K 80,000 µg/L - Fe; 200,000 µg/L - Al, Ca, Mg
S0 (Calibration Blank)	N/A	Add 12 mL concentrated HNO_3 and 10 mL concentrated HCl to Type II water in a 200-mL volumetric flask. Dilute to the mark with Type II water. Prepare daily.	6% HNO_3, 5% HCl
S7 (S1-S6 are standards used in another method; S7 is the low standard in this procedure.)	S8 (see below) EEMIX#2	Pipet 1 mL S8 and 1 mL EEMIX#2 into a 100-mL volumetric flask containing 6 mL HNO_3 and 5 mL HCl. Dilute to volume with ASTM Type II water. Prepare daily.	5 µg/L - Be, Cd, Pb, Sr; 10.0 µg/L - Ag, As, Ba, Co, Cr, Cu, Mn, Mo, Ni, Sb, Se, Sn, Ti, Tl, V, Zn, Zr; 100 µg/L - Al, Fe 110 µg/L - B 500 µg/L - Ca, K, Mg, Na

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	Amount of Stock Added to ____	Final Concentration
S8/CCV	CMIX#1 EEMIX#1	Pipet 2 mL CMIX#1A, CMIX#1B, and 2 mL EEMIX#1 into a 200-mL volumetric flask containing 12 mL of HNO_3 and 10 mL HCl. Dilute to volume with ASTM Type II water. Prepare daily.	500 µg/L - Be, Cd, Pb, Sr; 1000 µg/L - Ag, As, B, Ba, Co, Cr, Cu, Mn, Mo, Ni, Sb, Se, Sn, Ti, Tl, V, Zn, Zr; 10,000 µg/L - Al, Fe; 50,000 µg/L - Ca, K, Mg, Na
S9 (3300DV)	CMIX#2 EEMIX#3 1000 µg/mL Sb Calibration Stock	Pipet 1 mL CMIX#2, CMIX#2B, 1 mL of 1000 µg/mL Sb Calibration stock, and 4 mL EEMIX#3 into a 100-mL volumetric flask containing 6 mL of HNO_3 and 5 mL HCl. Dilute to volume with ASTM Type II water. Prepare daily.	4000 µg/L - Ag; 5000 µg/L - B, Be, Sr, Cd; 10,000 µg/L - As, Ba, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn, Zr
S10	10.0 mg/mL individual stocks of Al, Ca, Fe, K, Mg, Na	Pipet 1 mL each individual stock for Al, Ca, Fe, K, Mg, Na into a 50-mL volumetric flask containing 3 mL HNO_3 and 2.5 mL HCl. Dilute to volume with ASTM Type II water. Prepare daily.	200,000 µg/L - Al, Ca, Fe, K, Mg, Na,
S11 (3000XL)	10.0 mg/mL individual stock of Fe, K, Na	Pipet 0.5 mL each of K, Na and 2 mL Fe into a 200-mL volumetric flask containing 12 mL HNO_3 and 10 mL HCl. Dilute to volume with ASTM Type II water. Prepare daily.	25,000 µg/L - K, Na 100,000 µg/L - Fe
ICV 3 Stock	1000 µg/mL Ag Reference Stock	Pipette 10.0 mL of 1000 µg/L Ag Reference Stock into a 100-mL volumetric flask. Add 5.0 mL of concentrated HNO_3 and dilute to volume with ASTM Type II water. Prepare every 2 months.	100 µg/mL - Ag
ICV 4 Stock	1000 µg/mL B, Cd Mo, Pb, Sn, Sr, Ti, Zr Reference Stocks	To a 100-mL volumetric flask containing ASTM Type II water, 6 mL HNO_3 and 5 mL HCl acids, pipet 5 mL of Cd Reference Stock and 10 mL of B, Mo, Pb, Sn, Sr, Ti, Zr Reference Stocks. Dilute to volume. Prepare weekly.	50 µg/mL - Cd, Sr 100 µg/mL - B, Mo, Pb, Sn, Ti, Zr

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Added to ____	Final Concentration
ICV	ICV 1 stock ICV 2 stock ICV 3 stock ICV 4 stock	Into a 100-mL Class A volumetric pipette 1.0 mL of ICV-1 and 1.0 mL of ICV-2, 1 mL ICV-4 and 200 µL ICV-3 stock add 6 mL of nitric acid and 5 mL of hydrochloric acid. Fill to the mark with ASTM Type II water. Prepare weekly.	**All µg/L:** Ag 200 Mg 10000 Al 1000 Mn 1000 As 1000 Mo 1000 B 1000 Na 10000 Ba 1000 Ni 1000 Be 400 Pb 2000 Ca 10000 Sb 2000 Cd 1000 Se 1000 Co 1000 Sn 1000 Cr 1000 Sr 1000 Cu 1000 Ti 1000 Fe 1000 Zn 1000 K 10000 Zr 1000
CCB (Continuing Calibration Blank)	N/A	Add 12 mL concentrated HNO_3 and 10 mL concentrated HCl to Type II water in a 200-mL volumetric flask. Dilute to the mark with Type II water. Prepare daily.	6% HNO_3, 5% HCl

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9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Solid	Standard	6 months	6 months	8 oz. Glass
Water	Standard	6 months	6 months	Plastic, pH <2.0 with HNO_3

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10.0 PROCEDURE

10.1 Initial and Continuing Calibration

10.1.1 Turn the plasma on referring to appropriate instrument specifications. Allow the instrument to warm up for a minimum of 20 minutes.

10.1.2 Calibrate using the following standards each time the instrument is set up. Concentrations of standard solutions are listed.

10.1.3 Use a minimum of two exposures for calibration and analysis. Print individual exposures. Report the average. The coefficient of variance (CV) between exposures must be $\leq 5\%$ for positive results at or

above the reporting limit or at or above five times the MDL, whichever is greater. If criteria cannot be met, an additional exposure or filtering may be necessary.

10.1.4 Calibrate the instrument using a blank and 3 non-zero standards. Flush the instrument with calibration blank solution. Correlation coefficient must be 0.995 or greater.

10.1.5 S0 (calibration blank) - all elements, either instrument.

10.1.6 For the PE Optima 3000XL instrument, run the following calibration standards:

S7 (Low Level)
5 µg/L - Be, Cd, Pb, Sr
10 µg/L - Ag, As, Ba, Co, Cr, Cu, Mn, Mo, Ni, Sb, Se, Sn, Ti, Tl, V, Zn, Zr
100 µg/L - Al, Fe
110 µg/L - B
500 µg/L - Ca, K, Mg, Na

S8 (Medium Level)
500 µg/L - Be, Cd, Pb, Sr
1000 µg/L - Ag, As, B, Ba, Co, Cr, Cu, Mn, Mo, Ni, Sb, Se, Sn, Ti, Tl, V, Zn, Zr
10,000 µg/L - Al, Fe
50,000 µg/L - Ca, K, Mg, Na

S9 (High Level #1)
4000 µg/L - Ag
5000 µg/L - B, Be, Cd, Sr
10,000 µg/L - As, Ba, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn, Zr

S10
200,000 µg/L - Al, Ca, Mg

S11 (High Level #2)
25,000 µg/L - K, Na
100,000 µg/L - Fe

10.1.7 For the PE Optima 3300DV instrument, run the following calibration standards:

S7 (Low Level)
5 µg/L - Be, Cd, Pb, Sr
10 µg/L - Ag, As, Ba, Co, Cr, Cu, Mn, Mo, Ni, Sb, Se, Sn, Ti, Tl, V, Zn, Zr
100 µg/L - Al, Fe
110 µg/L - B
500 µg/L - Ca, K, Mg, Na

S8 (Medium Level)
500 µg/L - Be, Cd, Pb, Sr
1000 µg/L - Ag, As, B, Ba, Co, Cr, Cu, Mn, Mo, Ni, Sb, Se, Sn, Ti, Tl, V, Zn, Zr
10,000 µg/L - Al, Fe
50,000 µg/L - Ca, K, Mg, Na

S9 (High Level #1)
4000 µg/L - Ag
5000 µg/L – B, Be, Cd, Sr
10,000 µg/L - As, Ba, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn, Zr

S10 (High Level #2)
200,000 µg/L - Al, Ca, Fe, K, Mg, Na

10.1.8 Solutions needed for analytical run: Run calibration standards prior to sample analysis. Due to combinations of different elements required, multiple calibration standards are used. After calibration, analyze the ICV, CCB, CCV solutions. The next ten analytical samples may be analyzed. This is followed by a CCV/CCB pair for every 10 samples. The run is then closed off by a final CCV/CCB.

10.2 Run Sequence and Sample Analysis

10.2.1 Run the opening QC as follows. Refer to QC table for limits and necessary actions. Elements in addition to those below may be requested and added to the analysis sequence.
10.2.2 Flush system with the calibration blank between each sample as per the instrument manufacturer's suggestion.
10.2.3 ICV
10.2.4 ICB
10.2.5 CCV
10.2.6 CCB
10.2.7 ICSAB
10.2.8 Nine analytical samples.
10.2.9 Run a CCB and a CCV. Continue with analysis. Run a CCB and CCV every 10 analytical samples. Run one MS/MSD and LCS per preparation batch.
10.2.10 Perform a post analytical spike (PDS) on a sample that has an MS/MSD.
10.2.11 Analyze a 1/5 serial dilution, (1 part sample:4 parts dilution water), i.e., five-fold dilution, for each matrix per 20 samples. Perform on same sample as PDS. Results should be within +10% of the undiluted value.
10.2.12 Finish the analytical run with a CCB and CCV.
10.2.13 If QC is not within limits and cannot be corrected, note out of control QC on raw data and work order narrative.
10.2.14 **If any reading is above the high standard concentration, dilute within range.**
10.2.15 Print out a copy of the run prior to analysis as a run log.
10.2.16 Attach standards ID page and sample preparation log page to data.

Instrument Conditions and Maintenance

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Perkin Elmer ICP 3000XL Simultaneous ICP + 3300DV	N/A	Replace pump tubing, clean or replace nebulizer	Daily as needed

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11.0 DATA REDUCTION/EVALUATION/REPORTING

Table 11-1

TARGET COMPOUNDS AND QUANTITATION LIMITS

Element	Type	PQL		
		Water (µg/L)	Soil (mg/kg)	Casette or Filter (µg/Tube or Filter)
		Optima 3000XL and 3300DV	Optima 3000XL and 3300DV	Optima 3000XL and 3300DV
Al	TSL	100	10.0	10.0
Sb	TSL	10	2.0	1.0
As	TSL	20	2.0	1.0
Ba	TSL	10	2.0	2.0
Be	TSL	5	0.5	0.5
Cd	TSL	5	0.5	0.5
Ca	TSL	500	50	50
Cr	TSL	10	1.0	1.0
Co	TSL	10	2.0	2.0
Cu	TSL	10	1.0	3.0
Fe	TSL	100	100	100
Pb	TSL	10	1.0	0.5
Mg	TSL	500	50.0	50.0
Mn	TSL	10	1.0	1.0
Se	TSL	30	1.0	0.5
Ag	TSL	10	1.0	1.0
Na	TSL	1000	100	100
Tl	TSL	15	2.0	0.5
V	TSL	20	2.0	2.0
Zn	TSL	10	1.0	1.0
K	TSL	1000	100	100
Ni	TSL	10	2.0	2.0
B	M	200	10.0	10.0
Li	M	500	50.0	50.0
Mo	M	20	2.0	2.0
P	M	100	10.0	10.0
Si	M	500	50.0	50.0
Sr	M	5.0	0.5	0.5
Sn	M	10	3.0	1.0
Ti	M	10	2.0	1.0
Zr	C	10	1.0	1.0

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Note: Whenever requested by client, any nonroutine elements must be included in an LCS.

Key Type:

T = Element is target element routinely reported.
M = Element is listed in the method but is not routinely reported by E & E.
C = Element is specified by the client and can be analyzed under this method.
S = Element is routinely used as a matrix spike.
L = Element is routinely used as an LCS spike.

Table 11-1.1

TCLP ANALYTES AND QUANTITATION LIMIT

Element	PQL TCLP Extract (mg/L)	Regulatory Level (mg/L)
As	0.300	5.0
Ba	0.060	100.0
Cd	0.015	1.0
Cr	0.030	5.0
Pb	0.150	5.0
Se	0.300	1.0
Ag	0.030	5.0
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12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
ICV	After Calibration	See Table 8-2	± 10% recovery	Recalibrate
CCB (cal blk)	After ICV standards and after every 10 analysis at end of run	See Table 8-2	Less than absolute value of PQL	Repeat CCB. If still out, repeat samples from last acceptable blank that are less than 10 times PQL.
CCV	After CAL BLK – every 10 analysis at end of run	See Table 8-2	± 10% recovery	Recalibrate Reanalyze
ICSAB	After first CCB	See Table 8-2	± 20%	Recalibrate
LCS	1 per matrix per 20 samples	See appropriate digestion SOP	80-120%	Redigest batch and reanalyze for affected element(s)

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
MB	1 per matrix per 20 samples	See appropriate digestion SOP	< |PQL|	Recalibrate, repeat. If still out, repeat samples that are less than 10 times PQL.
MS	1 per matrix per 20 samples	See appropriate digestion SOP	Project requirements if specified, 75-125% otherwise	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
MSD	1 per matrix per 20 samples	See appropriate digestion SOP	Project requirements if specified, RPD 20% waters, 35% soils otherwise.	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Post-Spike	One per matrix per 20 samples	Spike sample analyte at concentration 10-100 x MDL	75-125% recovery	Note matrix interference. Perform serial dilution on this sample.
Serial dilution (L)	One per matrix per 20 samples	1:4 v:v dilution (1/5 dilution)	±10% if value 50 times MDL	Note interference from matrix. Inform project manager when both PDS and serial dilution fail. Further analytical tests, e.g., MSA may be required.
DUP	At client request	Duplicate sample at digestion	Project requirements if specified, RPD 20% waters, 35% soils, 20% tubes or filters	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.

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12.1 Post Spike Example: A value of 400 ppb is obtained for Pb for the original sample. The sample requires a post-analytical spike to produce a level between 10-100 x the MDL, e.g., at 800 µg/L. (If the sample contained nothing, the spike would be 10 times the MDL.) Pipet 800 µL of a 10 mg/L solution of Pb to 9.2 mL of sample. The resulting expected spiked sample result concentration is:

$$\frac{0.8\,\text{mL}\cdot 10{,}000\,\mu\text{g/L} + 9.2\,\text{mL}\cdot 400\,\mu\text{g/L}}{10\,\text{mL}} = 1168\,\mu\text{g/L}$$

General formula for determining expected concentration of sample with analytical spike:

$$\text{Expected SSR}\,(\mu\text{g/L}) = \frac{(V_a)SA + (V_s)SR}{V_t}$$

where:

SSR = Spiked sample result.
SA = Concentration of analytical spiking solution (µg/L).
SR = Original undiluted sample result (µg/L).
V_a = Volume of analytical spiking solution used (mL).
V_s = Volume of sample spiked with analytical spiking solution (mL).
V_t = Additive volume of sample plus volume of analytical spiking solution (mL).

% Recovery Calculation

$$\%\,\text{Recovery} = \frac{(\text{SSR} - [\text{Vs}\cdot\text{SR}])}{V_t} \;/\; \text{SA}$$

12.2 Calculation for Soils

12.2.1 The instrument software prints results. The following equation is provided as a check.

$$\text{C(mg/kg)} = \frac{\mu\text{g}}{\text{L}}(\text{reading})\cdot\frac{\text{V}}{\text{S}}\cdot\text{df}$$

where:

C = Concentration in the sample.
V = Final digestate volume (0.050 L for block digestion; 0.100 L for hotplate digestion).
S = Sample mass digested, g
Df = Any dilution factors.

12.3 Calculation for Filters

$$C(\mu g/m^3) = \frac{\mu g}{L}(\text{Reading}) \cdot \frac{0.100\,L}{\text{Strip}} \cdot \frac{9\ \text{Strips}}{\text{Filter}} \cdot \frac{\text{Filter}}{\text{Air Volume}\,(m^3)}$$

where:

C	=	Concentration of metal on filter.
Strip	=	1"x 8" area; exposed area is 1"x 7."
Filter	=	Total useable filter area 9" x 8" (exposed area is 9" x 7") (i.e., 9 strips/filter).
Air Volume	=	Air volume from field in m^3.

If air volume is supplied in liters, use

$$C(\mu g/m^3) = \frac{\mu g}{L}(\text{Reading}) \cdot \frac{0.100\,L}{\text{Strip}} \cdot \frac{9\ \text{Strips}}{\text{Filter}} \cdot \frac{\text{Filter}}{\text{Air Volume}\,(L)} \cdot \frac{1000\,L}{m^3}$$

where:

C	=	Concentration of metal on filter.
Strip	=	1"x 8" area; exposed area is 1"x 7."
Filter	=	Total useable filter area 9" x 8" (exposed area is 9" x 7") (i.e., 9 strips/filter).
Air Volume	=	Air volume from field in L.
One m^3	=	1000 L.

12.3 Calculation for Casettes

If air volume is supplied in cubic meters:

$$C(\mu g/m^3) = \frac{\mu g}{L}(\text{Reading}) \cdot \frac{0.100\,L}{\text{Casette}} \cdot \frac{\text{Casette}}{\text{Air Volume}\,(m^3)}$$

If air volume is supplied in liters:

$$C(\mu g/m^3) = \frac{\mu g}{L}(\text{Reading}) \cdot \frac{0.100\,L}{\text{Casette}} \cdot \frac{\text{Casette}}{\text{Air Volume}\,(L)} \cdot \frac{1000\,L}{m^3}$$

12.5 Often filters and casettes must be reported as µg/filter or µg/casette because air volumes are not available to the laboratory.

Control Limits for Routine Quality Control Samples

Table 12-2

CONTROL LIMITS FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameters Charted	Type of Samples Charted
LCS	All elements	Soil, water

Revision: 10	Status: Final	Method: 6010B	Minor Revision Date: 4/23/01

Table 12-3

ACCEPTANCE CRITERIA FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameter	Recovery (%)	RPD	Source
MS	Matrix spike	Project or 75-125	NA	Method
MSD (Control Chart Database Type = DUP)	Matrix spike duplicate	Project or 75-125	20% water 35% soils and filters	Laboratory
LCS	Laboratory control sample	80-120	NA	Method, Laboratory
CCV	Continuing calibration verification	90-110	NA	Laboratory
ICSAB	Interference check standard	80-120	NA	Method
ICV	Independent Calibration Verification	90-110	NA	Laboratory

Revision: 10	Status: Final	Method: 6010B	Minor Revision Date: 4/23/01

13.0 SPECIAL PROJECT REQUIREMENTS

13.1 U.S. Army Corps of Engineers (ACE)

13.1.1 Method Quantitation Limit (MQL) is between 3-10 times MDL. The low calibration standard concentration must be ≥3 times MDL. The Method Reporting Limit (MRL) may slide between two times the MDL and project-specific or regulatory requirements. Results below the MRL must be reported "ND." Results between the MQL and MRL must be reported as estimated (J flagged).

13.1.2 Initial Calibration Verification. The initial calibration curve shall be verified as accurate with a standard purchased or prepared from an independent source. This initial calibration verification (ICV) involves the analysis of a standard containing all of the target analytes, typically in the middle of the calibration range, each time the initial calibration is performed. The % recovery of each target analyte in the ICV is de-

termined from the initial calibration and compared with the specifications for the CCV in each method (except for mercury by CVAA) as outlined in the Shell Document Tables 7 - 14.

Note for methods which report several (>5) target analytes, a small percentage of sporadic marginal failures may be tolerated (i.e., will not trigger reextraction and analysis of the entire batch). The number of target analytes reported for the method will dictate the number of allowable QC failures as given below. Refer to the individual method tables for details on the implementation of this concept.

Number of Target Analytes	Number of Allowed Marginal Failures
5 - 15	1
16 - 30	2
31 - 45	3
46 - 60	4
61 - 75	5
76 - 90	6
91 - 105	7

The marginal failure allowance entails the application of an expanded acceptance criterion. If these QC criteria are not met, a new initial calibration must be performed.

13.1.3 ACE criteria are summarized in Table 13-1.

Table 13-1

Summary of Method Quality Objectives for Method 6010 ICP Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria
Initial Calibration (Shell document section 9.2.1.1)	Option 1 - 1 standard and blank, and a low-level check standard at MQL Option 2 - 3 standards and blank	Daily, using Option 2	Option 1- Low-level check standard ± 20% Option 2- r ≥ 0.995
Instrumental Precision (9.2.1.1)	%RSD 3 integrations (exposures)	Each calibration and calibration verification standards (ICV/CCV)	%RSD < 5%
Initial Calibration Verification (ICV) (9.3)	Mid-level (2nd source) verification	After initial calibration	%Recovery 90-110%
Initial Calibration Blank (ICB) (9.4)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes < MDL Check Sample (~2X MDL)
Interelement Check Standards (ICS) (8.1)	ICS-A - interferents only ICS-B - interferents and target analytes	Beginning of analytical sequence	%Recovery 80-120% for target analytes

Table 13-1

Summary of Method Quality Objectives for Method 6010 ICP Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria
Continuing Calibration Blank (CCB) (9.4)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes < MDL Check Sample (~2X MDL)
Continuing Calibration Verification (CCV) (9.5 / 9.5.1)	Mid-level verification	Every 10 samples and at end of analytical sequence	%Recovery 90-110%
Method Blank (MB) (10.2.1 / 11.4.1)	Interference-free matrix to assess overall method contamination	1 per sample batch	Analytes < MDL Check Sample (~2X MDL)
Laboratory Control Sample (LCS) (10.2.2 / 11.4.2)	Interference-free matrix containing all target analytes	1 per sample batch	%Rec = 80-120% Sporadic marginal failures[1]: %Rec = 60-140%
Matrix Spike (MS) (10.2.3 / 11.4.3 / 11.4.3.1)	Sample matrix spiked with all/subset of target analytes prior to digestion	1 per sample batch	%Rec = 75-125%
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD) (10.2.4 / 11.4.4)	Refer to text for MD or MS.	1 per sample batch	RPD ≤ 25%
Post Digestion Spike (PDS) (10.3.1 / 11.4.6)	Sample digestate spiked with all/subset of target analytes	As needed to confirm matrix effects	%Rec = 75-125%
Serial Dilution (SD) (10.3.2)	1:4 dilution analyzed to assess matrix effects	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results ± 10%
Method of Standard Addition (MSA) (12.2.1)	Method of quantitation	As needed for samples with suspected or confirmed matrix effects	r ≥ 0.995

[1]The number of Sporadic Marginal Failure (SMF) allowances depend upon the number of target analytes reported from the analysis. For instance, if between seven (7) to fifteen (15) metals are reported from the ICP analysis, one (1) SMF is allowed to the expanded criteria presented. If greater than 15 metals are reported from the ICP analysis, two (2) SMFs are allowed. Refer to Section 9.3 for additional information on the application of sporadic marginal failures.

Source: USACE, Shell for Analytical Chemistry Requirements, Version 1.0, November 2, 1998.

13.2 AFCEE - General Requirements

13.2.1 Reporting

13.2.1.2 MDL may not be > 1/2 reporting limit (RL) unless a variance was obtained.

13.2.1.3 Analyze low-level standards A, B, and C D to verify values at the RL.

13.2.1.4 Apply a "U" flag if analyte result <MDL; an "F" flag if MDL ≤ analyte result ≤ RL.

13.2.2 Calibration

13.2.2.1 All initial calibration curves, inorganic or organic, must be **verified with an independent source (vendor) standard** prior to sample analysis unless an independent vendor is unavailable and a variance was obtained.

13.2.2.2 The continuing calibration verifications are not required to be independent.

13.2.3 Batching

13.2.3.1 An AFCEE analytical batch is the environmental samples that are similar in composition (matrix) and are prepared at the same time and with the same **lot of reagents.**

13.2.3.2 A batch is not to exceed 20 samples. **The sample duplicate, MS, MSD are considered an environmental sample, therefore, the actual number of unique samples is less than 20.**

13.2.4 Laboratory Control Sample

13.2.4.1 Laboratory Control Sample for metals in a solid must be prepared using glass beads and spiked at a level ≤ the midpont of the calibration curve.

13.2.4.2 The LCS must be prepared from second source material.

13.2.4.3 The LCS must contain all target analytes unless noted differently under specific method requirements or a variance was obtained.

13.2.5 Matrix Spike/Matrix Spike Duplicate

13.2.5.1 In agreement with this SOP, AFCEE requires an MS/MSD, not an MS duplicate. The MS and MSD must be spiked at a concentration ≤ midpoint of the calibration curve for each analyte unless a variance was obtained.

13.2.5.2 AFCEE samples must be used for spiking.

13.2.6 Method blank must be less than RL unless a variance was obtained.

13.2.7 Holding times for analysis is calculated from the time of extraction completion to the time of completion of all **analytical runs including dilutions, confirmation, reanalyses.**

13.2.8 The ICSAB must contain both interfering and all analyte elements of known concentration.

13.3 NJDEP ~~RCRA Contract~~

~~**13.3.1** MS, MSD, and LCS must also contain boron and molybdenum.~~

~~**13.3.2** TCLP extracts for silver must be digested per Method 7760.~~

13.3.1 This method is suitable for preparation of aqueous samples containing silver concentrations up to 100 µg/L. For the analysis of water samples containing higher concentrations of silver, succeeding smaller volume, well mixed aliquots must be prepared until the analysis solution contains <100 µg/L silver.

13.3.2 The digestion of solid samples containing concentrations of silver >50 mg/kg (approximately 500 µg/L in digestate) should be treated such that a smaller portion of solid sample is digested to bring the raw value in the digestate to below 500 µg/L before conversion. Also, the digestion of tin from solid samples

should be prepared again using aliquots <1 g when determined sample concentrations exceed 1% (10,000 mg/kg).

13.4 North Carolina DENR

13.4.1 A low-level standard must be analyzed at or below the laboratory reporting limit with each analytical run.

14.0 SAMPLE DISPOSAL

14.1 Samples and standards are disposed of in the waste acid drum.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Soil pH			
Reviewer: D. Ruszczyk		Peer Reviewer: F. Lindauer	
Implementation Date: 1/27/00		Last Annual Review Date: 1/10/01	
Revision: 3	Status: Final	Method: SW-846, 9045C	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This procedure may be used to determine the pH in soils. This procedure is not to be used to measure the pH of soils for USEPA CLP soils.

2.0 METHOD SUMMARY

2.1 A portion of the sample is mixed with ASTM Type II water. The pH of the resulting aqueous solution is measured.

3.0 HEALTH AND SAFETY

3.1 All laboratory employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA library island shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 SW-846 Update 3, Method 9045C, June 1997.
4.2 Training SOP QA.5, A.23, and A.29.
4.3 Data Review SOP A.25.
4.4 Laboratory Quality Assurance Manual.
4.5 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 See standard list of definitions and acronyms in the Quality Assurance Manual.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Samples with very low or very high pH may give incorrect readings on the meter.
6.2 Temperature fluctuations will cause measurement errors.
6.3 Errors will occur when the electrodes become coated.

7.0 INSTRUMENTATION AND EQUIPMENT

Stir bar
100-mL Beakers
Accumet 925 pH Meter
Denver Instruments Model 225 pH Meter
Magnetic stirrer
pH Electrode
Temperature probe
Balance accurate to 1.0 mg
Centrifuge

8.0 REAGENTS AND MATERIALS

8.1 Reagent grade chemicals are to be used in all tests.

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Standard buffer 1.68	Lab Chem Inc., or equivalent	Use as is
Standard buffer 4.00	Fisher or equivalent	Use as is
Standard buffer 7.00	Fisher or equivalent	Use as is
Standard buffer 10.00	Fisher or equivalent	Use as is
Standard buffer 13.00	Lab Chem Inc., or equivalent	Use as is

Revision: 3	Status: Final	Method: SW-846, 9045C	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Solid	Standard	N/A	As soon as possible	Poly or glass 2-6 C
	NJDEP (BCM)		As soon as possible or within 7 days from sampling	

Revision: 3	Status: Final	Method: SW-846, 9045C	Minor Revision Date: None

10.0 PROCEDURE

10.1 Initial Calibration. Two-point standardization with automatic temperature entry.

10.1.1 The pH electrode system must be calibrated at a minimum of two points. (Use only two points as the make of meter does not temperature compensate with 3 points). The two points must bracket the ex-

pected pH of the samples and be 3 or more pH units apart, i.e., pH 7 and 1.68 or pH 7 and 13. The isopotential point should be set first using pH buffer 7.

10.2 Accumet 925

10.2.1 Press "mode" key until "pH" mode is displayed.

10.2.2 Place electrodes in first buffer, mixing gently. Press the "two point cal" key. The message display will show "enter buffer 1" or, if using an exact pH buffer, the buffer value will be in the data display and the message display will show "std. buffer 1". Note: the auto buffer recognition is only valid from 0-60 C.

10.2.3 Press the "enter" key if using an exact pH buffer or enter the value of the pH buffer and then press the "enter" key.

10.2.4 The message display will show "stabilizing MV 1". After a few seconds, the data display will show the MV output of the electrode. Press the "enter" key.

10.2.5 The data display will show a temperature value and the message display "ready temp 1". The temperature is read and automatically entered. Record pH reading and temperature.

10.2.6 The message display will show a "STD, BUFFER 2." Rinse electrodes with reagent water, then buffer to be used. Place electrodes in buffer, mixing gently. The data display will show a value near this buffer and the message display "enter buffer 2". Press the "enter" key if using an exact pH -buffer or enter the value of the pH buffer and then press the "enter" key.

10.2.7 The message display will show "stabilizing MV 2", then "enter MV 2". Press the "enter" key. Record pH reading and temperature.

10.2.8 All necessary data is now entered and the message display will show one of the following:

10.2.8.1 "XX.X'CPH'1". This means the efficiency factor is within acceptable range (0.95-1.05) and that samples can be analyzed.

10.2.8.2 "Efficiency" "Out of Tolerance". This means the efficiency factor is out of control and that the meter must be recalibrated prior to sample analysis.

10.3 Denver Instruments Model 225

10.3.1 Enter "MODE."

10.3.2 Enter "1" for pH.

10.3.3 Enter 'STANDARDIZE."

10.3.4 Enter "3" for clear buffers.

10.3.5 Enter "STANDARDIZE."

10.3.6 Enter "1" for auto-enter a buffer.

10.3.7 Prepare Buffer 7. Rinse electrode. Place electrode in solution. Stir moderately. Press "ENTER" to continue

10.3.8 Wait for the letter "S" to appeaer for stabilized pH. Record temperature and buffer readings.

10.3.9 Repeat for Buffer 4 steps 10.3.5-10.3.8.

10.3.10 Indicate in logbook which buffers were used for standardization.

10.4 Calibration Check. While gently stirring with a stir bar, measure the pH reading of a check buffer. When pH is stable, record pH reading and temperature. Compare results to acceptance criteria.

10.4.1 This buffer should be bracketed by the standardization buffers. Indicate in the logbook which buffer(s) was used as a check.

10.5 Sample Preparation and pH Measurement of Soils

10.5.1 Weigh 19-21g of soil in a 50-mL beaker, add 20 mL of ASTM Type II water and stir for 5 minutes.

10.5.2 Let the soil stand for about 1 hour or until the suspended clay settles. For samples which do not settle, centerfuge.

10.5.3 Insert the pH electrode and temperature probe into the suspension and measure pH. DO NOT MIX. Report results as "soil pH measured in water", in the pH logbook. Record temperature in logbook.

Instrument Conditions and Maintenance

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
pH Meter, Denver 225		Calibrate	With each use.
pH Meter, Accumet 925		Calibrate	With each use.

Revision: 3	Status: Final	Method: SW-846, 9045C	Minor Revision Date: None

11.0 DATA REDUCTION/EVALUATION/REPORTING

Calculate the relative percent difference (RPD) value between the sample result (S) and duplicate sample result (D) as follows:

$$RPD = \frac{|S - D|}{1/2(S + D)} \cdot 100$$

The vertical bar in the formula indicates the absolute value of the difference, hence RPD is always expressed as a positive value.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Routine Quality Control Samples

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Check Buffers (either a different pH or pH from a lot different than used for standardization).	Initially after calibration, every 10 samples and at the end of a batch.	Stock	± 0.1 of true value	Recalibrate, remeasure last 10 samples. Must calibrate with Buffer 10 if any sample is higher than pH of 7.
Duplicate sample	1 every batch of 20 samples	Prepare as a sample.	Project requirements, if specified ± 0.5 pH	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.

Revision: 3	Status: Final	Method: SW-846, 9045C	Minor Revision Date: None

Table 12-2

CONTROL CHARTED QUALITY CONTROL SAMPLES

QC Type		Analytes Charted	Matrix Charted
Duplicate		pH	Soil
Revision: 3	Status: Final	Method: SW-846, 9045C	Minor Revision Date: None

13.0 SPECIAL PROJECT REQUIREMENTS

NA

14.0 SAMPLE DISPOSAL

14.1 Pour aqueous layer down drain using copious amounts of water. In-house system will neutralize.

14.1.1 Less than 1.0 pH samples use metals digestion waste container, for aqueous layer.

14.2 Deposit soil in nonhazardous solid waste container.

15.0 EXAMPLE FORMS

N/A

END OF SOP

TITLE: TOC in Sediment and Soil - Lloyd Kahn			
Reviewer: D. Ruszczyk		Peer Reviewer: F. Lindauer	
Implementation Date: 6/13/01		Last Annual Review Date: 6/1/01	
Revision: 5	Status: Final	Method: Lloyd Kahn	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) is used to determine the concentration of total organic carbon in soil and sediment. The effective range of the Lloyd Kahn method is from 2000 to 800,000 mg/kg. The upper limit is based on 40 mg of sample and the lower limit on 1 g of sample and applying the respective calibration standards' masses. Results to the MDL may be reported at the client's request.

1.2 The sample results are reported on a dry weight basis by virtue of determination of percent solids on a second portion of the sample.

2.0 METHOD SUMMARY

2.1 The Lloyd Kahn method determines the concentration of Total Organic Carbon in a solid sample by pyrolyzing the sample in a stream of oxygen at approximately 900° C. The gas stream is then dried and analyzed for carbon dioxide by non-dispersive infrared detection. Inorganic carbon from carbonates and bicarbonates is removed prior to analysis by acid treatment of the sample.

2.2 The method uses dissolved KHP as calibration standard. This procedure is modified to use solid dextrose as calibration standards.

2.3 The method uses phosphoric acid to eliminate inorganic carbonates and bicarbonates. This procedure uses hydrochloric acid.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E Inc., Analytical Services Center Chemical Hygiene Plan, located in the QA Library Island shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Determination of Total Organic Carbon in Sediment, July 27, 1988, by Lloyd Kahn.
4.2 Shimadzu Manual for 5000A.
4.3 Training SOP QA.5, A.23, and A.29.
4.4 Data Review SOP A.25.
4.5 Laboratory Quality Assurance Manual (L-001).
4.6 MDL SOP A.18.
4.7 Corrective Action Procedure, QA.1.
4.8 Checklist C-164 for analyst and peer review.

5.0 DEFINITIONS/ACRONYMS

5.1 **TC**: Total Carbon, **IC**: Inorganic Carbon.

5.2 **The following are possible remarks displayed when using the SSM-5000A:**

T: The peak is not completed within designated time period.

H: The peak height has exceeded full scale.

P: Measurement performed with the sample boat in an abnormal position.

B: Baseline not stable during measurement.

F: The temperature of the furnace exceeded the designated range during measurement.

5.3 **TC - TOC** based on Lloyd Kahn method.

5.4 **LCS/ICV** - Laboratory Control Sample/Initial Calibration Verification - A 2nd lot number (2nd source) of dextrose used to verify that the calibration curve is valid.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 In many cases, the various components in a solid sample are not mixed together uniformly, as they are in liquid samples. For this reason, it is extremely important with solid samples to obtain an aliquot which is as representative as possible of the sampled material.

6.2 Volatile organics may be lost in the decarbonization step resulting in a low bias.

6.3 Bacterial decomposition and volatilization of the organic compounds are minimized by maintaining the sample at 2-6 C and analyzing within the specific hold time.

6.4 The combustion-oxidation reaction (TC measurement) and acidification reaction involving carbonate acid (IC measurement) are more efficient when the sample particles are smaller in size. Mix or breakup large chunks but do not grind.

6.5 Samples with high levels of carbonates such as sea sand and limestone are not appropriate for this method.

7.0 INSTRUMENTATION AND EQUIPMENT

Drying ovens, 105 C and 180 C
Analytical balance capable of weighting to 0.001 g
Shimadzu 5050A Total Organic Carbon Analyzer – resistance furnace, nondispersive infrared detector (SSM 5000A)
Ceramic combustion boats
Disposable pasteur pipettes
Ceramic fiber
TOC Control Software Verson 4.0

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard, Reagents, and Consumables

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Concentrated hydrochloric acid (HCl)	Fisher or equivalent	12 M HCl
Purified oxygen	Rodgers Welding or equivalent	Carbon dioxide free containing < 1 mg/L hydrocarbons.
Dextrose-Anhydrous (D-Glucose) (2 lots required)	Fisher or equivalent	Neat
Tungsten oxide (WO)	Fisher or equivalent	Catalyst
Ultra high purity compressed air	Rogers Welding VHP or equivalent	CO_2 free containing < 1 mg/L hydrocarbons

Revision: 5	Status: Final	Method: Lloyd Kahn	Minor Revision Date: None

8.2 See Table 8-2, Standard and Reagent Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
2 M HCl	HCl, Concentrated	Slowly add 166 mL of concentrated HCl to ASTM Type II water. Dilute to 1 L. Good for 6 months.	2 M HCl
ICV	Dextrose (2nd Source)	Weigh 40 mg into pretreated boat. Run initially after a curve.	16,000 µg C (400,000 mg/kg based on 40 mg sample size)
CCV/LCS	Dextrose (same lot as calibration)	Weigh 40 mg into pretreated boat	16,000 µg C (400,000 mg/kg based on 40 mg sample size)

Revision: 5	Status: Final	Method: Lloyd Kahn	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Solid	Standard	NA	14	Glass, 2-6 C

Revision: 5	Status: Final	Method: Lloyd Kahn	Minor Revision Date: None

10.0 PROCEDURE

10.1 Before instrument use check to make sure that the air flow is 500 mL/min with the flow meter.

10.2 Daily Instrument Calibration for TC Analysis as it appears on the instrument computer screen.

Table 10-1

GENERAL CONDITIONS

Description	Source
TC Catalyst	Normal Sens
Syringe Size	250 µL
Number of Washes	1
Unit of Concentration	%
Auto Ranging & Inj Vol	Auto Change
Auto Regeneration of IC	Off
Furnace on/off	SSM
Injection Speed	STD
ESU	Not Used
Bubble Removal	Off
Syringe Wash	STD
Cell Length	W-Short
TOC or SSM	SSM
Printer Device	Int New
Page Length	******
Calibration Curve Form	Least Squares
POC	Not Used
MEAS Interval (TC/NPOC)	0 Seconds
MEAS Interval (IC/POC)	0 Seconds

Revision: 5	Status: Final	Method: Lloyd Kahn	Minor Revision Date: None

Daily Instrument Calibration For TC Analysis as it appears on the instrument computer screen

Table 10-2

CALIBRATION CONDITIONS

Amount Select	% Carbon	Amount	µg C (Carbon)
1st STD Concentration	40%	80.0 mg (Dextrose)	32,000
2rd STD Concentration	40%	20.0 mg (Dextrose)	8,000
3rd STD Concentration	40%	5.0 mg (Dextrose)	2,000
4th STD Concentration	0	0.0 mg (empty boat)	0
Range x30			

Revision: 5	Status: Final	Method: Lloyd Kahn	Minor Revision Date: None

10.2.1 Cleaning of boats.
10.2.2 Scrape boats of all residue with spatula.
10.2.3 Put 2 M HCl in beaker with boats and sonicate for 15-30 minutes.
10.2.4 Rinse boats again after sonication with ASTM Type II water.
10.2.5 Put in 180 C oven for 30 minutes.

10.3 Start Up/Maintenance – PC Control

10.3.1 Turn on air tank, analyzer, and set carrier gas to 150 mL/min.
10.3.2 Check copper scrubber inside door of analyzer. When ¾ the length of the scrubber is black, replace the scrubber (halogen scrubber).
10.3.3 Double click "TOC Control" icon.
10.3.4 Click "Options" menu.
10.3.5 Click "SSM" tab. Choose Instrument Conditions.
10.3.6 Click "OK" at bottom of window.
10.3.7 Analyzer will go to ready state when furnace reaches temperature (it takes approximately 30 minutes). Check baseline at 30x.
10.3.8 Check humidifier level. Should be between two white lines. Add one sodium hydroxide pellet every 3 months to deionized water.
10.3.8.1 Check 25% H_3PO_4 level. Should be at 125 mL.
10.3.8.2 Check for bubbles in IC reactor.
10.3.8.3 Check drain pot level inside TOC 5050A. Should always be full.
10.3.8.4 Check all lines to see if any are disconnected.
10.3.8.5 Check bottle tops and make sure all are tightened.
10.3.8.6 Perform gross leak check. Pinch both tubes connected to CO_2 absorber lines, watch for bubbles in drain pot. This indicates there are no leaks in system.
10.3.8.7 Check drain separator on SSM 5000A and make sure it is full with ASTM Type II water.
10.3.8.8 Check for any clogs in SSM combustion tube and the flow lines.

10.4 Connecting the Instrument

10.4.1 Click on "Measure" menu.
10.4.2 Select "Connect."
10.4.3 Insert blank into injection tube.
10.4.4 Click "OK."

10.5 Creating the Sample Table

10.5.1 Click "Options" menu.
10.5.2 Select "General Information."
10.5.3 Enter the following information:
10.5.3.1 User: Analyst's name.
10.5.3.2 Title: Batch number.
10.5.3.3 Comment: Client names and work order numbers.
10.5.4 Click "OK."
10.5.5 Select "Measurement Parameters" from Options menu.
10.5.6 Enter the following: STOC.
10.5.6.1 Unit: % C.
10.5.6.2 No. of injections: 1.
10.5.6.3 Max. no of inj.: 1.
10.5.6.4 No. of Washes: 0.
10.5.6.5 Sparge time: 0.
10.5.6.6 Max SD: 200.
10.5.6.7 Max CV: 2.0.
10.5.6.8 Click "OK."
10.5.7 Leave default parameters.
10.5.8 In the Options menu, choose "Display Settings."
10.5.9 For each table, (Sample, Injection, and Statistics), click "Select All."
10.5.10 Click on "File" menu.
10.5.11 Select "Save As."
10.5.12 Enter "S Today's date.toc."

10.6 Setting Up the Standard Runs

(Note: Soil curve is good for 6 months. Does not have to be made each day like WTOC.)
10.6.1 Click in the "first line" of the sample table.
10.6.2 From the Edit menu, choose "Insert Standard."
10.6.3 Choose "New" from the Calibration Curve List dialog box.
10.6.4 Enter "Title" and "File Name" (i.e., Title: scal_1....File Name: scal_1.cal).
10.6.5 Change the Calculation Method to "Lin. Regression without Zero Shift."
10.6.6 Do not change range or injection volume.
10.6.7 Verify range and change to 30x if necessary for STOC.
10.6.8 Click "Data" tab.
10.6.9 Enter the standard run information.
10.6.10 Click "OK."
10.6.11 Click on "Conditions" tab.
10.6.12 Verify selections.
10.6.13 Click "OK."
10.6.14 Choose "Yes" to save calibration information.
10.6.15 Choose "OK."
10.6.16 Save the sample table created so far: Click "File" menu, then select "Save."

STOC Data Calibration Table					
Sample Name (mg)	Sample ID	Concentration	Range	Injection	Maximum Injection
80.0	STD1	40.0	30x	1	1
20.0	STD2	40.0	30x	1	1
5.0	STD3	40.0	30x	1	1
0.0	STD4	40.0	30x	1	1

10. 7 Setting Up the Unknown Run

10.7.1 Click on 5th line after making new curve or click on 1st line if using a prior made curve.
10.7.2 Click "Edit" menu.
10.7.3 Select "Insert Sample."
10.7.4 The "Insert Sample" opens the "Method" dialog box.
10.7.5 Select "New" to create a new method.
10.7.6 Enter the following information:
10.7.6.1 LK76 (Lloyd Kahn SOP GAC.76).
10.7.6.2 Comment: "STOC."
10.7.6.3 Sample Name: "<Untitled>."
10.7.6.4 Sample ID: "<Untitled>."
10.7.6.5 Analysis: "TC."
10.7.6.6 Dilution: "1.0."
10.7.6.7 Type "Unknown."
10.7.7 Click "OK."
10.7.8 Select the "TC" tab for soils.
10.7.9 With the Cursor in the "Cal Curves 1 field."
10.7.10 Select "Browse."
10.7.11 Choose curve you want to use.
10.7.12 Select "OK."
10.7.13 Click "Yes" to use the calibration file to fill out the method.
10.7.14 Click "OK" when all is competed correctly on "TC" tab.
10.7.15 Select "OK" to close the Method File list dialog box.
10.7.16 Save the completed sample table - click the "Save" button on the toolbar.

10.8 Making the Analyses

10.8.1 Prepare calibration curve standards.
10.8.2 Verify status of instrument before beginning analyses.
10.8.3 Click "View" menu.
10.8.4 Choose "Background Monitor."
10.8.5 The status of the baseline should be OK for each parameter at the 30x range.
10.8.6 Close the "Background Monitor" window after verifying everything is OK.
10.8.7 Click "Measure" menu.
10.8.8 Choose "Start" or click on "Start" button of instrument control bar.
10.8.9 The analyses will begin.
10.8.10 Click the "View" menu.

10.8.11 Open the "Realtime Window" to monitor the status of the measurements.

10.9 Evaluating the Results of the Analyses

10.9.1 Click "View" menu.
10.9.2 Select "Calibration Curve."
10.9.3 Select the "Curve" tab to see the curve data in graph form.
10.9.4 Select the "Data" tab to see curve statistics.
10.9.5 To see peak shape of all injections in a sample table:
10.9.5.1 Select row.
10.9.5.2 Click "View" menu.
10.9.5.3 Select "Peak Profile."
10.9.5.4 Select "Display."
10.9.5.5 Or select the "Peak Profile" button in the toolbar.
10.9.6 Click "View" menu.
10.9.7 Select the "Statistics (Summary)" table.

10.10 Printing a Report

10.10.1 Click "File" menu.
10.10.2 Select "Page Setup."
10.10.3 Make the following selections on the "General" tab:
10.10.3.1 General Information.
10.10.3.2 Organization.
10.10.3.3 User.
10.10.3.4 Title.
10.10.3.5 Instrument ID.
10.10.3.6 Filename.
10.10.3.7 Comment.
10.10.3.8 Instrument Conditions.
10.10.3.9 Instrument Attachments.
10.10.4 Make the following selections on the "Cal Curves" tab:
10.10.4.1 Print cal curves.
10.10.4.2 Filename.
10.10.4.3 Title.
10.10.4.4 Calculation method.
10.10.4.5 Unit.
10.10.4.6 Range.
10.10.4.7 Analysis.
10.10.4.8 Graph.
10.10.4.9 Slope.
10.10.4.10 Intercept.
10.10.4.11 R^2.
10.10.4.12 Number of washes.
10.10.4.13 Injection volume.
10.10.4.14 Spargetime.
10.10.4.15 Max SD.
10.10.4.16 Max CV.
10.10.4.17 Print Data.
10.10.4.18 Sample Name.

10.10.4.19 Sample ID.
10.10.4.20 Concentration.
10.10.4.21 Number of injections.
10.10.4.22 Mean Area.
10.10.4.23 SD.
10.10.4.24 CV.
10.10.4.25 Maximum number of injections.
10.10.5 Click on "Samples" tab and make the following selections:
10.10.5.1 Analysis.
10.10.5.2 Area of Confidence.
10.10.5.3 Calibration Curves.
10.10.5.4 Concentration.
10.10.5.5 Result.
10.10.5.6 CV.
10.10.5.7 Date/Time.
10.10.5.8 Dilution.
10.10.5.9 Mean Area.
10.10.5.10 Method.
10.10.5.11 Sample ID.
10.10.5.12 Sample name.
10.10.5.13 Type.
10.10.5.14 Unit.
10.10.5.15 Peak profile.
10.10.5.16 SD.
10.10.5.17 Unknown.
10.10.5.18 Standard.
10.10.6 Click on "Injections Table" tab.
10.10.7 Select "Select None."
10.10.8 Click on "Statistics Table" tab.
10.10.9 Select "Select All."
10.10.10 Click on "Page Setup" tab.
10.10.11 Add Title: Batch number.
10.10.12 Click on "User, Organization, Date/Time, Page No. for Footer."
10.10.13 Check off "New Page after every Sample."
10.10.14 Go back to "General" tab.
10.10.15 Select "Print Preview."
10.10.16 Click "Print" when satisfied with report.
10.10.17 Save data by clicking on "Save" button.

10.10.18 Enter data information sample table like this for STOC:

Sample Name	Sample ID
LCS-000718142R	LCS
MB-000718142R	MB
0007038-19A	Sample
0007038-19A	Quad
0007038-19B	Quad
0007038-19A	Quad
0007038-20A	Sample
CCV-1	CCV
CCB-1	CCB

Just the weights are needed. Do not enter weights into sample table, enter weights in pop-up window before analysis.

10.11 Initial Calibration for TC Analysis – Standalone

10.11.1 Weigh 80 mg of Dextrose (D-Glucose) into a ceramic boat. [Note: The weighed sample is set on the bottom of the sample boat toward the front (TC furnace side).]

10.11.2 Place ceramic boat and contents in sample port of SSM-5050A.

10.11.3 Close sample port and press F1 key (next). Screen will display to press "start;" **do this.**

10.11.4 Instrument will now read "push sample boat to measuring;" **do this.**

10.11.5 When 80 mg std is completed; screen will read "completed". Pull sample boat to "cooling position." After 30 seconds, pull lever to "sample change position."

10.11.6 Press F1 (next); the instrument will automatically print out results.

10.11.7 Follow 10.3.2 – 10.3.6 for remaining standards.

10.11.8 Calibration curve must be $r \geq 0.995$. Curve is good for 6 months or until ICV/CCV fall out.

10.12 Determination of Percent Solids - Standalone

10.12.1 Perform a percent solids determination on a separate (second) portion of sample following the procedure in the SOP GAC.14. The final sample concentration is reported on a dry weight basis.

10.13 Sample Analysis for TOC - Standalone

10.13.1 Mix sample if necessary but do not grind.

10.13.2 Weigh up to 1.0 g of sample into ceramic boat. Record weight in mg. (Note: If samples are high in total carbon – a smaller amount is acceptable. Do not use less than 125 mg before consulting supervisor). If samples are high in percent moisture, a larger amount may be used.

10.13.3 Add just enough drops of 2 M HCl to wet the sample. [Note: This is a pretreatment for removing IC. Inorganic carbon from carbonate and bicarbonates is removed by this acid treatment.]

10.13.4 Put ceramic boat and contents in drying oven at 103-105 C for 10 minutes or until sample is dry. [Note: Use tweezers.]

10.13.5 Add a spatula-tip amount of WO catalyst.

10.13.6 Place ceramic fiber over sample to prevent particle spattering in 900 C oven.

10.13.7 Place ceramic boat and contents in sample port of SSM-5050A.

10.13.8 Close sample port and press F1 key (next). Screen will display press "start;" **do this.**

10.13.9 Instrument will now read "push sample boat to measuring;" **do this.**

10.13.10 When sample is completed; screen will read "completed." Pull sample boat to cooling position, after 30 seconds, pull lever to "sample change position."
10.13.11 Press F1 (next) key; the instrument will automatically print out results.

10.14 Shutdown of TOC Analyzer - Standalone
10.14.1 Return to main menu.
10.14.2 Standby options.
10.14.3 Turn off instrument, **PRESS 1**, and then the **ENTER** key. Next, press the **STANDBY (F1)** key.
10.14.4 Screen will display "30 minute" countdown for cooling.
10.14.5 After cooling countdown turn off the monitor, the SSM500A and both oxygen tanks.

10.15 Daily and Continuing Calibration – PC Control and Standalone
10.15.1 Check the instrument calibration daily by running the CCV. If the CCV is outside acceptance limits, prepare a new curve (see Section 10.3).
10.15.2 Run Sequence
ICV (if applicable)
CCV
LCS
Up to 10 analyses (include MB, one sample run in quadruplicate)
CCV
CCB
Up to 10 analyses
CCV
CCB
End with CCV and CCB

Table 10-4

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Fill drain separator with ASTM Type II water when water level is low			
Oxygen Tank	Pressure less than 500 psi	Change Tank	When needed
Ceramic Boats	Poor baseline – Poor readings	Check port seal. Reclean boats. Check catalyst tube.	When needed
SSM- 5050A	Abnormality in measurement sensitivity or repeatability.	Check for leaks – see manual for Figure 7.1.	When needed
Compressed Air	Pressure less than 500 psi	Change tank	When needed
O-Rings	Gas leaks due to deformation or scratched "O" rings	Replace ring – see manual for figures 7.3 and 7.4.	When needed
Combustion Tube	Poor reproducible peaks, erratic baseline, tube clogged	Repack tube with new catalyst	When needed

Revision: 5	Status: Final	Method: Lloyd Kahn	Minor Revision Date: None

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 Lloyd Kahn: TOC in Sediment and Soil TC = TOC

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL Sediment/Soil (Units)
Lloyd Kahn TOC	T	2000 mg/kg based on 1 g sample

Revision: 5	Status: Final	Method: Lloyd Kahn	Minor Revision Date: None

Key Type:

T = Compound/analyte is target analyte routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike.
L = Compound/analyte is routinely used as a LCS spike.
Q = Compound/analyte is used as s surrogate spike.

11.2 Method Calculations

11.2.1 The instrument parameters have been set up to print results in %C. This equation is a check on the instrument. The result is dry weight corrected in the LIMS.

a.)

$$\%C = \frac{\text{Found } \mu g\, C}{\text{input sample size (mg)} \cdot 1000\ \mu g/mg} \cdot 100\%$$

b.)

$$\text{Sample concentration} = \left(\frac{mg}{kg} TOC \right) = \frac{\%C}{100\%} \cdot \frac{1000\,mg}{1\,g} \cdot \frac{1000\,g}{kg}$$

If the input sample size is different from the basis sample size (e.g., if 40 mg is input for the ICV but it is desired to report to a 1-gram basis:

c.) Sample Concentration

$$\frac{\text{TOC mg}}{\text{kg}} = \frac{\%C}{100\%} \cdot \frac{\text{input sample (mg)}}{\text{actual (basis) sample size (g)}} \cdot \frac{1000\,\text{g}}{\text{kg}}$$

This equation reduces to equation (b) above when the input sample size is the same as the actual size.

d.)

$$\text{Spike added concentration}\left(\frac{\text{mg}}{\text{kg}}\right) = \frac{\mu\text{g C added from dextrose}}{\text{weight (g) of sample}} = \frac{\text{mg dextrose added} \cdot 0.4 \cdot 1000\,\text{g/kg}}{\text{weight (g) of sample}}$$

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
ICV	After each initial calibration	40 mg of 2nd source dextrose	80-120%	Recalibrate
LCS	Beginning of a batch and one per batch	40 mg dextrose	See Control Limit Summary	Recalibrate with new multipoint curve
CCV	Beginning of a batch and after every 10 analyses, and at the end of a batch	40 mg of calibration lot dextrose	80 - 120%	Recalibrate instrument. Repeat previous 10 analyses.
CCB	After every CCV	An empty boat with 3 drops 2 M HCl	< 2000 mg/kg based on 1 g basis	Make sure boat is clean
MB (Method Blank)	After LCS	An empty boat with 3 drops 2 M HCl	< 2000 mg/kg based on 1 g basis	Make sure boat is clean
Quadruplicate analysis	1 per batch of 20 or fewer samples	Three additional aliquots of sample	Project requirements if specified or RSD ≤30%	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
MS (Matrix Spike)	When requested by client	Aliquot of sample plus a known amount of 2nd source dextrose[1]	Project requirements if specified	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Revision: 5	Status: Final	Method: Lloyd Kahn	Minor Revision Date: None	

[1]Spike with a known amount of dextrose to achieve a spike concentration within the calibration range. See Table 10-3.

Control Limits for Routine Quality Control Samples

Table 12-2

CONTROL LIMITS FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameters Charted	Type of Samples Charted	Source of Limits	Frequency Updated
LCS/ICV	TOC	Solids	In-house	Annually
Revision: 5	Status: Final	Method: Lloyd Kahn	Minor Revision Date: None	

13.0 SPECIAL PROJECT REQUIREMENTS

13.1 Griffis

13.1.1 Requires sample spikes at a frequency of 1 per 20 samples or fewer with 75 to 125% recovery.

13.1.2 A second aliquot of the sample is prepared and spiked with 40 mg of the 2nd source dextrose.

13.2 New Jersey Hexchrome Sites (Kimball). Follow the procedure in Section 10.4 with the following modifications.

13.2.1 Weigh 125 mg (a larger sample size, e.g., 250 mg may be used at the start if sample interferences such as tailing are absent) of as-received sample.

13.2.2 The quantitation limit for 125-mg sample size is 16,000 mg/kg. (The quantitation limit for 250-mg sample size is 8,000 mg/kg.)

13.2.3 The MDL (6/8/00) for a 125 mg sample (dry weight adjusted) is 1820 mg/kg (for 250 mg sample, 910 mg/kg).

13.2.4 If 125 mg sample was used, report values between the 1900 mg/kg (nominal MDL) and 16,000 mg/kg with a "J" flag. (If 250-mg sample was used, report values between 1000 mg/kg [nominal MDL] and 8,000 mg/kg with a "J" flag.)

13.2.5 If 125 mg sample was used, report values below 1900 mg/kg as "ND." (If 250-mg sample was used, report values below 1000 mg/kg as ND.)

13.2.6 Any values that are not detected at concentrations above 4000 mg/kg must be reanalyzed with a larger sample size.

13.2.7 One BCM sample must be chosen as a matrix spike.

13.2.8 Use the BCM project-specific requirements in Table 13-1 to replace the similar quality control requirements given in Table 12-1. The other QC specified in Table 12-1 (i.e., MB, CCV, CCB) must also be performed.

Table 13-1

ROUTINE QUALITY CONTROL SAMPLES – NEW JERSEY HEXCHROME SITES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
LCS/ICV	Beginning of a batch	40 mg of 2nd source dextrose	85-115%	Recalibrate with new multipoint curve.
Quadruplicate	1 per batch of 20 or fewer samples	Three additional aliquots of sample	30% RSD	Evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Matrix Spike	When requested by client	Aliquot of sample plus a known amount of 2nd source dextrose[1]	Project requirements, if specified	Evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Revision: 5	Status: Final	Method: Lloyd Kahn	Minor Revision Date: None	

13.3 For clients that wish spiking, prepare an appropriate spike level (approximately twice indigenous level if known) according to the following table.

Table 13-2

SPIKE AMOUNT

Amount of dextrose added to sample	Carbon (µg)	mg/kg of spike added to 1.0 g sample
10.0 mg	4000	4000
20.0 mg	8000	8000
30.0 mg	12000	12000
40.0 mg	16000	16000

Revision: 5	Status: Final	Method: Lloyd Kahn	Minor Revision Date: None

14.0 SAMPLE DISPOSAL

14.1 Residual sample and/or boats may be disposed of as nonhazardous solid waste.

14.2 See SOP A.10 for additional information on waste disposal.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Total Cyanide Analysis			
Reviewer: C. Okal		Peer Reviewer: F. Lindauer	
Implementation Date: 11/21/00		Last Annual Review Date: 11/20/00	
Revision: 11	Status: Final	Method: ILM04.1, 9012A, 335.2, 335.3, 335.4	Minor Revision Date: 2/9/01

1.0 SCOPE AND APPLICATION

1.1 This SOP covers the analysis of all cyanide distillates by semi-automated colorimetric analysis.

1.2 This procedure may be referenced to ILM04.0 when analyzing CLP samples or to 9012A (modified by MIDI) when analyzing SW-846 samples or to Method 335.3 when analyzing NPDES/SPDES samples or to Method 335.4 when analyzing drinking water samples.

2.0 METHOD SUMMARY

2.1 The MIDI distillation procedure is taken from USEPA Method 335.2 CLP-M. HCN is released from cyanide complexes in the midi distillation unit and absorbed in a scrubber containing sodium hydroxide solution using one of the following preparation SOPs: GAC.106 (Method 9012A – modified by MIDI for soil and water); GAC.118 (CLP Water); GAC.119 (CLP Soil); GAC.122 (Method 9013/9010B). The sodium hydroxide solution is then analyzed using a Lachat 8000 semi-automated flow injector analyzer.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 USEPA Statement of Work for Inorganic Analysis ILM0 4.1, Method 335.2 CLP-M.

4.2 Method 335.3, Methods for the Chemical Analysis of Water and Wastes, USEPA.

4.3 Method 335.4, Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.

4.4 General Lachat start up SOP GAC.64.

4.5 Method 9012A (rev.1, 12/96), SW-846, Update III, June, 1997.

4.6 Checklist C-162 for analyst and peer review.

4.7 Training SOP QA.5, A.23, and A.29.

4.8 Data Review SOP A.25.

4.9 Laboratory Quality Assurance Manual.

4.10 MDL SOP A.18.

4.11 Corrective Action Procedure, QA.1

5.0 DEFINITIONS/ACRONYMS

5.1 See general list in the Quality Assurance Manual.

5.2 **Carrier** - Solution used to carry samples through Lachat Analyzer.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 The normality of sodium hydroxide is extremely important as variance in normality can cause negative peaks on the Lachat system.

6.2 Volatile constituents may cause volatilization during analysis because heat is used to facilitate the color reaction. If this occurs heating unit is not used for those samples. QC is also run and should be still within acceptable limits.

7.0 INSTRUMENTATION AND EQUIPMENT

Lachat 8000 auto analyzer
Cyanide manifold and interference filter
Volumetric flask (100-mL Class A)
Balance, accurate to 0.10 mg
Volumetric pipettes (Class A)
50-mL Burette

8.0 REAGENTS AND MATERIALS

8.1 **See Table 8-1, Summary of Standards, Reagents and Consumables**

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Sodium hydroxide (NaOH)	Fisher or equivalent	Pellets
Potassium cyanide (KCN, 2 different vendors or lot numbers)	Fisher, J.T. Baker, or equivalent	Neat
Silver nitrate ($AgNO_3$)	Fisher or equivalent	Neat
Sodium chloride (NaCl)	Fisher or equivalent	Neat
Potassium chromate (K_2CrO_4)	Fisher or equivalent	Neat
p-dimethylaminobenzalrhodanine (rhodanine indicator)	Eastman Kodak or equivalent	Neat
Anhydrous potassium dihydrogen phosphate	Fisher or equivalent	Neat
Pyridine	Fisher or equivalent	Instrument grade
Barbituric Acid	Fisher or equivalent	Neat
Hydrochloric Acid (HCl)	Fisher or equivalent	Concentrated
Chloramine-T	Fisher or equivalent	Neat
Silver Nitrate Solution	Fisher or equivalent	0.0192 N; good 6 months

Revision: 11	Status: Final	Method: ILM04.1, 9012A, 335.2, 335.3, 335.4	Minor Revision Date 2/9/01

8.2 Standard Solution Preparation

Degas noted solutions for 3 -5 minutes with helium before use.

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Added to ____	Final Concentration
Calibration stock (for preparing calibration standards and CCV. Used also in preparative SOP to prepare distilled LCSL and LCSH.)	Potassium cyanide (KCN)	In a 1-L volumetric flask, dissolve 2.0 g potassium hydroxide in approximately 500 mL ASTM Type II water. Add 2.51 g potassium cyanide (Note: Very toxic). Dilute to the mark with ASTM Type II water. **Standardize the stock solutions every week during use with standard silver nitrate solution** (see Table 8-3). This stock is independent of the Reference Stock. Note different KCN lot numbers. **Prepare fresh every 6 months.**	1000 mg/L nominal value
Calibration working standard	Calibration stock	Use the concentration of the Calibration stock determined from the standardization to prepare a 10 mg/L working standard using syringes. Pipet approximately 1.0 mL Calibration stock into a 100-mL volumetric flask. Dilute to mark with 0.25N NaOH. When preparing in this manner, adjust the calibration standard concentration to 1/100 of the determined standardization value of the stock. Make fresh each day of use.	10 mg/L
Reference stock (Use for ICV. Also used in preparative SOP to prepare MS, LCS, and MSD.)	Potassium cyanide (KCN). Different lot from calibration stock.	**This stock is independent of the calibration Stock. Note different KCN lot numbers.** In a 1-L volumetric flask, dissolve 2.0 g potassium hydroxide in approximately 500 mL ASTM Type II water. Add 2.51 g potassium cyanide (Note: Very toxic). Dilute to the mark with ASTM Type II water. **Standardize the stock solution every week during use with standard silver nitrate solution (Table 8-3). Prepare fresh every 6 months.**	1000 mg/L Nominal value
Reference spiking solution	Reference stock	1.0 mL Reference stock into a 100-mL volumetric flask. Dilute to mark with 0.25N NaOH. Make fresh each day of use.	10 mg/L Nominal

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Added to ____	Final Concentration
Carrier	Sodium hydroxide	Dissolve 10 g sodium hydroxide in 800 mL ASTM Type II water. Cool and dilute to 1 L with ASTM Type II water. **Good for 6 months. Degas with helium for 3-5 minutes before use.**	0.25 N
Color reagent	Pyridine barbituric acid	Pyridine barbituric acid: In a fume hood, in a 1-L volumetric flask, add 15.0 g barbituric acid and 100 mL ASTM Type II water. Rinse down sides to wet the barbituric acid. Add 75 mL pyridine while stirring and mix until the barbituric acid is dissolved. Add 15 mL concentrated HCl; mix. Dilute to the mark with ASTM Type II water. Do not degas. **Prepare every month**. Store in amber bottle.	NA
Chloramine-T solution	Chloramine-T	Dissolve 2.0 g chloramine-T hydrate in 500 mL ASTM Type II water. **Prepare fresh daily. Degas before use.**	NA
Phosphate buffer	Potassium dihydrogen phosphate anhydrous (KH_2PO_4)	Dissolve in a 1-L volumetric flask 97 g anhydrous potassium dihydrogen phosphate (KH_2PO_4) in 800 mL ASTM Type II water. Dilute to the mark with ASTM Type II water. **Good 6 months. Degas before use.**	0.71 M
Purchased pre-standardized $AgNO_3$ or titrant listed	Silver nitrate solution ($AgNO_3$)	Dry $AgNO_3$ at 104 C to constant weight at 40 C. Weigh out 3.2647 g of dry $AgNO_3$ (correct for stated assay of $AgNO_3$) Dissolve and dilute to 1 L with ASTM Type II water. **Good 6 months**. Store in glass amber bottle.	0.0192 N
Primary standard	Sodium Chloride (NaCl)	Dissolve 0.8240 g NaCl which has been dried at 104 C in approximately 800 mL ASTM Type II water. Dilute to 1 L with ASTM Type II water. **Good 6 months**. (1.00 mL = 500 μg Cl^-).	0.0141 N
Indicator (K_2CrO_4)	Potassium chromate	Dissolve 50 g K_2CrO_4 in a sufficient volume of ASTM Type II water. Add $AgNO_3$ solution until a definite red precipitate is formed. Let solution stand for 12 hours, filter, and dilute to 1 L with ASTM Type II water. **Good 6 months**.	NA

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Added to ____	Final Concentration
Indicator	Rhodanine (p-dimethylamino-benzalrhodanine)	Dissolve 20 mg p-dimethylaminobenzal-rhodanine in 100 mL acetone. **Good 6 months.**	NA
Revision: 11	Status: Final	Method: ILM04.1, 9012A, 335.2, 335.3, 335.4	Minor Revision Date: 2/9/01

Table 8-3

FINAL CONCENTRATIONS DETERMINED FROM STANDARDIZATION STANDARDIZATION TABLE

Standardization of Cyanide Stock Solution	Pipet 10.0 mL of 1000 mg/L CN stock solution into a beaker. Add 0.5 mL Rhodanine indicator solution and mix. While stirring, titrate with standard $AgNO_3$ solution to the first change in color from canary yellow to salmon. Record the volume of titrant used. Repeat one more time and calculate the average volume of titrant used. $$CN\frac{mg}{L}=\frac{(N\,AgNO_3)(mL\,AgNO_3)}{mL\,CN\,1000\,mg/L\,stock}\cdot 52\cdot 1000$$ 52 = mg CN/meq for the reaction. $Ag^+ + 2\,CN^- = Ag(CN)^-_2$ 1000 = Conversion factor, mL/L.
Standardization of Silver Nitrate Solution	Pipet 25.0 mL 0.0141 N NaCl solution into a beaker. Add 1.0 mL K_2CrO_4 indicator solution and mix. While stirring, titrate with $AgNO_3$ solution to a pinkish yellow end point. Record the volume of titrant used. Repeat one more time and use the average volume of the titrant used. $$N\,AgNO_3=\frac{(Normality\,NaCl)(mL\,NaCl)}{mL\,AgNO_3}$$
Revision: 11	Status: Final	Method: ILM04.1, 9012A, 335.2, 335.3, 335.4	Minor Revision Date: 2/9/01

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Soil	Standard (SW-846)	14 from sampling	14 from sampling	Glass at 2-6 C
Water	Standard (SW-846)	14 from sampling	14 from sampling	Glass with NaOH To pH >10 at at 2-6 C
Soil/Water	NYSDEC and CLP	12 from VTSR	12 from VTSR	As above
Revision: 11	Status: Final	Method: ILM04.1, 9012A, 335.2, 335.3, 335.4		Minor Revision Date: 2/9/01

10.0 PROCEDURE

10.1 Prepare a curve each day of use by adding the following amounts of working standard with syringes or volumetric pipettes (working standard prepared after weekly determination of concentration of stock by standardization procedure) (See Table 8-3).

10.1 If samples were treated for sulfides, use the treated distilled curve prepared in GAC.106, Section 10.2.5

Table 10-1

STANDARDS

Name	Calibration Standard (mL)	Amount of Carrier (0.25N NaOH)	Final Concentration (mg/L)
1	10.0 mL of calibration working standard	100-mL volumetrically	1.0
2	5.0 mL of calibration working standard	100-mL volumetrically	0.50
3	2.5 mL of calibration working standard	100-mL volumetrically	0.25
4	1.0 mL of calibration working standard	100-mL volumetrically	0.10
5	0.5 mL of calibration working standard	100-mL volumetrically	0.05
6	0.1 mL of calibration working standard	100-mL volumetrically	0.01
7	Carrier (0.25N NaOH)	Carrier may be added directly to tube	0.00
Revision: 11	Status: Final	Method: ILM04.1, 9012A, 335.2, 335.3, 335.4	Minor Revision Date: 2/9/01

10.3 Instrument Start-up

10.3.1 Attachment of Manifold; Use the Following Features:

10.3.2 Interference filter: 570 nm.

10.3.3 150-mm sample loop.

10.3.4 Heating unit: 650 cm of tubing.

10.3.5 See attached manifold diagram for proper line connections.

10.3.6 See attached pyridine trap sheet for proper set up and use.

10.3.7 Pump speed: 35 (Normal).
10.3.8 Turn on power. Computer self test takes approximately 80 seconds. Allow heating unit to warm up to 60 C, approximately 15 minutes.
10.3.9 Place reagent lines into proper containers. Raise tension levers on pump tube cassettes one click.
10.3.10 Allow reagents to pump through the system to remove air bubbles.
10.3.11 Download "CN^-" method needed.
10.4 Analyze calibration standards high to low concentrations. The curve correlation coefficient must be greater than or equal to 0.995 in order to proceed with analysis. If it is not, take corrective action (e.g., change tubing or other maintenance, reprepare standards) and rerun the curve until correlation coefficient is $\geq$ 0.995. Must print "Runtime Report" to print curve information and calibration coefficient.
10.5 When analyzing for cyanide in a soil matrix, remember to enter the mass of sample (g) taken for distillation in "weights".
10.6 Results for diluted samples must be greater than 10x the reporting limit, but within the accepted linear range of the analysis. The results should be mid-range or above. Use carrier solution to make dilutions, not ASTM Type II water.
10.7 Any further manual dilution factor is entered in "Dilutions." Make note of dilutions in logbook.
10.8 **SW-846 Analytical Sequence After Calibration**. See Table 12-1.1 also.
10.8.1 ICV (Initial Calibration Verification, **undistilled** reference standard)
10.8.2 ICB
10.8.3 CCV (Continuing Calibration Verification, undistilled calibration standard)
10.8.4 CCB
10.8.5 MB
10.8.6 LCSL (distilled low **calibration** standard)
10.8.7 LCSH (distilled high **calibration** standard)
10.8.8 Seven samples or fewer (includes LCSW or LCSS, MS, MSD, and DUP if required)
10.8.9 CCV 2
10.8.10 CCB 2
10.8.11 Repeat 10 samples, CCV, CCB to closing.

10.9 **CLP Analytical Sequence After Calibration (see Table 12-1.2)**
10.9.1 LCS (This is a **distilled** reference standard).
10.9.2 ICB
10.9.3 CCV
10.9.4 CCB
10.9.5 MB
10.9.6 Nine samples or fewer (includes LCS solid if necessary, MS, DUP)
10.9.7 CCV2
10.9.8 CCB2
10.9.9 Repeat 10 samples, CCV, CCB to closing

Instrument Conditions and Maintenance
See Table 10-1, Routine Maintenance Procedures.

Table 10-2

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Date of Repair or Corrective Action
Lachat 8000 Auto analyzer	Stunted peaks or restricted flow.	Check lines for clogs and/or flat lines going through pump or contaminated reagents.
Auto analyzer	Checks lamps intensity and other vital functions.	Computer runs a self test each time system is turned on.

Revision: 11	Status: Final	Method: ILM04.1, 9012A, 335.2, 335.3, 335.4	Minor Revision Date: 2/9/01

10.14 Maintenance for Pyridine Vapor Trap

The Pyridine cyanide vapor trap used in conjunction with the Lachat 8000 program for cyanides to eliminate the noxious vapors associated with this analytical method.

Every 3 months the carbon, HCl vapor trap for pyridine must be checked and changed if necessary. Failing to follow this schedule could result in the release of foul smelling pyridine vapors into the lab.

Disposal of Spent Trap Material

1. Wearing gloves, remove vapor lines from trap. Wipe these lines with paper toweling. They will probably be covered with HCl, pyridine by products.
2. Place the trap container under a fume hood.
3. Empty the contents of the trap container into a wide-mouth amber jar.
4. Label this jar "Waste carbon, with Pyridine" securely seal the container and take to storage area for proper disposal in a "HP" lab pack.

Rebuilding Pyridine Vapor Trap

1. Reinstall vent line so that they rest on the bottom of the container.
2. Cover the bottom of the container (as uniformly possible) with 1"– 1 ½" of glass fiber.
3. Fill a 2-liter beaker with approximately 1.5 liter of activated carbon.
4. Soak the carbon with 1 N HCl for one-half hour.
5. Pour off the HCl (try to keep the loss of carbon to a minimum) so that you have a beaker of damp – NOT wet carbon. Air must be capable of getting through the carbon.
6. Fill the trap container with the damp carbon. Make sure the carbon is equally distributed across the glass fiber, totally covering it.
7. Seal the container and place it back in the pyridine waste vent system.

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 The instrument prints results in mg/L on runtime report. The following is provided as a check on the instrument printout.

Water samples:

$$\text{Sample concentration (mg/L)} = \frac{\text{mg}}{\text{L}}\text{curve} \cdot \frac{\text{V}}{\text{S}} \cdot \text{F/d}$$

where:

S = Sample volume (mL) distilled, generally 50 mL.
V = Volume of distillate collected, 50 mL.
F/d = Dilution factor.
d = Amount of distillate taken for dilution.
F = Final diluted volume of distillate diluted with carrier solution.

Soil samples:

$$\text{Sample concentration (mg/kg)} = \frac{\text{mg}}{\text{L}}\text{curve} \cdot \frac{\text{V}}{\text{S}} \cdot \text{df}$$

where:

S = Sample size (g), generally 1 g.
V = Volume of distillate collected, 50 mL.
df = Dilution factor, as above (F/d).

Target Compounds and Reporting Limits

See Table 11-1, Target Compounds and Quantitation Limits.

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL	
		Water (mg/L)	Soil (mg/kg)
Cyanide	T, S, L	0.010	0.5
Amenable cyanide	T, S, L	0.010	0.5

Revision: 11	Status: Final	Method: ILM04.1, 9012A, 335.2, 335.3, 335.4	Minor Revision Date: 2/9/01

Key Type:

T = Compound/Analyte is target compound routinely reported.
M = Compound/Analyte is listed in the method but is not routinely reported by E & E.
C = Compound/Analyte is specified by the client and can be analyzed under this method.
S = Compound/Analyte is routinely used as a matrix spike.
L = Compound/Analyte is routinely used as a LCS spike.
Q = Compound/Analyte is used as a surrogate spike.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

12.1 Use Table 12-1.1 when analyzing by SW-846 methodology and Table 12-1.2 when analyzing by CLP or Methods 335.2, 335.3, or 335.4.

Table 12-1.1

ROUTINE QUALITY CONTROL SAMPLES (SW-846: GAC.106)

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
ICV (undistilled)	At start of run	Dilute 5.0 mL of Reference spiking solution to 100 mL in a volumetric flask with carrier solution (0.25 N NaOH). Resulting nominal concentration 0.5 mg/L.	(SW-846) 85-115% recovery	Reanalyze. Check lines, and concentration of NaOH. Redistill the batch if corrective action fails to resolve the problem.
ICB	After ICV	Undistilled carrier (see Table 8-2)	| < 0.01 | mg/L	Reanalyze. Check lines, and concentration of NaOH. Redistill the batch if corrective action fails to resolve the problem.
CCV	Not distilled – after ICB, every 10 samples, and at end.	5.0 mL calibration working standard diluted to 100 mL with 0.25N NaOH (concentration 0.5 mg/L).	85-115%	Recalibrate and reanalyze samples since last successful CCV/CCB.
CCB (Continuing Calibration Blank)	After each CCV	Undistilled carrier (see Table 8-2)	| < 0.010 mg/L |	Recalibrate and reanalyze samples since last successful CCV/CCB.
Low LCS (LCSL) (SW-846 only)	1 per batch (distilled). See GAC.106	See preparative SOP (concentration 0.2 mg/L).	90-110 %	Investigate and correct the cause.

Table 12-1.1

ROUTINE QUALITY CONTROL SAMPLES (SW-846: GAC.106)

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
High LCS (LCSH) (SW-846 only)	1 per batch (distilled). See GAC.106	See preparative SOP (concentration 0.6 mg/L).	90-110 %	Investigate and correct the cause.
LCSW (Water LCS)	One per 20 or fewer samples	See preparative SOP (Nominal concentration 0.4 mg/L).	Laboratory limits	Reprepare batch
LCSS (Solid LCS)	One per 20 or fewer samples	See preparative SOP (Nominal concentration 20 mg/kg).	Laboratory limits	Reprepare batch
MB (Method Blank)	1 per batch of 20 samples or fewer. See GAC.106 (MIDI)	See preparative SOP	$\|< 0.01\|$ mg/L for waters $\|< 0.50\|$ mg/kg for soil	Recalibrate and reanalyze
MS/MSD	One pair per 20 or fewer samples. See GAC.106 (MIDI)	100 µg/L or 5 mg/kg nominal. See preparative SOP.	- Project requirements if specified. - Compare against LCS limits for bias.	Project requirements if any, otherwise evaluate results in conjunction with other QC information (e.g., LCS recoveries) to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
DUP	If required, client specified. See GAC.106 (MIDI)	See preparative SOP	- Project requirements, if specified - 20% for waters - 35% for soils If one or both sample results are <5 x PQL, then duplicate results are acceptable if the difference between the two results is < 2 x PQL.	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.

Revision: 11	Status: Final	Method: ILM04.1, 9012A, 335.2, 335.3, 335.4	Minor Revision Date: 2/9/01

Table 12-1.2

ROUTINE QUALITY CONTROL SAMPLES (CLP, GAC.118, GAC.119)

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
ICV/LCSW	For both water and soil batches. One per 20 or fewer samples. See GAC.118, GAC.119 (CLP).	See GAC.118, GAC.119 (nominal concentration 0.4 mg/L).	~~85-115%~~ 85-120%	Reprepare batch
ICB	After ICV	Undistilled carrier (see Table 8-2)	|< 0.01| mg/L	Reanalyze check lines and concentration of NaOH.
CCV	Not distilled – after ICB, every 10 samples, and at end.	5.0 mL calibration working standard diluted to 100 mL with 0.25N NaOH (concentration 0.5 mg/L).	~~85-115%~~ 85-120%	Recalibrate and reanalyze
CCB	After each CCV	Undistilled carrier (see Table 8-2)	|< 0.010 mg/L|	Recalibrate and reanalyze
MB or PB (Method or Preparation Blank)	1 per batch of 20 samples or fewer	See appropriate preparative SOP	|< 0.01| mg/L for waters |< 0.50 mg/kg| for soil	Recalibrate and reanalyze
LCS (solid)	When analyzing soils only. See GAC.119 (CLP).	See GAC.119	CLP: Manufacturer's criteria.	Reprepare batch
MS	One per SDG (see GAC.119 or GAC.118)	See preparative SOP (concentration ~100 µg/L water; ~5 mg/kg soil)	75-125%	For CLP: Perform post digestion spike at two times original result or at 0.020 mg/L, whichever is greater.
DUP	One per SDG (see GAC.118 (CLP) or GAC.119 (CLP).	See appropriate preparative SOP	± 20% RPD	Flag as appropriate

Revision: 11	Status: Final	Method: ILM04.1, 9012A, 335.2, 335.3, 335.4	Minor Revision Date: 2/9/01

Table 12-2

CONTROL CHARTED QUALITY CONTROL SAMPLES

QC Type		Analytes Charted	Matrix Charted
LCSW		Cyanide	Water
LCSS		Cyanide	Soil
Revision: 11	Status: Final	Method: ILM04.1, 9012A, 335.2, 335.3, 335.4	Minor Revision Date: 2/9/01

Table 12-3.1

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES
(SW-846: GAC.106)

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
ICV (undistilled)	Water	Cyanide	0.5 mg/L	85-115	NA	Method
Low LCS	Water	Cyanide	0.20 mg/L	90-110	NA	Method
LCSW	Water	Cyanide	0.40 mg/L	See Table 12-1.1	NA	Laboratory
High LCS	Water	Cyanide	0.60 mg/L	90-110	NA	Method
MS	Water	Cyanide	0.1 mg/L	See Table 12-2	NA	Laboratory
DUP	Water	Cyanide	NA	NA	20	Laboratory
LCSS	Soil	Cyanide	20 mg/kg	See Table 12-1.1	NA	Laboratory
MS	Soil	Cyanide	5 mg/kg	See Table12-2	NA	Laboratory
DUP	Soil	Cyanide	NA	NA	35	Laboratory
CCV	Water	Cyanide	0.5 mg/L	85-115	NA	Laboratory
Revision: 11	Status: Final	Method: ILM04.1, 9012A, 335.2, 335.3, 335.4			Minor Revision Date: 2/9/01	

Table 12-3.3

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES
(CLP Water and Soil)

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
ICV/LCS (undistilled)	Water	Cyanide	0.4 mg/L	~~85-115%~~ 85-120%	NA	Method
MS	Water	Cyanide	0.1 mg/L	75 – 115	NA	Method
DUP	Water	Cyanide	NA	NA	20	Method
LCSS	Soil	Cyanide	Manufacturer	Manufacturer	NA	Manufacturer
MS	Soil	Cyanide	5 mg/kg	75 – 125	NA	Method

Table 12-3.3

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES (CLP Water and Soil)

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
DUP	Soil	Cyanide	NA	NA	20	Method
CCV	Water	Cyanide	0.5 mg/L	85 – 115	NA	Laboratory
Revision: 11	Status: Final	Method: ILM04.1, 9012A, 335.2, 335.3, 335.4			Minor Revision Date: 2/9/01	

13.0 SPECIAL PROJECT REQUIREMENTS

Client-Specific Quality Control Requirements

13.1 U.S. Army Corps of Engineers (ACE)

13.1.1 Method Quantitation Limit (MQL) is between 3-10 times MDL. The low calibration standard concentration must be ≥3 times MDL. The Method Reporting Limit (MRL) may slide between two times the MDL and project-specific or regulatory requirements. Results below the MRL must be reported "ND." Results between the MQL and MRL must be reported as estimated (J flagged).

13.2 AFCEE General Requirements

13.2.1 Batching

13.2.2 An AFCEE analytical batch is the environmental samples that are similar in composition (matrix) and are prepared at the same time and with the same lot of reagents.

13.2.3 A batch is not to exceed 20 samples. The sample duplicate, MS, MSD are considered an environmental sample, therefore, the actual number of unique samples is less than 20.

13.2.4 Laboratory Control Sample

13.2.4.1 Laboratory Control Samples for solid must be prepared using Ottawa sand unless a variance was obtained.

13.2.4.2 The LCS must be prepared from second source material.

13.2.4.3 The LCS must be spiked at a concentration ≤ the midpoint of the calibration curve for each analyte unless a variance was obtained.

13.2.5 Matrix Spike/Matrix Spike Duplicate.

13.2.5.1 The MS and MSD must be spiked at a concentration ≤ the midpoint of the calibration curve for each analyte unless a variance was obtained.

13.2.5.2 AFCEE samples must be used for spiking.

13.2.5.3 The AFCEE term second source calibration verification is the laboratory term initial calibration verification ICV), run once after a curve is analyzed. Do not confuse with the AFCEE term ICV (see below).

13.2.5.4 The AFCEE term of initial calibration verification is the same as the laboratory term daily calibration check. It may not be prepared from the same material used to prepare the LCS. Use second source material for the LCS and use calibration material for the AFCEE term ICV and continuing calibration verifications.

13.3 North Carolina DENR

13.3.1 Perform one duplicate every **10** samples (or an MS/MSD and duplicate every 20 samples).

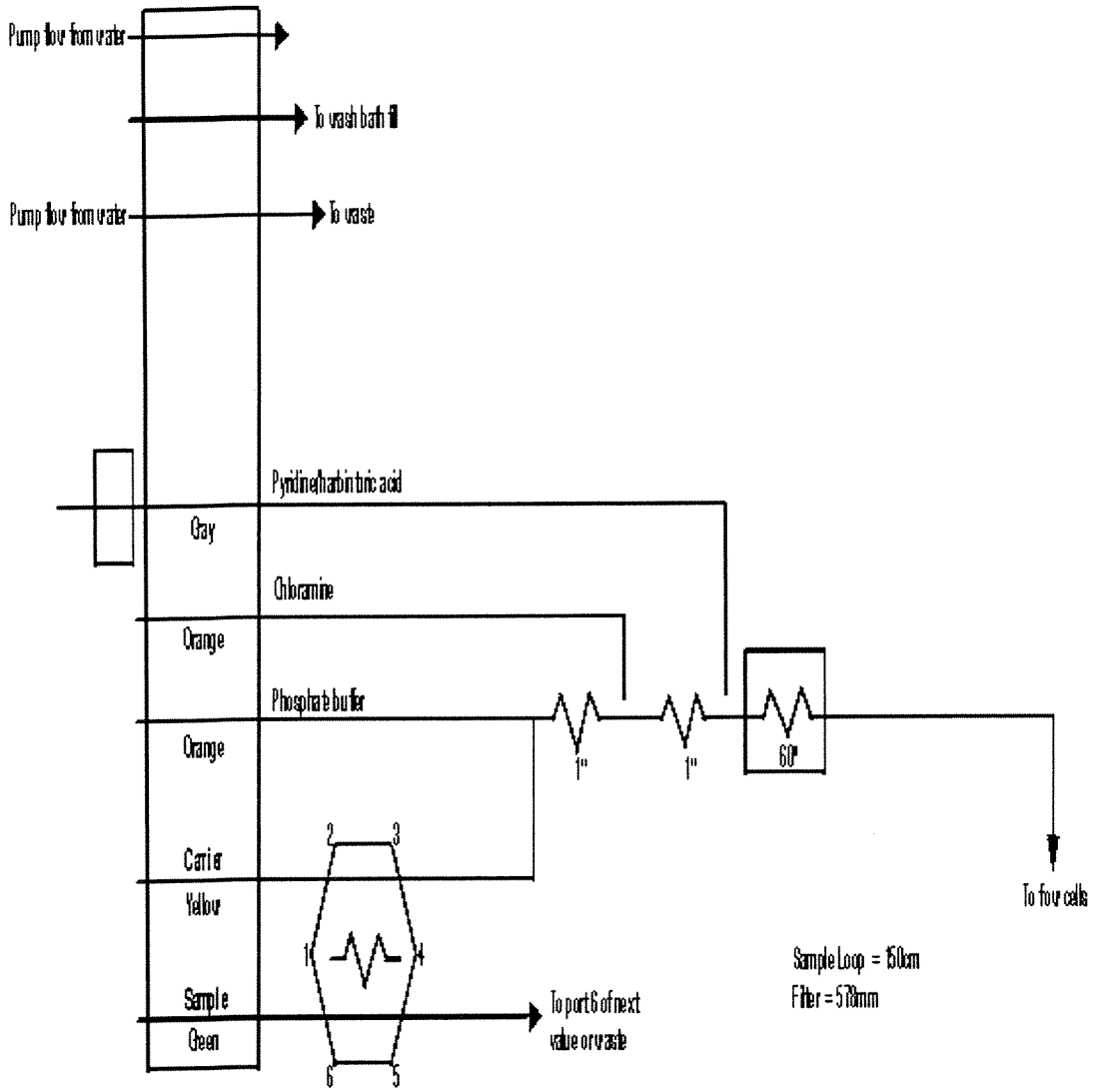
14.0 SAMPLE AND WASTE DISPOSAL

14.1 Sample waste is poured down the lab sink drain with copious amounts of tap water. If known to contain cyanide >250 mg/L, treat with sodium hypochlorite first. Waste colored sample flows into a satellite waste container and when full is dumped down a drain in a hood. For more details see the waste disposal SOP A10.

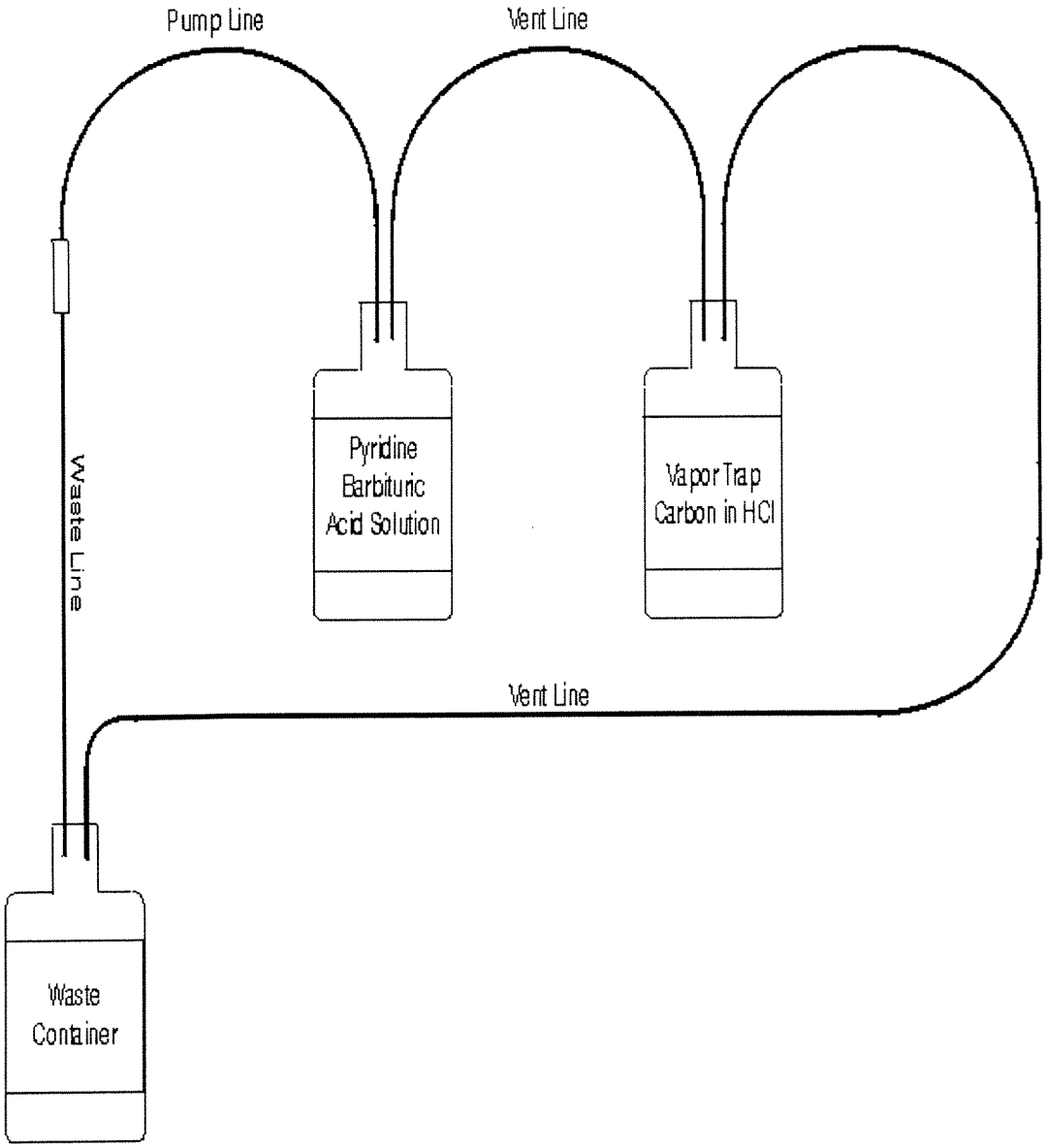
15.0 EXAMPLE FORMS

NA

MANIFOLD DIAGRAM



5839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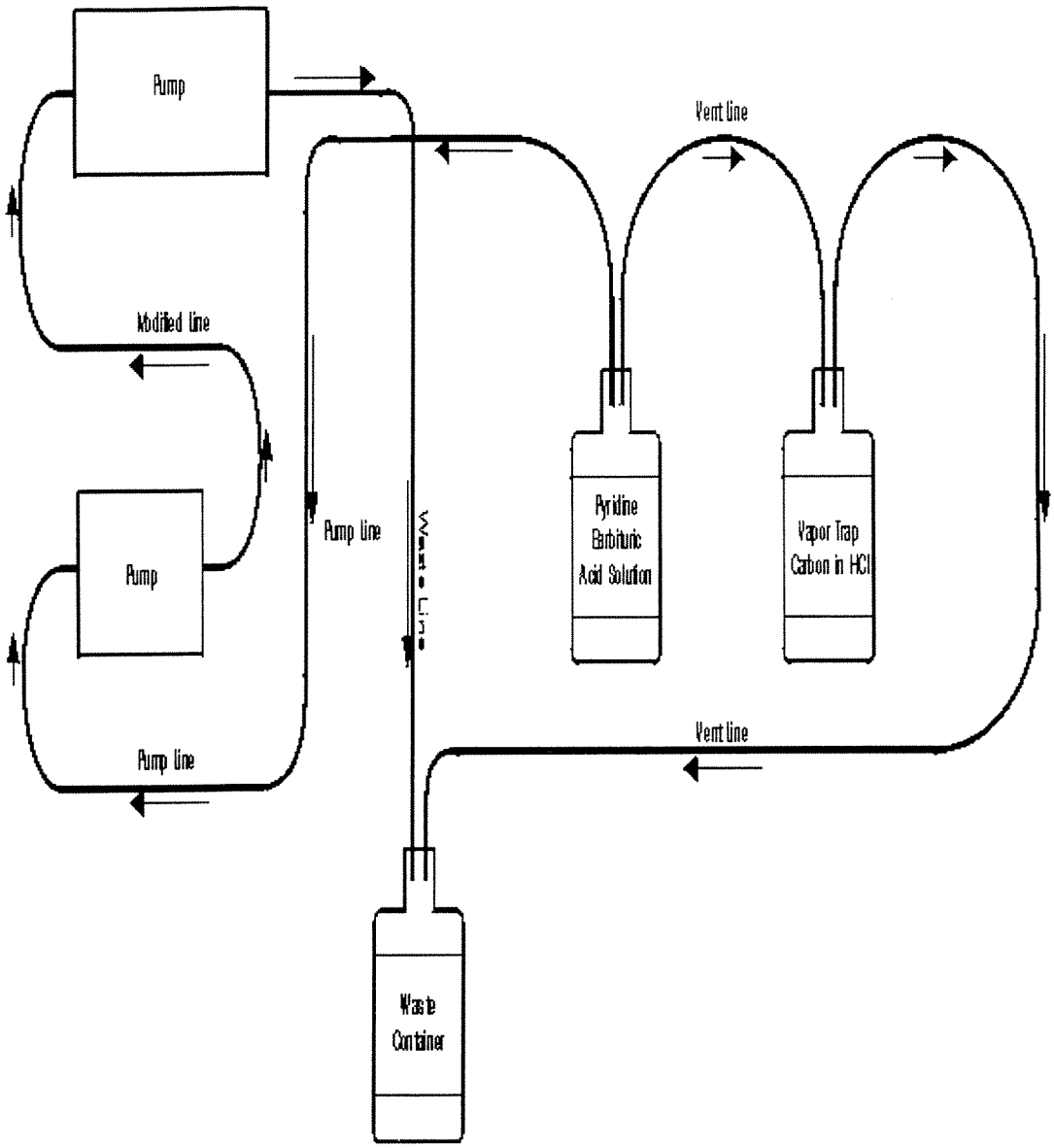
Vent Line
Waste Line
Pyridine
Barbituric
Acid Solution
Vapor Trap
Carbon in HCl
Vent Line
Waste
Container


Pump
Vent line
Modified line
Pump
Pump line
Waste Line
Pyridine
Barbituric
Acid Solution
Vapor Trap
Carbon in HCl
Vent line
Pump line
Waste
Container

END OF SOP

TITLE: Total Suspended Solids, Nonfilterable Residue			
Reviewer: C. Okal		Peer Reviewer: None	
Implementation Date: 5/5/98		Last Annual Review Date: 6/4/01	
Revision: 2	Status: Final	Method: 160.2	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 To determine the quality of suspended, nonfilterable solids in drinking, surface and saline waters, domestic and industrial wastes. The practical range of the determination is approximately 4 mg/L to 20,000 mg/L.

2.0 METHOD SUMMARY

2.1 A well mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to a constant weight at 103-105 C.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 EPA Method 160.2.
4.2 Standard Methods, Method 2540D, 18th Edition, 1992.
4.3 Training SOP QA.5.
4.4 Data Review SOP A.25.
4.5 Laboratory Quality Assurance Manual (L-001).
4.6 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 **TSS** = Non-filterable residue. Those solids retained by a glass fiber filter and dried to constant weight at 103 - 105°C. The determination is commonly referred to as total suspended solids (TSS).

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Particles such as leaves, sticks, fish and lumps of fecal matter should be excluded from the sample if it is determined their inclusion is not desired in the final result (goal is uniform representative sample).

6.2 Filtration apparatus, filter material, pre-washing, post-washing and drying temperature are specified because these variables have been shown to affect results.

6.3 Samples high in Filterable Residue (dissolved solids), such as saline waters, brines, and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter minimizes this potential interference.

7.0 INSTRUMENTATION AND EQUIPMENT

Filtering apparatus
Glass fiber filters, Gelman type A/E or equivalent (47 mm diameter)
Aluminum dishes
Graduated cylinders
Drying oven, 103-105° C
Desiccator
Analytical balance capable of weighing to 0.1 mg.
Foreceps

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard, Reagents, and Consumables

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
ASTM Type II water	In-house	Neat

Revision: 2	Status: Final	Method: 160.2	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Analysis (Days)	Container Type and Preservative
Water	Standard	7	Plastic or glass, 2-6 C
Water	NYSDEC	5	Plastic or glass, 2-6 C

Revision: 2	Status: Final	Method: 160.2	Minor Revision Date: None

10.0 PROCEDURE

10.1 Set up the vacuum filtering apparatus.

10.2 Place a glass fiber filter on the membrane filter apparatus with wrinkled side up. Wash with three successive 20-L volumes of ASTM Type II water. Remove the filter with forceps and place on a labeled aluminum weighing dish. Dry at 103-105 C for at least one hour. Remove and place in a desiccator. Cool and record weight immediately before use. Record weights in a lab notebook.

10.3 Place the weighed filter on the membrane filter apparatus. Wet the filter with a small portion of ASTM Type II water. Always use forceps when handling glass fiber filters.

10.4 Vigorously shake the sample. Using a graduated cylinder, quantitatively transfer 100 mL or an aliquot of sample that will provide at least 1 mg of residue onto filtering apparatus. Continue to apply vacuum after sample has passed through.

10.5 Wash the graduated cylinder, funnel wall, and filter with three portions of ASTM Type II water. Allow complete drainage between washings. Use approximately 30 mL total volume of wash water.

10.6 Carefully move the filter back to the proper aluminum dish and place in a 103-105 C oven for at least one hour. Record in and out times and temperatures. Cool in a desiccator and weigh.

10.7 Repeat the drying cycle (drying, cooling, desiccating and weighing) until the change in weight is less than 0.5 mg or 4% of the previous weight. Record all weights in lab notebook.

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Drying Oven	Not keeping temperature – not working.	Notify Supervisor/ call repairman	Check and record temperature with each use.
Analytical balance	Not calibrating	Repair balance	As needed.

Revision: 3	Status: Final	Method: 160.1	Minor Revision Date: None

11.0 DATA REDUCTION/EVALUATION/REPORTING

Calculations:

$$\frac{mg}{L}TSS = \frac{F-I}{V} \cdot \frac{1000\,mg}{1g} \cdot 1000\frac{mL}{L}$$

where:

F = Final filter dish weight plus residue, grams.
I = Initial filter dish weight, grams.
V = Sample volume, mL.

$$RPD = \frac{|S - D|}{1/2(S + D)} \cdot 100$$

where:

S = Sample result.
D = Duplicate sample result.

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL Water (Units)
TSS, Non-Filterable Residue	T	4 mg/L for 100 mL of sample

Revision: 2	Status: Final	Method: 160.2	Minor Revision Date: None

Key Type:

T = Compound/analyte is target compound routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike.
L = Compound/analyte is routinely used as a LCS spike.
Q = Compound/analyte is used as a surrogate spike.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Blank	1 per batch of 20 or fewer samples	Aliquot of ASTM Type II water	< 4 mg/L for 100 mL sample	Dry again, reweigh
Duplicate	1 per 10 or fewer samples in each batch	2nd aliquot of sample	- Project requirements, if specified - 20% for waters If one or both sample results are <5 x PQL, then duplicate results are acceptable if the difference between the two results is < 2 x PQL.	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.

Revision: 2	Status: Final	Method: 160.2	Minor Revision Date: None

Table 12-2

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
DUP	Water	TSS	NA	NA	20	Laboratory

Revision: 2	Status: Final	Method: 160.2	Minor Revision Date: None

13.0 SPECIAL PROJECT REQUIREMENTS

NA

14.0 SAMPLE DISPOSAL

14.1 Filtrate may be poured down lab drain with copious amounts of tap water. After analysis is complete, the filter may be placed in nonhazardous solid waste receptical.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Total Dissolved Solids (TDS), Filterable Residue			
Reviewer: C. Okal		Peer Reviewer: None	
Implementation Date: 4/13/98		Last Annual Review Date: 6/4/01	
Revision: 3	Status: Final	Method: 160.1	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 Determination of soluble, filterable residue of water samples. The practical range of the method is approximately 8,000 mg/L.

2.0 METHOD SUMMARY

2.1 Filterable residue. Those solids capable of passing through a glass fiber filter and dried to constant weight at 180 C. The determination is commonly referred to as total dissolved solids (TDS).

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 EPA Method 160.1.
4.2 Standard Methods, Method 2540 C, 18th edition, 1992.
4.3 Training SOP QA.5.
4.4 Data Review SOP A.25.
4.5 Laboratory Quality Assurance Manual (L-001).
4.6 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 See the generic list in the Quality Assurance Manual.

5.2 **TDS = Total Dissolved Solids = Filterable Residue** = The portion of sample that passes through the filter and is dried at 180 ± 2 C.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

No interferences observed.

7.0 INSTRUMENTATION AND EQUIPMENT

Gelman type A/E glass fiber 47 mm or equivalent filter disks
Filtering apparatus
Evaporation dishes, 175-mL Pyrex or equivalent
Drying oven ($180° \pm 2°$ C)
Analytical balance to $\pm$ 0.1 mg
Dessicator
Graduated cylinders
Tongs

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standard, Reagents, and Consumables

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
ASTM Type II water	In-house	Neat

Revision: 3	Status: Final	Method: 160.1	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Matrix	Client/Project	Analysis (Days)	Container Type and Preservative
Water	Standard	7	Plastic or Glass, 2-6 C
Water	NYSDEC (1)	5	Plastic or Glass, 2-6 C

Revision: 3	Status: Final	Method: 160.1	Minor Revision Date: None

10.0 PROCEDURE

10.1 Label dishes. Heat clean evaporating dishes at 180 C $\pm$ 2 C for at least one hour, cool in dessicator.

10.2 Weigh each dish with analytical balance to 0.1 mg, record weight in logbook in grams. Note: check to see if analytical balance has been calibrated and recorded before use.

10.3 Pre-rinse filter disks, use wrinkled side up, with three 20 mL portions of ASTM type II water and discard filtrate.

10.4 Vigorously shake sample. Using a graduated cylinder, quantitatively transfer 100 mL of sample to the filtering apparatus. Less sample may be used if unable to pass 100 mL through the filter but do not use less than 25 mL or a volume that will yield less than 1 mg. Limit total residue to about 200 mg to avoid entrapment of water during drying. If filterable residue is low, a larger volume may be used.

10.5 Rinse each sample with three approximately 10 mL portions of ASTM Type II water. Transfer the filtrate into the corresponding evaporation dish. Record the sample volume used and dish number for each sample.

10.6 Place the dish in a 180 C ± 2 C oven to evaporate. Once completed evaporated, leave the dish in at 180 C ± 2 C for at least 1 hour. Record in and out times and temperatures of oven.

10.7 Cool dishes in dessicator and weigh. Record weight in grams. NOTE: Handle dishes only with tongs or gloved hands.

10.8 Repeat drying cycle (drying, cooling, dessicating, weight) until the change in weight is <0.5 mg or less than 4% of the previous weight.

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Drying Oven	Not keeping temperature – not working.	Notify Supervisor/ call repairman	Check and record temperature with each use.
Analytical balance	Not calibrating	Repair balance	As needed.

Revision: 3	Status: Final	Method: 160.1	Minor Revision Date: None

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 Calculations

$$\text{mg/L TDS} = \frac{(\text{Final dish weight plus residue, g}) - (\text{Initial dish weight, g})}{\text{Sample volume, mL}} \cdot (1000\,\text{mL/L}) \cdot (1000\,\text{mg/g})$$

$$\text{RPD} = \left[\frac{S - D}{1/2(S + D)} \cdot 100 \right]$$

where:

D = Duplicate sample result.
S = Sample result.

Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL Water (Units)
TDS (Total Dissolved Solids)	T	10 mg/L for 100 mL of sample

Revision: 3	Status: Final	Method: 160.1	Minor Revision Date: None

Key Type:

T = Compound/analyte is target compound routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike.
L = Compound/analyte is routinely used as a LCS spike.
Q = Compound/analyte is used as s surrogate spike.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Routine Quality Control Samples

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Blank	1 per batch of 20 or fewer samples	ASTM Type II water	<10 mg/L for 100 mL of sample	Dry again, reweigh
Duplicate	1 per batch or 10%	Second aliquot of sample	Project requirements, if specified - 20% for waters If one or both sample results are <5 x PQL, then duplicate results are acceptable if the difference between the two results is < 2 x PQL.	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.

Revision: 3	Status: Final	Method: 160.1	Minor Revision Date: None

TITLE: Total Organic Carbon, Method 5310B			
Reviewer: D. Ruszczyk		Peer Reviewer: F. Lindauer	
Implementation Date: 6/13/01		Last Annual Review Date: 6/4/01	
Revision: 6	Status: Final	Method: 5310B	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedure to determine the concentration of organic carbon in groundwater, surface and saline waters, domestic and industrial wastes. It also may be used to report inorganic carbon.

1.2 This method is not appropriate for waters with heavy particulates if the size of the particulates remains greater than the sampling needle aperture after blending. Filtering can be done. Results are reported as Dissolved Organic Carbon. The Project Manager must be advised.

1.3 The Method 5310B analytical procedure does not differ in an analytical sense from Method 9060. The stricter QC between the methods is used and this procedure may be used for Method 9060 or Method 5310B.

1.4 Cite only Method 5310B when analyzing drinking water, NPDES, or SPDES samples.

2.0 METHOD SUMMARY

2.1 The sample is homogenized and diluted as necessary and a micro portion is injected into a heated combustion (reaction) chamber packed with cobalt oxide. The water is vaporized and the organic carbon is oxidized to CO_2 and H_2O. The CO_2 from oxidation of organic carbon is transported in the carrier gas stream and is measured by means of a nondispersive infrared analyzer. The amount of CO_2 in the sample is directly proportional to the carbonaceous material in the sample.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 Technician should be aware and not look directly into light source while instrument is operating.

3.3 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 SW-846, Method 9060, Rev 0, September 1986
4.2 Shimadzu 5050A, Manual M393-E120
4.3 Standard Methods 5310B, 18th ed., 1992
4.4 Use checklist C-164 for analyst and peer review.
4.5 Training SOP QA.5, A.23, and A.29.
4.6 Data Review SOP A.25.
4.7 Laboratory Quality Assurance Manual (L-001).
4.8 MDL SOP A.18.
4.9 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 **TOC = Total Organic Carbon** = all carbon atoms covalently bonded in organic molecules.

5.2 **TIC = Total Inorganic Carbon** = Carbonate, bicarbonate and dissolved CO_2.

5.3 **POC (VOC) = Purgeable Organic Carbon** = the fraction of the TOC removed from an aqueous solution by gas stripping under specified conditions.

5.4 **NPOC = Non-Purgeable Organic Carbon** = the TOC fraction not removed by gas stripping.

5.5 See more definitions in Method 5310A introduction.

5.6 Total carbon is total inorganic carbon plus total organic carbon in the sample.

TC = TIC + TOC or

TC = TIC + POC + NPOC

5.7 **Total organic carbon** is itself comprised of purgeable organic carbon plus non-purgeable organic carbon. TOC = POC + NPOC

5.8 Generally, clients are interested in TOC. Since POC is generally negligible with respect to NPOC, in practice the NPOC determination is substituted for TOC.

5.9 **Dissolved organic carbon (DOC)** is defined as the TOC result after passing the sample through a 0.45-µm pore diameter filter.

5.10 **ASC Control Chart Database** - Access ™ database used for control charting by ASC. Control limits are tabulated by lab, method, and by matrix is the "Control Limit Summary".

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Inorganic carbon must be separated from the total organic carbon. Carbonate and bicarbonate removal by acidification and sparging with inert gas can result in loss of volatile organic substances.

6.2 Large particulate can clog instrument tubes. Samples containing particulates should be homogenized in a blender. If the sample still has large particulates, filter (0.45-µm pore diameter filter), report to the Laboratory Project Manager, and note in logbook. Report as Dissolved Organic Carbon.

7.0 INSTRUMENTATION AND EQUIPMENT

ASI 5000A Autosampler
TOC Control Software Version 4.0
TOC Manual for Operation and Maintenance Procedures
5050A Shimadzu Total Organic Carbon Analyzer
100-mL Volumetric flasks
Class A pipettes
Air supply for sparging (UHP grade)

8.0 REAGENTS AND MATERIALS

8.1 All reagents are reagent grade unless stated differently.

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Anhydrous potassium hydrogen Phthalate (KHP)	Fisher or equivalent. Two lots are required.	Neat
Phosphoric acid	Fisher or equivalent	Neat
ASTM Type II water	In-house system	Neat
Sodium bicarbonate	Fisher or equivalent	Neat
Sodium carbonate	Fisher or equivalent	Neat
Ultra Pure Air, compressed	Rodgers Welding or equivalent	< 0.1 ppm of CO/CO_2 < 0.1 ppm of total hydrocarbon
Sodium hydroxide (NaOH)	Fisher or equivalent	Neat
Oxygen, carrier gas	Rodgers Welding or equivalent	NA

Revision: 6	Status: Final	Method: 5310B	Minor Revision Date: None

8.2 Standard Solution Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration (mg/L)
KHP, Anhydrous 2 Lots	KHP	Dry KHP for 1 hour at 105 ± 5 C before use.	Neat
Calibration Stock Standard	Anhydrous KHP	Dissolve 2.125 g KHP to 1 L with ASTM Type II water. Good for 1 month.	1000 mg/L organic carbon
Reference Stock Standard	Anhydrous KHP	Dissolve 2.125 g KHP to 1 L in a volumetric flask, dilute with ASTM Type II water. Good for 1 month. Different lot number than calibration standard (2nd source).	1000 mg/L organic carbon
LCS (ICV)	Reference Stock Standard	Add 2.0 mL of Reference Stock Standard and dilute to 100 mL with ASTM Type II water. Make fresh daily.	10 mg/L organic carbon
Carbonate/Bicarbonate Stock Solution (used for TIC if requested)	Sodium Carbonate, anhydrous; sodium bicarbonate, anhydrous	Stock Solution: 0.3500 g sodium bicarbonate and 0.4412 g sodium carbonate in a 100-mL volumetric flask with ASTM Type II water. Good 6 months.	1000 mg/L inorganic carbon

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration (mg/L)
Carbonate/Bicarbonate Standard Solution	Carbon-ate/Bicarbonate Stock Solution	Add 5.0 mL of Carbonate/ Bicarbonate Stock Solution to 100 mL volumetric to volume with ASTM Type II water. Make fresh daily.	50 mg/L inorganic carbon
25% Phosphoric acid	Phosphoric acid	294.0 mL of 85% H_3PO_4 and dilute to 1000 mL with ASTM Type II water. Good for 6 months.	25% H_3PO_4
50 mg/L Calibration standard	Calibration Stock Standard	5 mL of Calibration Stock Standard and dilute to 100 mL with ASTM Type II water. Make fresh daily.	50 mg/L Organic carbon
30 mg/L Calibration standard; CCV	Calibration Stock Standard	3 mL of Calibration Stock Standard and dilute to 100 mL with ASTM Type II water. Make fresh daily.	30 mg/L Organic carbon
10 mg/L calibration standard	Calibration Stock Standard	1.0 mL Calibration Stock Standard and dilute to 100 mL with ASTM Type II water. Make fresh daily.	10 mg/L Organic carbon

Revision: 6	Status: Final	Method: 5310B	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Analysis (Days)	Container Type and Preservative
Water	Standard	28 Days	125 mL poly, cool, 2-6 C H_2SO_4 to pH of < 2.

Revision: 6	Status: Final	Method: 5310B	Minor Revision Date: None

10.0 PROCEDURE

10.1 Start Up/Maintenance – PC Control

10.1.1 Turn on air tank, analyzer, and set carrier gas to 150 mL/min.

10.1.2 Check the copper halogen scrubber inside door of analyzer. When ¾ of the length of the scrubber is black, replace the scrubber.

10.1.3 Double click "TOC Control" icon.

10.1.4 Click "Options" menu.

10.1.5 Choose Instrument Conditions

10.1.6 Click "TOC" tab:

- Furnace on
- Auto ranging and injection volume
- Auto regeneration of IC solution

10.1.7 Click ASI tab:
- ASI used "2" (number of needle washes); "2" (number of flowline washes)
- Rinse

10.1.8 Click "OK" at bottom of window.

10.1.9 The analyzer will go to ready state when furnace reaches temperature (it takes approximately 30 minutes). Check that the baseline is at 1x.

10.1.10 Check humidifier level. It should be between two white lines. Add one sodium hydroxide pellet every 3 months to the deionized water in the humidifier.

10.1.10.1 Check the 25% H_3PO_4 level. It should be at 125 mL.

10.1.10.2 Check for bubbles in the IC reactor.

10.1.10.3 Check the drain pot level. It should always be full.

10.1.10.4 Check all lines to see if any are disconnected.

10.1.10.5 Check bottle tops and make sure all are tightened.

10.1.10.6 Perform a gross leak check. Pinch both tubes connected to CO_2 absorber lines, watch for bubbles in drain pot. This indicates there are no leaks in system.

10.1.10.7 Check for bubbles in the syringe.

10.1.10.8 Zero point the syringe.

10.1.10.9 Run 10 injections of acidified ASTM Type II water (pH ~3) at beginning of each use of the TOC 5050A.

10.2 Connecting the Instrument

10.2.1 Click on "Measure" menu.

10.2.2 Select "Connect."

10.2.3 Click on "Options/Maintenance" sub-menu.

10.2.4 Select "Zero Point Detection."

10.2.5 Insert a blank into the injection tube.

10.2.6 Click "OK."

10.3 Creating the Sample Table

10.3.1 Click "Options" menu.

10.3.2 Select "General Information."

10.3.3 Enter the following information:

10.3.3.1 User: Analyst's name.

10.3.3.2 Title: Batch number.

10.3.3.3 Comment: Client names and work order numbers.

10.3.4 Click "OK."

10.3.5 Select "Measurement Parameters" from Options menu.

10.3.6 Enter the following: WTOC.

10.3.6.1 Unit: mg/L.

10.3.6.2 No. of injections: 4 (for 5310B Method) 4 (for 9060 Method).

10.3.6.3 Max. no of inj.: 5 (for 5310B Method) 5 (for 9060 Method).

10.3.6.4 No. of Washes: 4.

10.3.6.5 Sparge time: 3 (time can be increased due to sample matrix).

10.3.6.6 Max SD: 200.

10.3.6.7 Max CV: 2.0.
10.3.6.8 Click "OK."
10.3.7 Leave default parameters.
10.3.8 In the Options menu, choose "Display Settings."
10.3.9 For each table, (Sample, Injection, and Statistics), click "Select All."
10.3.10 Click on "File" menu.
10.3.11 Select "Save As."
10.3.12 Enter "Tutorial.toc."
10.3.13 Once all these preliminary steps are set in the future open "Tutorial.toc," enter new sample table information, and save the file under a new name (the batch number).

10.4 Setting Up the Standard Runs
10.4.1 Click in the "first line" of the sample table.
10.4.2 From the Edit menu, choose "Insert Standard."
10.4.3 Choose "New" from the Calibration Curve List dialog box.
10.4.4 Enter "Title" and "File Name" (i.e., Title: Curve 1 wcal_1....File Name: wcal_1.cal).
10.4.5 Change the Calculation Method to "Lin. Regression without Zero Shift."
10.4.6 Verify range is at 1x for WTOC. Change if necessary. Do not change injection volume.
10.4.7 Click "Data" tab.
10.4.8 Enter the standard run information.
10.4.9 Click "OK."
10.4.10 Click on "Conditions" tab.
10.4.11 Verify selections.
10.4.12 Click "OK."
10.4.13 Choose "Yes" to save calibration information.
10.4.14 Choose "OK."
10.4.15 Save the sample table created so far: Click "File" menu, then select "Save."

10. 5 Setting Up the Unknown Run
10.5.1 Click on fifth line of the sample table.
10.5.2 Click "Edit" menu.
10.5.3 Select "Insert Sample."
10.5.4 The "Insert Sample" opens the "Method" dialog box.
10.5.5 Select "New" to create a new method.
10.5.6 Enter the following information:
10.5.6.1 File Name: 5310B Method or 9060 Method.
10.5.6.2 Comment: "WTOC."
10.5.6.3 Sample Name: "<Untitled>."
10.5.6.4 Sample ID: "<Untitled>."
10.5.6.5 Analysis: "NPOC."
10.5.6.6 Dilution: "1.0."
10.5.6.7 Type "Unknown."
10.5.7 Click "OK."
10.5.8 Select the "NPOC" tab.
10.5.9 With the Cursor in the "Cal Curves 1 field."
10.5.10 Select "Browse."
10.5.11 Choose curve you want to use.

10.5.12 Select "OK."
10.5.13 Click "Yes" to use the calibration file to fill out the method.
10.5.14 Click "OK" when all is competed correctly on NPOC tab.
10.5.15 Select "OK" to close the Method File list dialog box.
10.5.16 Save the completed sample table - click the "Save" button on the toolbar.

10.6 Making the Analyses
10.6.1 Prepare calibration curve standards (see Table 8-2).
10.6.2 Verify status of instrument before beginning analyses.
10.6.3 Click "View" menu.
10.6.4 Choose "Background Monitor."
10.6.5 The status of the baseline should be OK for each parameter at the 1x Range for WTOC.
10.6.6 Close the "Background Monitor" window after verifying everything is OK.
10.6.7 Click "Measure" menu.
10.6.8 Choose "Start" or click on "Start" button of instrument control bar.
10.6.9 The analyses will begin.
10.6.10 Click the "View" menu.
10.6.11 Open the "Realtime Window" to monitor the status of the measurements.

10.7 Evaluating the Results of the Analyses
10.7.1 Click "View" menu.
10.7.2 Select "Calibration Curve."
10.7.3 Select the "Curve" tab to see the curve data in graph form.
10.7.4 Select the "Data" tab to see curve statistics.
10.7.5 To see peak shape of all injections in a sample table:
10.7.5.1 Select row.
10.7.5.2 Click "View" menu.
10.7.5.3 Select "Peak Profile."
10.7.5.4 Select "Display."
10.7.5.5 Or select the "Peak Profile" button in the toolbar.
10.7.6 Click "View" menu.
10.7.7 Select the "Statistics (Summary)" table.

10.8 Printing a Report
10.8.1 Click "File" menu.
10.8.2 Select "Page Setup."
10.8.3 Make the following selections on the "General" tab:
10.8.3.1 General Information.
10.8.3.2 Organization.
10.8.3.3 User.
10.8.3.4 Title.
10.8.3.5 Instrument ID.
10.8.3.6 Filename.
10.8.3.7 Comment.
10.8.3.8 Instrument Conditions.
10.8.3.9 Instrument Attachments.
10.8.4 Make the following selections on the "Cal Curves" tab:
10.8.4.1 Print cal curves.

10.8.4.2 Filename.
10.8.4.3 Title.
10.8.4.4 Calculation method.
10.8.4.5 Unit.
10.8.4.6 Range.
10.8.4.7 Analysis.
10.8.4.8 Graph.
10.8.4.9 Slope.
10.8.4.10 Intercept.
10.8.4.11 R^2.
10.8.4.12 Number of washes.
10.8.4.13 Injection volume.
10.8.4.14 Spargetime.
10.8.4.15 Max SD.
10.8.4.16 Max CV.
10.8.4.17 Print Data.
10.8.4.18 Sample Name.
10.8.4.19 Sample ID.
10.8.4.20 Concentration.
10.8.4.21 Number of injections.
10.8.4.22 Mean Area.
10.8.4.23 SD.
10.8.4.24 CV.
10.8.4.25 Maximum number of injections.
10.8.5 Click on "Samples" tab and make the following selections:
10.8.5.1 Analysis.
10.8.5.2 Area of Confidence.
10.8.5.3 Calibration Curves.
10.8.5.4 Concentration.
10.8.5.5 Result.
10.8.5.6 CV.
10.8.5.7 Date/Time.
10.8.5.8 Dilution.
10.8.5.9 Mean Area.
10.8.5.10 Method.
10.8.5.11 Sample ID.
10.8.5.12 Sample name.
10.8.5.13 Type.
10.8.5.14 Unit.
10.8.5.15 Peak profile.
10.8.5.16 SD.
10.8.5.17 Sparge time.
10.8.5.18 Number washes.
10.8.5.19 Unknown.
10.8.5.20 Standard.
10.8.6 Click on "Injections Table" tab.
10.8.7 Select "Select None."
10.8.8 Click on "Statistics Table" tab.

10.7.6.8 Zero point the syringe.
10.7.6.9 Run 10 injections of acidified ASTM Type II water (pH ~3) at start of each use of TOC 5050A.

10.8 Initial Calibration (Daily) – Standalone (sequence listed in Section 10.3.6)
10.8.1 Press the **numeric** and then **enter** keys to set conditions for calibration.
10.8.2 Go to **main menu** screen then to the **maintenance screen**.
10.8.3 Set date and time and **carry out zero point** detection of syringe pump with sample line in ASTM Type II water.
10.8.4 Go back to **main menu** then press **calibration**.
10.8.5 Set conditions, then go to measurement start by pressing **F1** key.
10.8.6 Standards are measured in triplicate and a peak and peak area are displayed. When completed, press **F1 to print results. Then introduce next standard to sampling tube and press start**.
10.8.7 Once all standards have been read and printed, the calibration curve screen will appear.
10.8.8 Designate the **protect option** to store calibration curve data and **least squares option** for calibration curve form.
10.8.9 Press **F5 to print** curve. Calibration is complete. Correlation coefficient must be at least ≥ 0.995 to be acceptable. Run a new curve daily. See calibration curve standards sequence in Section 10.4.6.

10.9 Analysis – Standalone
10.9.1 Go to **main menu** screen, then to **sample measurement**.
10.9.2 Set each condition, number of injections to equal 4 for analysis and press **F1** for measurement start. Then press start button to begin. Sparging is automatic.
10.9.3 Sample is measured against conditions entered at sample measurement/conditions screen. The progress of the measurement, a peak, and result are displayed on screen. When the completed sign is displayed on the screen, Press F1 to print. After printing, the screen will go back to the sample conditions screen for the next sample analysis information.
10.9.4 When done measuring all samples – press **F2**. Go to **data report screen** via main menu. Here all results for every sample are displayed. Press **F5 to print.**
10.9.6 Calibration Run Sequence (curve prepared daily).
10.9.7 Calibration Curve Standards:
50 mg/L Cal Std.
30 mg/L Cal Std.
10 mg/L Cal Std.
0.0 mg/L Cal Std.
10.9.8 Sample Analysis Sequence:
LCS/ICV 10 mg/L
MB
10 analyses (includes sample spike and duplicate)
CCV
CCB
10 analyses
CCV
CCB

10.9.9 System Shutdown - Standalone
10.9.1 System should remain "on" when running samples daily. System should be shut down when not in use for one or more days. Then follow shutdown procedure.
10.9.2 After all samples have been analyzed and printed, press F2 for the **main menu** screen.

10.9.3 Then press **7** followed by the **enter** button for standby options.
10.9.4 To **stop equipment press 1 then enter** button, followed by **F1** key.
10.9.5 Instrument will indicate on screen "**Wait 30 Minutes" to turn off main power.**
10.9.6 After zero minutes displayed on screen, turn off main power and carrier gas supply.

10.10 Daily Instrument Calibration for TC Analysis as it Appears on the Instrument Computer Screen – PC Control and Standalone

Table 10-1

GENERAL CONDITIONS

Description	Source
TC Catalyst	Normal Sens
Syringe Size	250 µL
Number of Washes	4
Unit of Concentration	mg/L
Auto Ranging & Inj Vol	Auto Change
Auto Regeneration of IC	On
Furnace on/off	TOC
Injection Speed	STD
ESU	Not Used
Bubble Removal	Off
Syringe Wash	STD
Cell Length	STD
TOC or SSM	TOC
Printer Device	Int New
Page Length	******
Calibration Curve Form	Least Squares
POC	Not Used
MEAS Interval (TC/NPOC)	0 Seconds
MEAS Interval (IC/POC)	0 Seconds
Revision: 6 | Status: Final | Method: 5310B	Minor Revision Date: None

Table 10-2

CALIBRATION CONDITIONS

Description	Weight	Amount
Cal Curve F#		5
Type		NPOC
1st STD Conc.		50.0 mg/L
2nd STD Conc.		30.0 mg/L
3rd STD Conc.		10.0 mg/L
4th STD Conc.		0.0 mg/L
Range		X 1
Inj Vol		10 µL
No of Injects		5310B: 4; 9060: 4
Max No of Inj		5310B: 5; 9060: 5
SD		200
CV		2.0
Sparging Time		3 minutes

Revision: 6	Status: Final	Method: 5310B	Minor Revision Date: None

Table 10-3

ROUTINE MAINTENANCE PROCEDURES

Equipment/ Instrument	Symptom	Operation	Frequency
Copper scrubber	Black colored	Replace	Check daily
Sampling tube	No peak	Check sampling tube for block; replace when needed.	As required

Revision: 6	Status: Final	Method: 5310B	Minor Revision Date: None

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 Method Calculations

Calculate the relative percent difference (RPD) value between the sample result (S) and duplicate sample result (D) as follows:

$$RPD = \frac{|S - D|}{1/2(S + D)} \cdot 100$$

$$\%\,\text{Recovery} = \frac{(\text{Spiked Sample Result [mg/L]}) - (\text{Original Sample Result [mg/L]})}{\text{Spike Amount (mg/L)}} \cdot 100$$

$$\text{Spike Amount (mg/L)} = \frac{(\text{Volume of Spike Solution [mL]}) \cdot (\text{Concentration Spike Solution [mg/L]})}{\text{Volume Sample (mL)}}$$

11.2 The analytical results are reported as the average of four replicates.

11.3 Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL Water
Total Organic Carbon, TOC	T,L	1.0 mg/L

Revision: 6	Status: Final	Method: 5310B	Minor Revision Date: None

Key Type:

T = Compound/analyte is target compound routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike.
L = Compound/analyte is routinely used as a LCS spike.
Q = Compound/analyte is used as s surrogate spike.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
LCS/ICV	1 per batch. At start after curve.	See Table 8-2. Resulting concentration is 10 mg/L.	See Control Chart	Reanalyze; recalibrate.
CCV	After every 10 analyses and at the end of the run	Table 8-2. Resulting concentration is 30 mg/L	85%-115%	Reanalyze samples back to the last acceptable CCV. Recalibrate.
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 per 20 or fewer samples	0.2 mL of 1000 mg/L Reference Stock Standard to 20 mL of sparged sample. (10 mg/L spike)	Project requirements, if specified	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
CCB/Method Blank (MB)	MB after LCS. CCB after every CCV.	ASTM Type II water acidified with sulfuric acid < 2 pH	< 1.0 mg/L	Reanalyze. Repeat samples back to the last acceptable blank.
Duplicate (D)	If client specified	2nd aliquot of sample	Client specified	Client specified
Revision: 6	Status: Final	Method: 5310B	Minor Revision Date: None	

Table 12-2

CONTROL CHARTED QUALITY CONTROL SAMPLES

QC Type		Analytes Charted	Matrix Charted
LCS		TOC	Water
Revision: 6	Status: Final	Method: 5310B	Minor Revision Date: None

13.0 SPECIAL PROJECT REQUIREMENTS

13.1 U.S. Army Corps of Engineers (ACE)

13.1.1 Method Quantitation Limit (MQL) is between 3-10 times MDL. The low calibration standard concentration must be ≥3 times MDL. The Method Reporting Limit (MRL) may slide between two times the MDL and project specific or regulatory requirements. Results below the MRL must be reported "ND." Results between the MQL and MRL must be reported as estimated (J flagged).

14.0 SAMPLE DISPOSAL

14.1 Samples with less than 1000 mg/L TOC flush down laboratory drain. Use plenty of water to flush traps below sinks.

14.2 For more information see the Waste Disposal SOP A10.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Chemical Oxygen Demand			
Reviewer: C. Okal		Peer Reviewer: F. Lindauer	
Implementation Date: 4/13/00		Last Annual Review Date: 3/26/01	
Revision: 3	Status: Final	Method: SM 5220 C	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 COD at levels of 5 to 1500 mg/L can be analyzed without dilution. Higher levels require dilutions. Premixed vials of reagents are used for all three levels of COD analysis. The ranges for these levels are as follows:

Low level	< 5 - 150 mg/L
Med level	< 5 - 1500 mg/L
High level	< 50 - 15000 mg/L

Dilutions are only made at high level.

2.0 METHOD SUMMARY

2.1 This SOP describes the procedure to determine the quantity of oxygen required to oxidize all organic and inorganic material present within the sample with potassium dichromate in a sulfuric acid solution. The excess dichromate is titrated with standard ferrous ammonium sulfate using ferroin as an indicator. Method modification: premixed vials of reagents for this analysis are purchased and not made in house.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 Use Extreme Caution - This test uses sulfuric acid and mercuric salts at high temperatures.

3.2 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Standard Methods, 18th Edition 1992, Method 5220 C.
4.2 HACH COD - Microdigestion Procedure.
4.3 Training SOP QA.5, A.23, and A.29.
4.4 Data Review SOP A.25.
4.5 Laboratory Quality Assurance Manual.
4.6 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 **COD** – Chemical Oxygen Demand.
5.2 **F.A.S.** –Ferrous Ammonium Sulfate(Fe $(NH_4)_2$ $(SO_4)_2 \cdot 6$ H_2O).
5.3 **Phenanthroline ferrous sulfate indicator (Ferroin)** - Orthophenzntroline Ferrous Complex.
5.4 **KHP** – Potassium hydrogen phthalate.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Traces of organic material either from the glassware or atmosphere may cause a gross, positive error.

6.2 Chlorides are quantitatively oxidized by the dichromate and represent a positive interference. Mercuric sulfate is added to the digestion flask to complex the chlorides, thereby effectively eliminating the interference on all but brine and estuarine samples. Record all pretreatment in the lab notebook.

7.0 INSTRUMENTATION AND EQUIPMENT

Hach micro block digester
Prepared digestion vials with digestion reagent already added (Hach Low, medium, and high levels or equivalent)
Microburet (10.0-mL)
Stir plate with magnetic stir bar
Variety of Class A volumetric pipettes
50-mL buret (for FAS standardization)

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standards Reagents and Reagents

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Potassium hydrogen phthalate (KHP) ($HOOCC_6H_4COOK$)	Fisher or equivalent	Neat
Potassium dichromate ($K_2Cr_2O_7$) Analytical reference grade	Fisher or equivalent	Neat
Sulfuric Acid (H_2SO_4) reagent grade	Fisher or equivalent	Concentrated
Ferrous ammonium sulfate (FAS) ($Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$)	Fisher or equivalent	Neat
Phenanthroline ferrous sulfate indicator (Ferroin)	Lab chem or equivalent	0.025 M
Mercuric Sulfate ($HgSO_4$)	Lab chem or equivalent	Neat

Revision: 3	Status: Final	Method: SM 5220 C	Minor Revision Date: None

8.2 Standard Solution Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
Potassium dichromate standard.	$K_2Cr_2O_7$	Dissolve 12.2588 g $K_2Cr_2O_7$ (dried at 103° C for 2 hrs.) in ASTM Type II water. Dilute to 1000 mL with ASTM Type II water. Good for 6 months.	0.25 N
0.125N Ferrous ammonium sulfate titrant (for high level)	Ferrous Ammonium Sulfate (FAS)	Dissolve 49.0 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in ASTM Type II water. Add 20 mL concentrated H_2SO_4, cool, and dilute to 1000 mL with ASTM Type II water. Good for 6 months. NOTE: Standardize against 0.25 N Potassuim dichromate standard daily. (See Section 8.3)	Approximately 0.125 N
0.0125N Ferrous ammonium sulfate titrant (for low level)	0.125 N Ferrous ammonium sulfate titrant	Pipette 10 mL of standardized 0.125 N $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ and dilute to 100 mL with ASTM Type II water. (See Section 8.3) Make fresh daily.	Approximately 0.0125 N
KHP 1200 (for high and medium level)	KHP	Dissolve 1.0200 g KHP (dried to a constant weight at 120° C) in ASTM Type II water. Dilute to 1000 mL with ASTM Type II water. Good for 6 months.	1200 mg/L
KHP 60 (for low level)	KHP 1200	Pipette 5 mL of KHP 1200 and dilute to 100 mL with ASTM Type II water. Make fresh daily.	60 mg/L

Revision: 3	Status: Final	Method: SM 5220 C	Minor Revision Date: None

8.3 Standardization Procedure for FAS solutions

8.4 Standardize titrant (high level) FAS solution daily against standard potassium dichromate solution as follows:

Dilute 10.0 mL potassium dichromate standard to about 100 mL. Add 10 mL concentrated sulfuric acid (H_2SO_4) and cool. Titrate with FAS titrant using 2 to 3 drops Ferroin Indicator.

$$Normality\ of\ FAS\ Solution = \frac{Volume\ 0.25\ N\ Potassiumdicromates\ \text{tan}\ dard\ Titrated, mL}{Volume\ FAS\ (\sim 0.125\ N)\ used\ in\ Titration, mL} \cdot 0.25\ N\ K_2Cr_2O_7$$

8.5 For low level FAS Titrant, divide the result of the above equation by 10.

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Water	Standard	NA	28 Days	Poly or glass, sulfuric acid pH< 2, 2-6 C
Water	NYSDEC	NA	26 Days	Poly or glass, sulfuric acid pH< 2, 2-6 C
Revision: 3	Status: Final	Method: SM 5220 C		Minor Revision Date: None

10.0 PROCEDURE

10.1 Standards (analyze KHP 1200 standard for high- and medium-level vials, and KHP 60 standard for low-level vials): pipette 2 mL of standard into the appropriate level vial.
Blanks: pipette 2 mL of ASTM Type II water into each level vial used.

10.2 Pipette 2 mL of sample into a low-level vial. If sample turns green, use a medium-level or high-level vial. If 2 mL of sample turns green using a high-level vial, prepare a dilution to get into high-level vial range.

10.3 Cap and invert each vial a few times.

10.4 Place vials into block digester at 150 ± 5° C and digest for at least 2 hours.

10.5 If a green color appears after digestion, the titration should be attempted for mid-level and high-level samples, titrate with 0.125 N FAS. If the titration does not require at least 0.25 mL of titrant, the sample must be redigested using a higher-level vial.

10.6 Remove vials, cool, add 2 drops Ferroin, and titrate using the appropriate FAS until the endpoint is reached. For low-level samples titrate with 0.0125 N FAS. For medium- and high-level samples, use 0.125N FAS. The endpoint goes from blue-green to orange-brown.

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 Sample and LCS Calculations

$$COD\,mg/L = \frac{(B-S)\cdot N\cdot 8000}{V}$$

where:

B = mL of FAS to reach endpoint on the blank (average of two titrations).
S = mL of FAS to reach endpoint on the sample.
V = Milliliters of sample used for the test.
N = FAS normality.

11.2 Matrix Spike Recovery Calculation

$$\%R = \frac{SSR - \frac{1}{2}SR}{SA} \cdot 100$$

where:

SSR = Spiked sample result using Equation 11.1 with 1 mL sample (v).
SA = Spike added to 1 mL sample, 1200 or 60 mg/L (see Table 12-1).
SR = Original sample result found.

The factor ½ results from putting the sample volume to the same basis – 1 mL volume.

Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL Water
COD low level and medium level	T	5 mg/L
COD high level	T	50 mg/L

Revision: 3	Status: Final	Method: SM 5220 C	Minor Revision Date: None

Key Type:

T = Compound/Analyte is target compound routinely reported.
M = Compound/Analyte is listed in the method but is not routinely reported by E & E.
C = Compound/Analyte is specified by the client and can be analyzed under this method.
S = Compound/Analyte is routinely used as a matrix spike.
L = Compound/Analyte is routinely used as a LCS spike.
Q = Compound/Analyte is used as a surrogate spike.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Reference Standard: 60 mg/L (low level LCS)	1 per batch of 20 or fewer samples	Pipette 2.0 mL KHP 60 into low-level vial	See Control Chart Summary	Reanalyze standards and blank. Reprepare batch.
Reference Standard: 1200 mg/L (medium- and high-level LCS)	1 med and/or 1 high per batch[1]	Pipette 2.0 mL KHP 1200 into medium- and/or high-level vial	See Control Chart Summary	Reanalyze standards and blank. Reprepare batch.
Duplicate (DUP)	1 per batch of 20 or fewer samples	2nd aliquot of sample analyzed	Project requirements, if specified: - 20% for waters - 35% for soils If one or both sample results are <5 x PQL, then duplicate results are acceptable if the difference between the two results is < 2 x PQL.	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Blank	2 per level per batch	Pipette 2.0 mL ASTM Type II water in appropriate level vials	NA	NA
Matrix Spike (MS) For medium- or high-level samples	1 per batch of 20 or fewer samples	1 mL KHP 1200 plus 1 mL sample into medium- or high-level vial. Resulting spike added is 1200 mg/L. See Equation 11.2 for proper recovery calculation.	Project requirements if specified. Compare against LCS limits for bias.	Project requirements if any, otherwise evaluate results in conjunction with other QC information (e.g., LCS recoveries) to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Matrix Spike (MS) For low-level samples	1 per batch of 20 or fewer samples	1 mL KHP 60 plus 1 mL sample into a low-level vial. Resulting spike added is 60 mg/L. See Equation 11.2 for proper recovery calculation.	Project requirements if specified. Compare against LCS limits for bias.	Project requirements if any, otherwise evaluate results in conjunction with other QC information (e.g., LCS recoveries) to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.

Revision: 3	Status: Final	Method: SM 5220 C	Minor Revision Date: None

Control Limits for Routine Quality Control Samples

Table 12-2

CONTROL LIMITS FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameters Charted	Type of Samples Charted	Source of Limits	Frequency Updated
LCS	COD	Water	In-House	Annually
Revision: 3	Status: Final	Method: SM 5220 C	Minor Revision Date: None	

13.0 SPECIAL PROJECT REQUIREMENTS

NA

14.0 SAMPLE DISPOSAL

14.1 Titrated vials are stored until disposal in hazardous waste lab pack.

14.2 See Waste Disposal SOP A.10 for more information.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Total Kjeldahl Nitrogen (TKN) by Method 351.3			
Reviewer: C. Okal		Peer Reviewer: F. Lindauer	
Implementation Date: 6/30/99		Last Annual Review Date: 1/16/01	
Revision: 2	Status: Final	Method: 351.3	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP directs the procedure for determining the concentration of organic nitrogen plus ammonia nitrogen, total Kjeldahl Nitrogen (TKN), in drinking water and surface waters, domestic and industrial wastes.

2.0 METHOD SUMMARY

2.1 The applicable range of this method is 0.1 to 20 mg/L TKN. The range may be extended with sample dilution.

2.2 Modification; An autodistillation apparatus (Büchi 316) is used.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves, and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 ***Toxic Sulfur Trioxide is released during digestion. Perform in a fume hood.***

3.3 **Pollution Prevention** – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 EPA Method 351.3.

4.2 Standard Methods 4500-NH_3 B, E, 18th edition, 1992.

4.3 Standard Methods 4500-N_{org}B, 18th edition, 1992.

5.0 DEFINITIONS/ACRONYMS

5.1 **TKN** - Total Kjeldahl Nitrogen is the sum of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate $(NH_4)_2 SO_4$, under the conditions of digestion.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

NA

7.0 INSTRUMENTATION AND EQUIPMENT

Büchi 316 Distillation Unit

- 250-mL Erlenmeyer flasks
- 250-mL Digestion/distillation tubes
- Bump rods

Table 8-2

STANDARD AND REAGENT PREPARATION

Stock	Standard Name	____ Amount of Stock Added to ____	Final Concentration
Ammonium Chloride (NH_4Cl)	Ammonia Reference Stock	Dissolve 3.819 g NH_4Cl in a 1-L volumetric flask and diluted to the mark with ASTM Type II water. Good for 6 months.	1000 mg/L as N
Sodium Hydroxide (NaOH) Sodium Thiosulfate (Na_2SO_3-$5N_2O$)	Sodium Hydroxide-Sodium Thiosulfate Solution	Dissolve 500 g sodium hydroxide and 25 g sodium thiosulfate in ASTM Type II water, dilute to 1000 mL with ASTM Type II water. Good for 6 months.	NA
Boric Acid	Boric Acid Solution	Dissolve 20 g boric acid $B(OH)_3$ in ASTM Type II water and dilute to 1 L.	2% by volume
Sulfuric Acid (H_2SO_4)	0.02 N H_2SO_4	Buy direct from Fisher.	0.02 N
Sulfuric Acid	0.10 N Sulfuric Acid	3.0 mL concentrated sulfuric acid to 1000 mL with ASTM Type II water.	0.10 N
Hydrochloric Acid	1:1 HCl	Mix 50 mL concentrated HCl into 50 mL ASTM Type II water. Good for 6 months.	1:1
Methyl Red Methylene Blue in Ethyl Alcohol	Mixed Indicator	Mix two volumes of 0.2% methyl red in 95% ethyl alcohol with one volume 0.2% methylene blue in 95% ethyl alcohol. This solution should be prepared fresh every 30 days.	NA

Revision: 2	Status: Final	Method: 351.3	Minor Revision Date: None

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Water	Standard	NA	28	Concentrated sulfuric acid 2-6 C to pH <2
Soil	Standard	NA	28	2-6 C no preserve

Revision: 2	Status: Final	Method: 351.3	Minor Revision Date: None

10.0 PROCEDURE

10.1 Digestion

10.1.1 Place 100 mL of sample into 250-mL digestion/distillation tube.

10.1.2 Place one bump rod into each tube.

10.1.3 Add 20 mL of digestion reagent into each tube.
10.1.4 Place rack holder with tubes into block digestor.
10.1.5 Turn on block digestor and press start when samples are in the digestor.
10.1.6 Digest samples until white vapor is formed, approximately 2.5 hours at 380 C.
10.1.7 Caution: Digestion must be performed in a hood - white vapor (sulfur trioxide) is toxic.
10.1.8 Continue digestion for an additional 30 minutes after white vapor forms.
10.1.9 Remove the rack and tubes from block, turn off block digestor. Cool.
10.1.10 Add 70 mL ASTM Type II water to digestion tube.

10.2 Distillation

10.2.1 With cold tap water running through the condenser, turn the Büchi 316 on and let it warm up for at least 5 minutes. Make sure Büchi water reservoir jug is at least half full with ASTM Type II water.
10.2.2 Set Büchi timer at 6 minutes.
10.2.3 To clean out distillation apparatus add approximately 15 mL of 1:1HCl to 200 mL ASTM Type II water and distill. Then distill an instrument blank to ensure the apparatus is clean.
10.2.4 Turn asparation knob to "yes" for waters and "no" for soils.
10.2.5 Add 20 mL sodium hydroxide-sodium thiosulfate solution.
10.2.6 Distill into 25 mL of 2% boric acid contained in a 250-mL Erlenmeyer flask. **Note:** The condenser tip must be below the surface level of the boric acid solution.
10.2.7 With rubber grip tongs remove the digestion distillation tube from the apparatus. **Caution: Tube is HOT**.
10.2.8 Distillation tube waste must be disposed of into the mercury salts waste drum.
10.2.9 Add a few drops mixed indicator to the distillate and titrate with 0.02 N H_2SO_4. The color will change from green to a purple end point.
10.2.10 Turn off Büchi 316 distillation unit.
10.2.11 Turn off cold water for the condenser.

10.3 Soil Preparation

10.3.1 The procedure can be modified for soils by weighing 5 g sample then adding 100 mL of ASTM Type II water to the digestion/distillation tube. Then follow procedure starting at step 10.1.2.

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment Instrument	Symptom	Operation	Frequency
Glassware	Star cracks, chips, damaged	Do not use	Replace as needed
Fumehood	Not Drawing	Notify Supervisor or Lab Manager	As necessary
Büchi 316	Contaminated blank	Clean system with 1:1 HCl	As needed
Büchi 316	No distillate collected.	Fill reservoir with ASTM Type II water	As needed

Revision: 2	Status: Final	Method: 351.3	Minor Revision Date: None

10.4 Analytical Run Sequence

- MB
- LCS
- Up to 10 analyses
- LCS

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL	
		Water	Soil
TKN	T	< 0.3 mg/L for 100 mL sample	< 6.0 mg/kg for a 5 g sample
Revision: 2	Status: Final	Method: 351.3	Minor Revision Date: None

Key Type:

T = Compound/analyte is target compound routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike.
L = Compound/analyte is routinely used as a LCS spike.
Q = Compound/analyte is used as a surrogate spike.

11.2 Method Calculation

$$\text{TKN}\,(\text{mg/L or mg/kg}) = \frac{N \cdot (V - B) \cdot 14{,}000}{C}$$

11.3 where:

B = Volume of 0.02 N H_2SO_4 necessary to titrate blank.
C = Volume of sample (mL) or weight in grams.
N = Normality of H_2SO_4 titrant used.
V = Volume of 0.02 N H_2SO_4 used (mL) to titrate sample.
14,000 = Conversion from meq N/mL to mg N/L.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Routine Quality Control Samples

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
LCS 10 mg/L	1 per batch of 20 or fewer samples	1 mL of 1000 mg/L Ammonia Reference Stock added to 100 mL ASTM Type II water and processed.	See Control Limit Summary, TKN	Repeat digestion. Make new standard.
Duplicate	1 per batch of 10 or fewer samples	A second portion of sample processed.	Project requirements, if specified: 20% for waters 35% for soils If one or both sample results are <5 x PQL, then duplicate results are acceptable if the difference between the two results is < 2 x PQL.	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Matrix Spike Soil: 200 mg/kg Water: 10 mg/L	1 per batch of 20 or fewer samples	1 mL of 1000 mg/L Ammonia Reference Spike added to another 100-mL aliquot of sample and processed or a 5-g portion of soil sample and processed.	Project requirements if specified. Compare against LCS limits for bias.	Project requirements if any, otherwise evaluate results in conjunction with other QC information (e.g., LCS recoveries) to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Method Blank (MB)	1 per batch of 20 or fewer samples	100 mL of ASTM Type II water processed.	<0.3 mg/L waters <6.0 mg/kg soils	Repeat

Revision: 2	Status: Final	Method: 351.3	Minor Revision Date: None

Control Limits for Routine Quality Control Samples

Table 12-2

CONTROL LIMITS FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameters Charted	Type of Samples Charted	Source of Limits	Frequency Updated
LCS	TKN	Water, Soil as Water	In-House	Annually
DUP	TKN	Water, Soil	In-House	Annually
Spike	TKN	Water, Soil	In-House	Annually
Revision: 2	Status: Final	Method: 351.3	Minor Revision Date: None	

13.0 SPECIAL PROJECT REQUIREMENTS

NA

14.0 SAMPLE DISPOSAL

14.1 Residue in the digestion/distillation has to be dumped in the hazardous waste mercury salts drum located in the General Analytical Laboratory.

14.2 The boric acid solution (that was used for titration) can be flushed down the sink with copious amounts of tap water.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Ammonia-Nitrogen by Method SM4500-NH3 B E			
Reviewer: D. Ruszczyk		Peer Reviewer: None	
Implementation Date: 5/18/98		Last Annual Review Date: 7/27/00	
Revision: 1	Status: Final	Method: SM4500-NH3 B E	Minor Revision Date: None

1.0 SCOPE AND APPLICATION

1.1 This SOP covers the determination of ammonia-nitrogen, in drinking, surface and saline waters, domestic and industrial wastes. The method has been modified to determine ammonia-nitrogen in soils and waste. The macro distillation option of the method is used in this SOP.

2.0 METHOD SUMMARY

2.1 The sample is buffered at pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanides and organic nitrogen compounds, and is then distilled into a solution of boric acid.

2.2 The ammonia in the distillate is then determined titrimetrically with standard sulfuric acid with the use of a mixed indicator.

2.3 Modification: An automated distillation system (Büchi 316) is used to distill the samples.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves and a lab coat. For more information see the E & E, Inc., Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 Pollution Prevention – Purchase chemicals based on expected usage, shelf life, and disposal cost. Prepare standard volumes on anticipated usage. Make appropriately sized dilutions and use serial dilutions where practical.

4.0 REFERENCES

4.1 Method SM4500 NH3 B E, Methods for Chemical Analysis of Water and Wastes, EPA –600/4-79-020, March 1979.

4.2 Training SOP QA.5, A.23, and A.29.

4.3 Data Review SOP A.25.

4.4 Laboratory Quality Assurance Manual.

4.5 Corrective Action Procedure, QA.1.

5.0 DEFINITIONS/ACRONYMS

5.1 **NH_3-N** = Ammonia as nitrogen.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Volatile alkaline compounds, such as certain ketones, aldehydes, and alcohols, may cause an off color in this distillation method.

6.2 Residual chlorine must be removed by pretreatment with sodium thiosulfate prior to distillation

7.0 INSTRUMENTATION AND EQUIPMENT

Büchi 316 distillation unit
Distillation tubes (300-mL)
Erlenmeyer flask (250-mL)
Stir plate
pH meter
Stir bars
Stir bar retriever
Micro burette -10-mL
Rubber grip tongs
Large tube rack
Potassium iodine paper (KI paper)

8.0 REAGENTS AND MATERIALS

8.1 See Table 8-1, Summary of Standards, Reagents, and Consumables

Table 8-1

SUMMARY OF STANDARD AND REAGENTS

Description	Source	Concentration
Ammonium chloride (NH_4Cl)	Fisher or equivalent	Neat
Boric acid (H_3BO_3)	Fisher or equivalent	Neat
Methyl red	Kodak or equivalent	Neat
Methylene blue	Kodak or equivalent	Neat
Sodium hydroxide (NaOH)	Fisher or equivalent	Neat
Sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$)	Fisher or equivalent	Neat
Sodium tetraborate ($Na_2B_4O_7$)	Fisher or equivalent	Neat
Sulfuric acid (H_2SO_4)	Fisher or equivalent	Concentrated
Sulfuric acid 0.02 N	Fisher or equivalent	0.02 N
Sodium carbonate (Na_2Co_3)	Fisher or equivalent	Neat

Revision: 1	Status: Final	Method: SM4500-NH3 B E	Minor Revision Date: None

8.2 Standard Solution Preparation

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
Ammonium chloride stock solution	Ammonium chloride	Dissolve 3.819 g NH_4Cl in ASTM Type II water and bring to volume in a 1-L volumetric flask. Good for 6 months.	1000 mg/L NH_3-N
2% Boric acid solution	Boric acid	Dissolve 20 g boric acid in 1-L ASTM Type II water. Good for 6 months.	2%

Table 8-2

STANDARD AND REAGENT PREPARATION

Standard Name	Stock	____ Amount of Stock Diluted to ____	Final Concentration
Mixed indicator	Methyl red ethyl alcohol Methylene blue	Dissolve 0.20 g methyl red indicator in 100 mL, 95% ethyl or isopropyl alcohol. Dissolve 0.10 g methylene blue in 50 mL, 95% ethyl or isopropyl alcohol. Combine solutions. Prepare monthly.	NA
Sodium tetraborate solution	Sodium borate ($Na_2B_4O_7 \cdot 10H_2O$)	Dissolve 9.5 g sodium borate into 1 L of ASTM Type II water. Good for 6 months.	0.025M
Borate buffer	Sodium tetraborate/sodium hydroxide	Add 88 mL of 0.1 N NaOH solution to 500 mL of sodium tetraborate solution and dilute to 1-L with ASTM Type II water. Good for 6 months.	NA
1 N NaOH	Sodium hydroxide	Dissolve 40 g sodium hydroxide (NaOH) into 1-L ASTM Type II water. Good for 6 months.	1 N
0.1 Normal sulfuric acid	Sulfuric acid	3.0 mL concentrated sulfuric acid to 1000 mL with ASTM Type II water. Standardize against sodium carbonate solution. Good for 6 months.	0.1 N
0.02 Normal sulfuric acid	Sulfuric acid	Purchase or prepare by adding 200 mL of 0.10 normal sulfuric acid to 1000 mL with ASTM Type II water (standardize against sodium carbonate solution every 3 months).	0.02 N
0.05 Normal sodium carbonate solution	Sodium carbonate (Na_2CO_3)	Dissolve 2.50 of anhydrous sodium carbonate (oven dried at 140°C for at least 4 hours) to 1000 mL with ASTM Type II water. Good 1 week.	0.05 N Nominal
Revision: 1	Status: Final	Method: SM4500-NH3 B E	Minor Revision Date: None

8.3 STANDARDIZATION

8.3.1 Standardize sulfuric acid against 40.00 mL 0.05N Na_2CO_3 solution, with about 60 mL water in a beaker by titrating potentiometrically to pH of about 5. Lift out electrodes, rinse into the same beaker and boil gently for 3 to 5 minutes under a watch glass cover. Cool to room temperature, rinse cover glass into beaker, and finish titrating to the pH inflection point. Calculate normality:

$$\text{Normality, N} = \frac{A \cdot B}{53.00 \cdot C}$$

where:

A = g Na_2CO_3 weighed into 1-L flask (g/L).
B = mL Na_2CO_3 solution taken for titration.
C = mL acid used.

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Water	Standard	NA	28	Poly or glass, H_2SO_4 to pH <2, 2-6 C
Soil	Standard	NA	28	Poly or glass jar – no preservative
Revision: 1	Status: Final	Method: SM4500-NH3 B E		Minor Revision Date: None

10.0 PROCEDURE

10.1 Turn Büchi 316 on, making sure that you have the cold tap water running through the condenser tube. Make sure the Büchi water reservoir jug is at least half full with ASTM Type II water. Let the Büchi 316 warm up for at least 5 minutes.

10.2 Set the Büchi 316 at 4 minutes.

10.3 To clean out distillation apparatus, add approximately 15 mL 1:1 HCl to 200 mL ASTM Type II water and run. Then distill an instrument blank to insure the apparatus is clean making sure aspiration dial is set at "yes."

10.4 Water samples: Check for residual chlorine with potassium iodide paper.

10.5 Remove any residual chlorine in the sample by adding dechlorinating agent equivalent to the residual chlorine.

10.6 Water sample, use up to 200 mL in distillation tube.

10.7 Soil sample use 10 g and 200 mL ASTM Type II water in a distillation tube.

10.8 Adjust pH by adding 1 N sodium hydroxide, until pH is 9.5, checking pH during addition with a calibrated pH meter.

10.9 Distillation: Add approximately12-13 mL of the borate buffer.

10.10 Turn knob to aspiration "yes" for waters "no" for soils.

10.11 Distill into 25 mL of 2% boric acid contained in a 250-mL Erlenmeyer flask.

10.12 Note: The condenser tip or an extension of the condenser tip must extend below the level of the boric acid solution.

10.13 Remove distillation tube from apparatus. **Tube is hot**, use rubber grip tongs. Add a few drops of the mixed indicator to the distillate and titrate with 0.02 N sulfuric acid to a purple end point. The color change goes from green to an end point of purple.

10.14 Turn off Büchi 316 distillation unit.

10.15 Turn off cold water for the condenser.

10.16 Analytical Run Sequence

Prep Blank
LCS
Up to 10 Analyses
DUP
MS

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment Instrument	Symptom	Operation	Frequency
Büchi 316 Distillation apparatus	Apparatus Contamination	Clean instrument by running a solution of 15 mL 1:1 HCl and 200 mL ASTM Type II water. Distill a blank to ensure it is clean.	Before and after use.
Büchi 316	No distillate being collected.	Add ASTM Type II water to reservoir.	As needed.

Revision: 1	Status: Final	Method: SM4500-NH3 B E	Minor Revision Date: None

11.0 DATA REDUCTION/EVALUATION/REPORTING

Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL	
		Water	Soil
NH_3-N	T	0.2 mg/L for 200 mL sample based on 0.1 mL titrant	3.0 mg/kg for 10 g sample based on 0.1 mL titrant

Revision: 1	Status: Final	Method: SM4500-NH3 B E	Minor Revision Date: None

Key Type:

T = Compound/analyte is target compound routinely reported.
M = Compound/analyte is listed in the method but is not routinely reported by E & E.
C = Compound/analyte is specified by the client and can be analyzed under this method.
S = Compound/analyte is routinely used as a matrix spike.
L = Compound/analyte is routinely used as a LCS spike.
Q = Compound/analyte is used as a surrogate spike.

11.1 Method Calculations

$$\text{Ammonia nitrogen (mg/L, mg/kg)} = \frac{\Delta V \cdot N \cdot 14{,}000}{C}$$

where:

ΔV = mL 0.02N H_2SO_4 titrant used.
N = Normality of H_2SO_4 titrant used.
C = Volume of sample (mL or g).
14,000 = mg NH_3–N/meq · 1000 mL/L.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Routine Quality Control Samples

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Method Blank	1 Batch	200 mL ASTM Type II water and process as sample.	< 0.2 mg/L	Repeat samples >0.2 mg/L and <10 times blank.
LCS Ref. Std.	At beginning and after every 20 analyses	1 mL of ammonium chloride stock solution (1000 mg/L) into 200 mL ASTM Type II water. (5 mg/L)	See Control Limit Summary, Ammonia distilled	Repeat once. If fails, repeat all samples.
Duplicate (DUP)	1/10 or less	Up to 200 mL sample for water, 10 g sample for soil.	- Project requirements, if specified - 20% for waters - 35% for soils If one or both sample results are <5 x PQL, then duplicate results are acceptable if the difference between the two results is < 2 x PQL.	Project requirements if any, otherwise evaluate results in conjunction with other QC information to determine the effect of the matrix on the bias of the analysis. Comment in narrative if appropriate.
Matrix Spike	1/20 or less	1 mL ammonium chloride stock solution (1000 mg/L) into a total of 200 mL sample for water and 10 g sample for soils. (5 mg/L; 100 mg/kg)	- Project requirements if specified. - Compare against LCS limits for bias.	Project requirements if any, otherwise evaluate results in conjunction with other QC information (e.g., LCS recovery) to determine the

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
				fect of the matrix on the bias of the analysis. Comment in narrative if appropriate.

Revision: 1	Status: Final	Method: SM4500-NH3 B E	Minor Revision Date: None

Table 12-2

CONTROL CHARTED QUALITY CONTROL SAMPLES

QC Type	Analytes Charted	Matrix Charted
LCS	NH_3-N	Water Soil as water

Revision: 1	Status: Final	Method: SM4500-NH3 B E	Minor Revision Date: None

Table 12-3

CRITERIA ASSIGNED TO ROUTINE QUALITY CONTROL SAMPLES

QC Type	Matrix	Analyte	Nominal Spike Amount	Recovery (%)	RPD	Source
LCS	Water Soil as water	NH_3-N	5 mg/L	See Control Limit Summary, Ammonia	NA	Laboratory

Revision: 1	Status: Final	Method: SM4500-NH3 B E	Minor Revision Date: None

13.0 SPECIAL PROJECT REQUIREMENTS

NA

14.0 SAMPLE DISPOSAL

14.1 Rinse down sink drain with water.

15.0 EXAMPLE FORMS

NA

END OF SOP

Quality Assurance Project Plan
Attachment C
Data Qualifier Flags

Data qualifier definitions were supplied by the Office of Solid Waste and Emergency Response (September 1989) and are included in the functional guidelines. Data qualifiers may be combined (UJ, QJ) with the corresponding combination of meanings. Additional qualifiers may be added to provide additional, more specific information (JL, UB, QJK), modifying the meaning of the primary qualifier. Additional qualifiers utilized by E & E are H, L, K, B, Q, and D.

U - The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation or detection limit, which has been adjusted for sample weight/sample volume, extraction volume, percent solids, sample dilution, or other analysis-specific parameters.

An additional qualifier, B, may be appended to indicate that the analyte was detected in the sample. The presence of the analyte may be attributable to blank contamination. The analyte is therefore considered undetected with the sample detection or quantitation limit for the analyte being elevated.

J - The analyte was analyzed for, but the associated numerical value may not be consistent with the amount actually present in the environmental sample or may not be consistent with the sample detection or quantitation limit. The value is an estimated quantity. The data should be seriously considered for decision-making and are usable for many purposes.

An additional qualifier will be appended to the J qualifier that indicates the bias in the reported results:

L Low bias

H High bias

K Unknown bias

Q The reported concentration is less than the sample quantitation limit for the specific analyte in the sample

The L and H qualifiers will be employed only when a single qualification is required. When more than one quality control parameter affects the analytical result and a conflict results in assigning a bias, the result will be flagged JK.

R - Quality control indicates that data are unusable for all purposes. The analyte was analyzed for, but the presence or absence of the analyte has not been verified. Resampling and reanalysis are necessary for verification to confirm or deny the presence of an analyte.

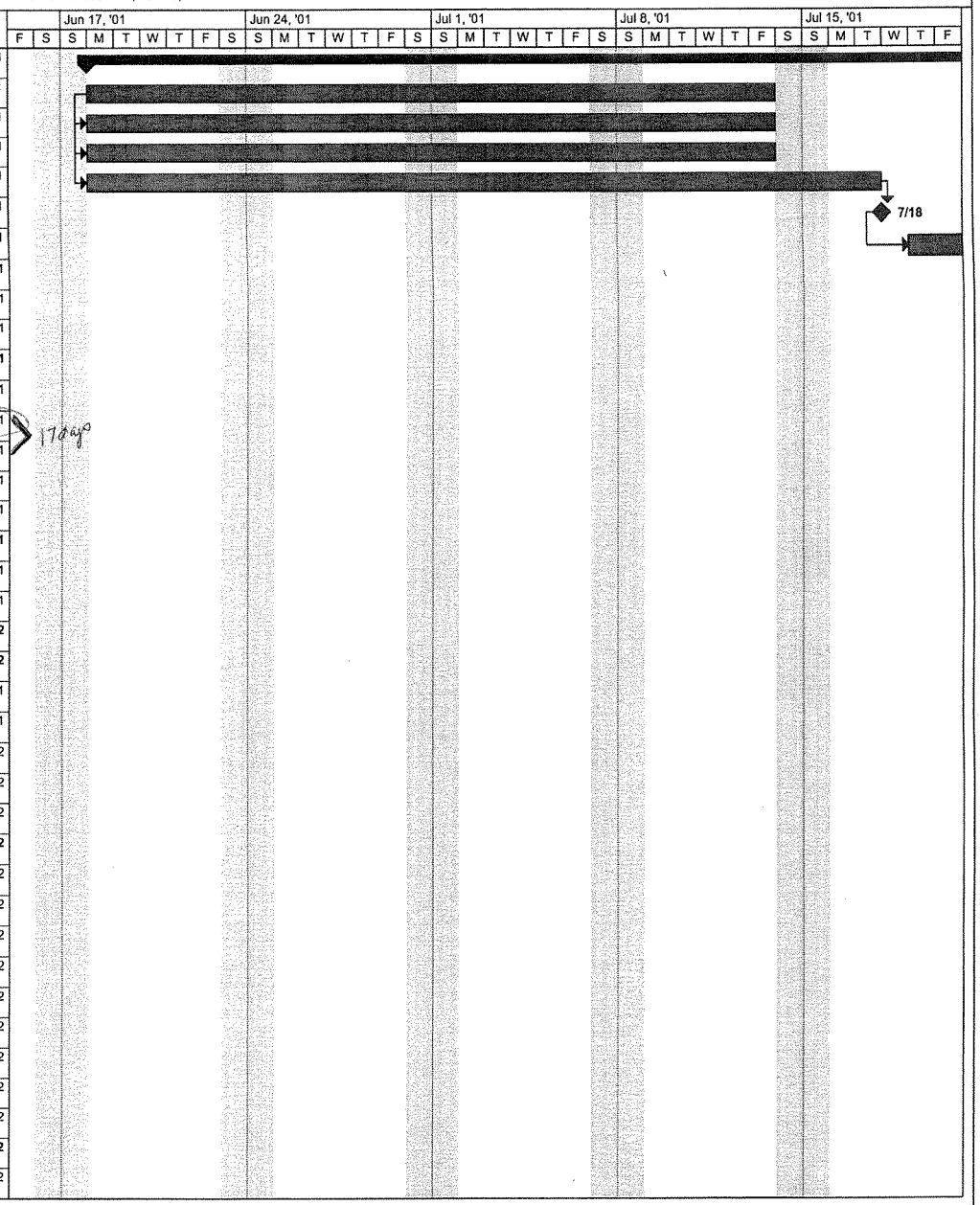
N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."

D - The reported concentration was determined in the reanalysis of the sample at a secondary dilution.

Appendix D
Project Schedule

Former Laredo AFB, Laredo, TX **DRAFT**

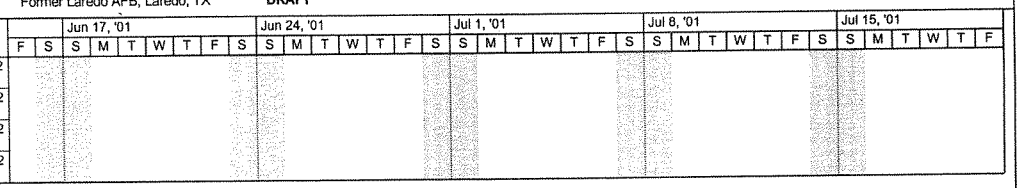
ID	Task Name	Duration	Start	Finish
1	**Planning Documents**	**96 days**	**Mon 6/18/01**	**Mon 10/29/01**
2	Work Plan	20 days	Mon 6/18/01	Fri 7/13/01
3	Site Safety & Health Plan	20 days	Mon 6/18/01	Fri 7/13/01
4	Quality Assurance Project Plan	20 days	Mon 6/18/01	Fri 7/13/01
5	Field Sampling Plan	22 days	Mon 6/18/01	Tue 7/17/01
6	Submit Draft Planning Documents	1 day	Wed 7/18/01	Wed 7/18/01
7	USACE/TNRCC Review	31 days	Thu 7/19/01	Thu 8/30/01
8	Respond to Comments/Revise Planning Documents	16 days	Fri 9/14/01	Fri 10/5/01
9	Submit Final Planning Documents	1 day	Mon 10/8/01	Mon 10/8/01
10	USACE Approval of Final Planning Documents	15 days	Tue 10/9/01	Mon 10/29/01
11	**Field Activities**	**29 days**	**Tue 10/30/01**	**Fri 12/7/01**
12	Procure Subcontractors	9 days	Tue 10/30/01	Fri 11/9/01
13	Install Monitoring Wells	5 days	Mon 11/12/01	Fri 11/16/01
14	Collect Groundwater Samples	17 days	Thu 11/15/01	Fri 12/7/01
15	Collect Soil Samples	3 days	Mon 11/5/01	Wed 11/7/01
16	Survey Monitoring Wells	2 days	Mon 11/19/01	Tue 11/20/01
17	**Laboratory Analysis**	**45 days**	**Tue 10/30/01**	**Mon 12/31/01**
18	Procure Laboratory	9 days	Tue 10/30/01	Fri 11/9/01
19	Analyze samples	40 days	Tue 11/6/01	Mon 12/31/01
20	**Statistical Analysis and Report**	**244 days**	**Wed 11/21/01**	**Mon 10/28/02**
21	**Data Analysis**	**75 days**	**Wed 11/21/01**	**Tue 3/5/02**
22	Generate Well/Soil Logs	5 days	Wed 11/21/01	Tue 11/27/01
23	Generate Potentiometric Surface	3 days	Mon 12/17/01	Wed 12/19/01
24	QA Laboratory Results	20 days	Wed 1/2/02	Tue 1/29/02
25	Compile Laboratory Data	5 days	Wed 1/30/02	Tue 2/5/02
26	Statistical Analysis	20 days	Wed 2/6/02	Tue 3/5/02
27	**Background Study Report**	**169 days**	**Wed 1/2/02**	**Mon 8/26/02**
28	Complete Draft Report	68 days	Wed 1/2/02	Fri 4/5/02
29	Submit Draft Report	1 day	Mon 4/8/02	Mon 4/8/02
30	USACE/TNRCC Review of Draft Report	20 days	Tue 4/9/02	Mon 5/6/02
31	Address Comments/Complete Draft Final Repo	34 days	Tue 5/7/02	Fri 6/21/02
32	Submit Draft Final Report	1 day	Mon 6/24/02	Mon 6/24/02
33	USACE/TNRCC Review of Draft Final Report	15 days	Tue 6/25/02	Mon 7/15/02
34	Address Comments/Complete Final Report	19 days	Tue 7/16/02	Fri 8/9/02
35	Submit Final Report	1 day	Mon 8/12/02	Mon 8/12/02
36	USACE Approval of Final Report	10 days	Tue 8/13/02	Mon 8/26/02
37	**Technical Memoranda**	**45 days**	**Tue 8/27/02**	**Mon 10/28/02**
38	Complete Draft Technical Memoranda	14 days	Tue 8/27/02	Fri 9/13/02



Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

ID	Task Name	Duration	Start	Finish
39	Submit Draft Technical Memoranda	1 day	Mon 9/16/02	Mon 9/16/02
40	USACE/TNRCC Review of Draft Memoranda	15 days	Tue 9/17/02	Mon 10/7/02
41	Address Comments/Complete Final Technical	14 days	Tue 10/8/02	Fri 10/25/02
42	Submit Final Technical Memoranda	1 day	Mon 10/28/02	Mon 10/28/02



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Former Laredo AFB, Laredo, TX **DRAFT**

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5	Field Sampling Plan	22 days	Mon 6/18/01	Tue 7/17/01
6	Submit Draft Planning Documents	1 day	Wed 7/18/01	Wed 7/18/01
7	USACE/TNRCC Review	31 days	Thu 7/19/01	Thu 8/30/01
8	Respond to Comments/Revise Planning Documents	16 days	Fri 9/14/01	Fri 10/5/01
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12	Procure Subcontractors	9 days	Tue 10/30/01	Fri 11/9/01
13	Install Monitoring Wells	5 days	Mon 11/12/01	Fri 11/16/01
14	Collect Groundwater Samples	17 days	Thu 11/15/01	Fri 12/7/01
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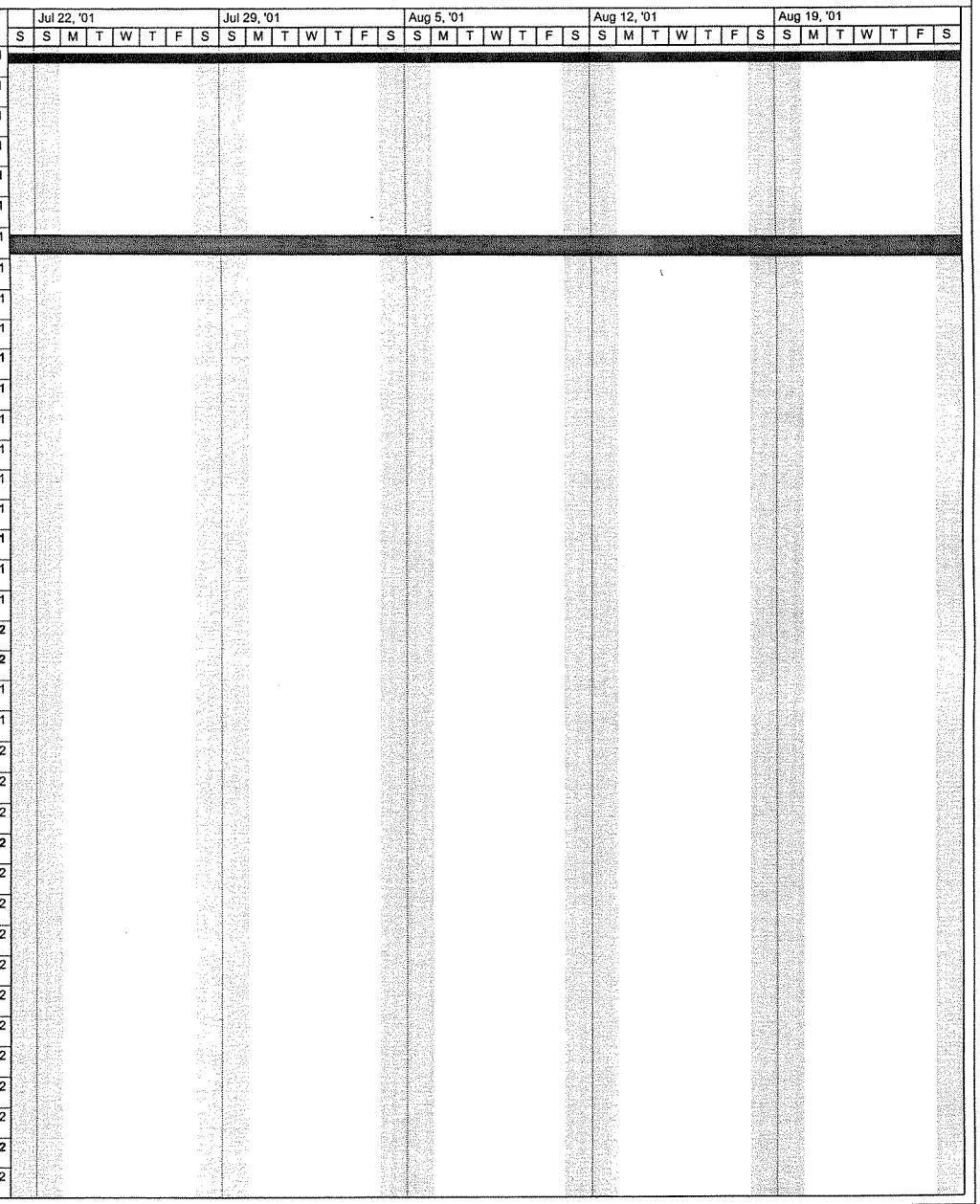

Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

Former Laredo AFB, Laredo, TX **DRAFT**

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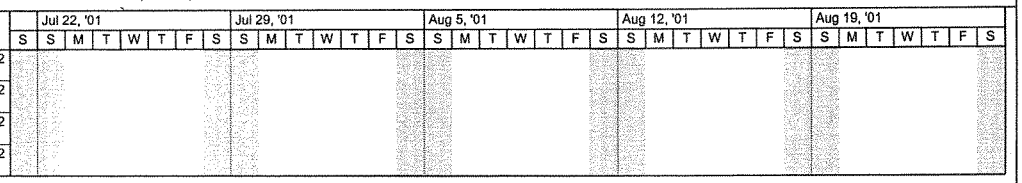

Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

Former Laredo AFB, Laredo, TX **DRAFT**

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14	Collect Groundwater Samples	17 days	Thu 11/15/01	Fri 12/7/01
15	Collect Soil Samples	3 days	Mon 11/5/01	Wed 11/7/01
16	Survey Monitoring Wells	2 days	Mon 11/19/01	Tue 11/20/01
17	**Laboratory Analysis**	**45 days**	**Tue 10/30/01**	**Mon 12/31/01**
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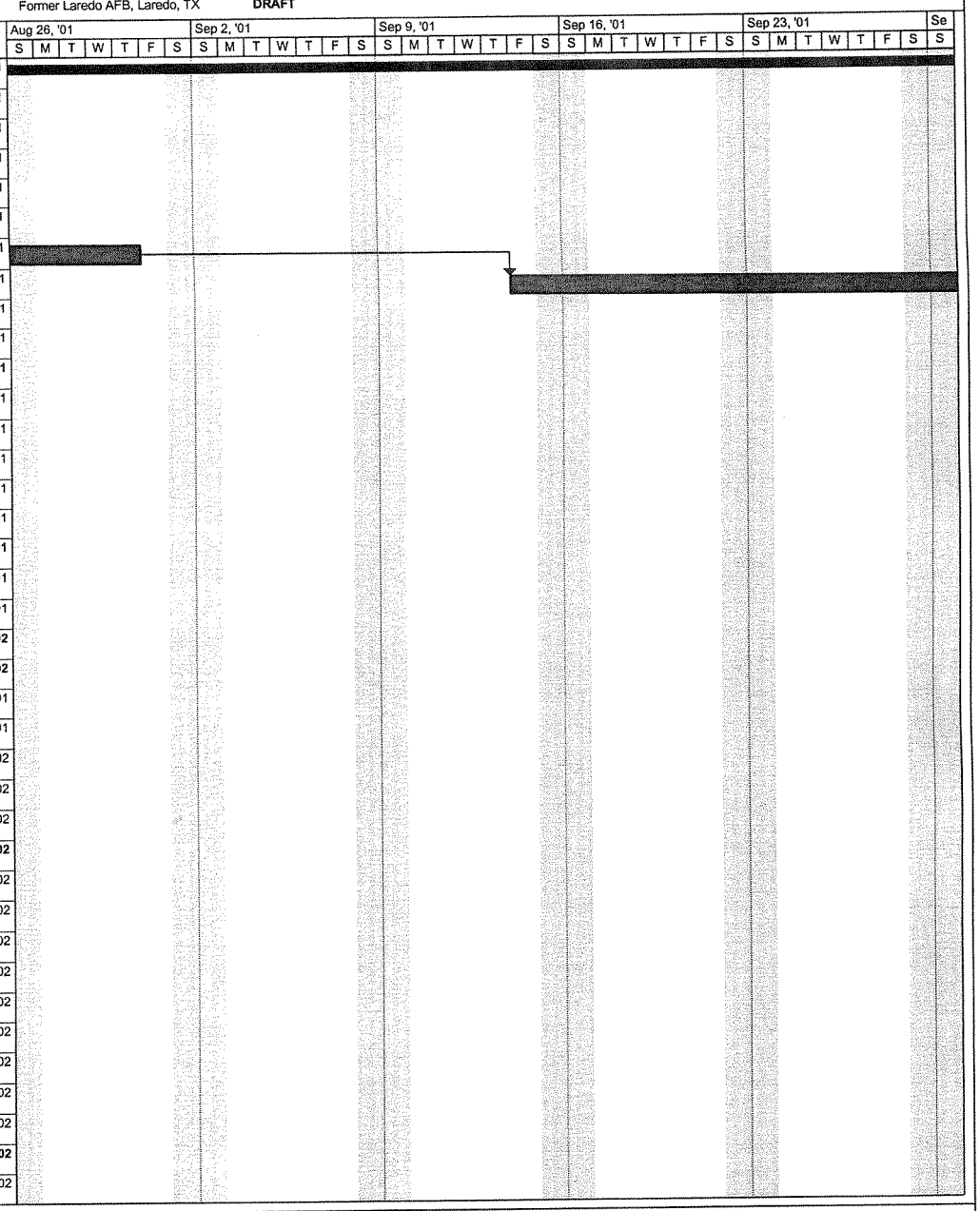
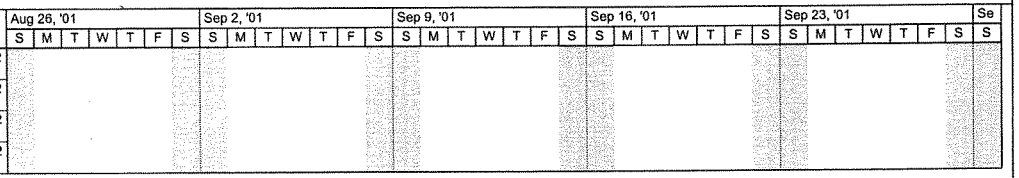
26	Statistical Analysis	20 days	Wed 2/6/02	Tue 3/5/02
27	**Background Study Report**	**169 days**	**Wed 1/2/02**	**Mon 8/26/02**
28	Complete Draft Report	68 days	Wed 1/2/02	Fri 4/5/02
29	Submit Draft Report	1 day	Mon 4/8/02	Mon 4/8/02
30	USACE/TNRCC Review of Draft Report	20 days	Tue 4/9/02	Mon 5/6/02
31	Address Comments/Complete Draft Final Repo	34 days	Tue 5/7/02	Fri 6/21/02
32	Submit Draft Final Report	1 day	Mon 6/24/02	Mon 6/24/02
33	USACE/TNRCC Review of Draft Final Report	15 days	Tue 6/25/02	Mon 7/15/02
34	Address Comments/Complete Final Report	19 days	Tue 7/16/02	Fri 8/9/02
35	Submit Final Report	1 day	Mon 8/12/02	Mon 8/12/02
36	USACE Approval of Final Report	10 days	Tue 8/13/02	Mon 8/26/02
37	**Technical Memoranda**	**45 days**	**Tue 8/27/02**	**Mon 10/28/02**
38	Complete Draft Technical Memoranda	14 days	Tue 8/27/02	Fri 9/13/02



Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

ID	Task Name	Duration	Start	Finish
39	Submit Draft Technical Memoranda	1 day	Mon 9/16/02	Mon 9/16/02
40	USACE/TNRCC Review of Draft Memoranda	15 days	Tue 9/17/02	Mon 10/7/02
41	Address Comments/Complete Final Technical	14 days	Tue 10/8/02	Fri 10/25/02
42	Submit Final Technical Memoranda	1 day	Mon 10/28/02	Mon 10/28/02



Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
1	**Planning Documents**	**96 days**	**Mon 6/18/01**	**Mon 10/29/01**
2	Work Plan	20 days	Mon 6/18/01	Fri 7/13/01
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9	Submit Final Planning Documents	1 day	Mon 10/8/01	Mon 10/8/01
10	USACE Approval of Final Planning Documents	15 days	Tue 10/9/01	Mon 10/29/01
11	**Field Activities**	**29 days**	**Tue 10/30/01**	**Fri 12/7/01**
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13	Install Monitoring Wells	5 days	Mon 11/12/01	Fri 11/16/01
14	Collect Groundwater Samples	17 days	Thu 11/15/01	Fri 12/7/01
15	Collect Soil Samples	3 days	Mon 11/5/01	Wed 11/7/01
16	Survey Monitoring Wells	2 days	Mon 11/19/01	Tue 11/20/01
17	**Laboratory Analysis**	**45 days**	**Tue 10/30/01**	**Mon 12/31/01**
18	Procure Laboratory	9 days	Tue 10/30/01	Fri 11/9/01
19	Analyze samples	40 days	Tue 11/6/01	Mon 12/31/01
20	**Statistical Analysis and Report**	**244 days**	**Wed 11/21/01**	**Mon 10/28/02**
21	**Data Analysis**	**75 days**	**Wed 11/21/01**	**Tue 3/5/02**
22	Generate Well/Soil Logs	5 days	Wed 11/21/01	Tue 11/27/01
23	Generate Potentiometric Surface	3 days	Mon 12/17/01	Wed 12/19/01
24	QA Laboratory Results	20 days	Wed 1/2/02	Tue 1/29/02
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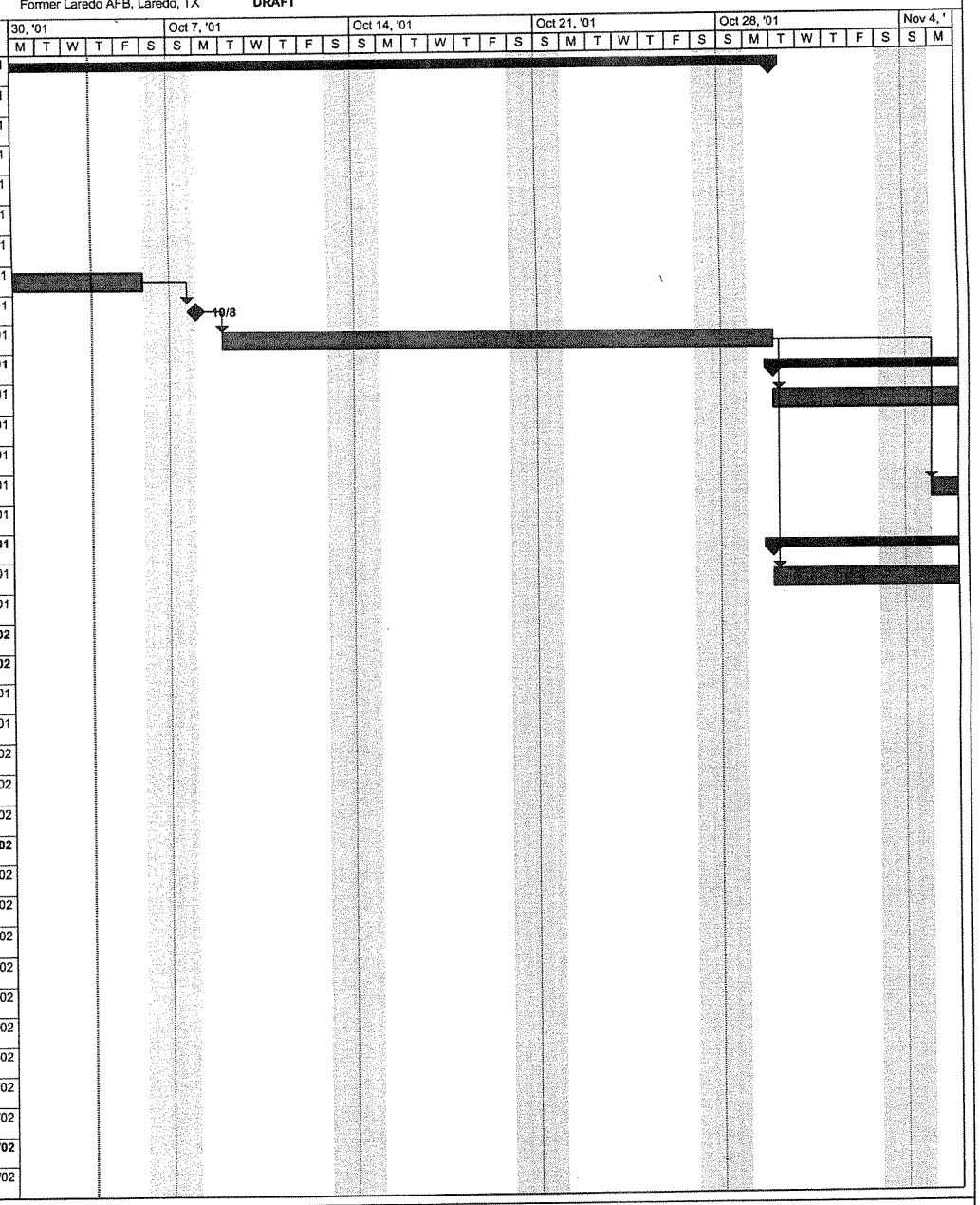

Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

5897

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
39	Submit Draft Technical Memoranda	1 day	Mon 9/16/02	Mon 9/16/02
40	USACE/TNRCC Review of Draft Memoranda	15 days	Tue 9/17/02	Mon 10/7/02
41	Address Comments/Complete Final Technical	14 days	Tue 10/8/02	Fri 10/25/02
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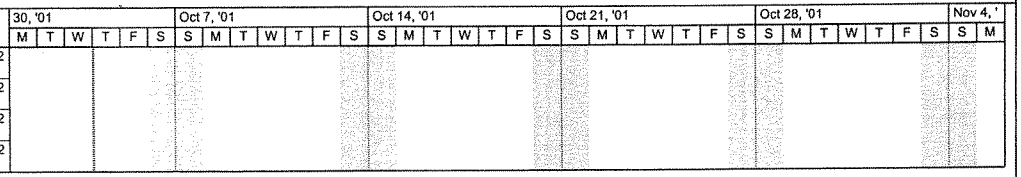

Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

5989

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
1	**Planning Documents**	**96 days**	**Mon 6/18/01**	**Mon 10/29/01**
2	Work Plan	20 days	Mon 6/18/01	Fri 7/13/01
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16	Survey Monitoring Wells	2 days	Mon 11/19/01	Tue 11/20/01
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22	Generate Well/Soil Logs	5 days	Wed 11/21/01	Tue 11/27/01
23	Generate Potentiometric Surface	3 days	Mon 12/17/01	Wed 12/19/01
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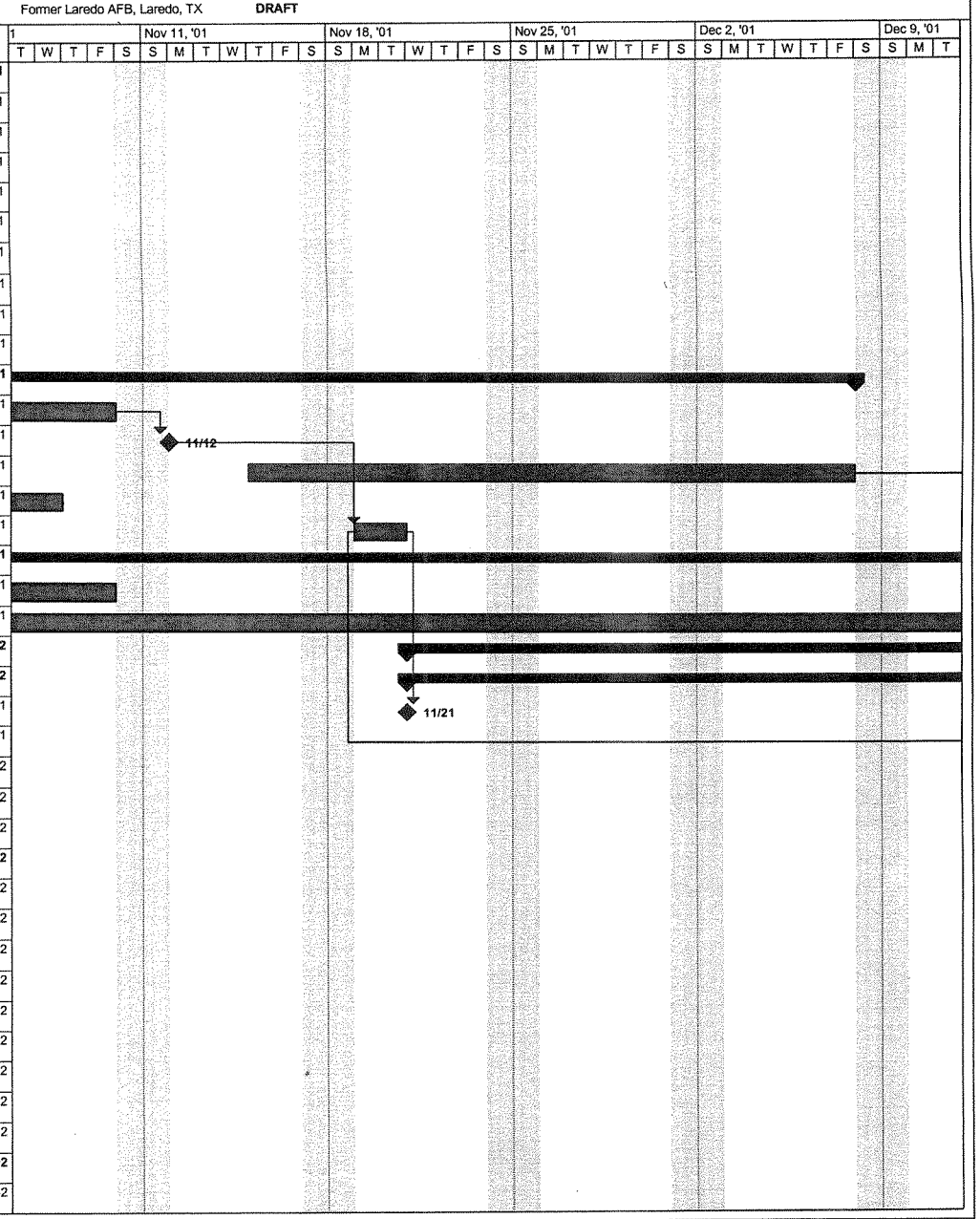

Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

5893

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
39	Submit Draft Technical Memoranda	1 day	Mon 9/16/02	Mon 9/16/02
40	USACE/TNRCC Review of Draft Memoranda	15 days	Tue 9/17/02	Mon 10/7/02
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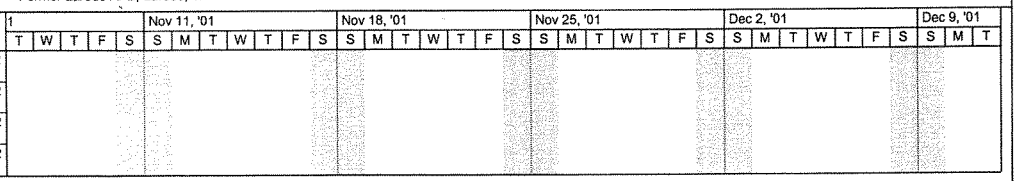

Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

5900

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
1	**Planning Documents**	**96 days**	**Mon 6/18/01**	**Mon 10/29/01**
2	Work Plan	20 days	Mon 6/18/01	Fri 7/13/01
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16	Survey Monitoring Wells	2 days	Mon 11/19/01	Tue 11/20/01
17	**Laboratory Analysis**	**45 days**	**Tue 10/30/01**	**Mon 12/31/01**
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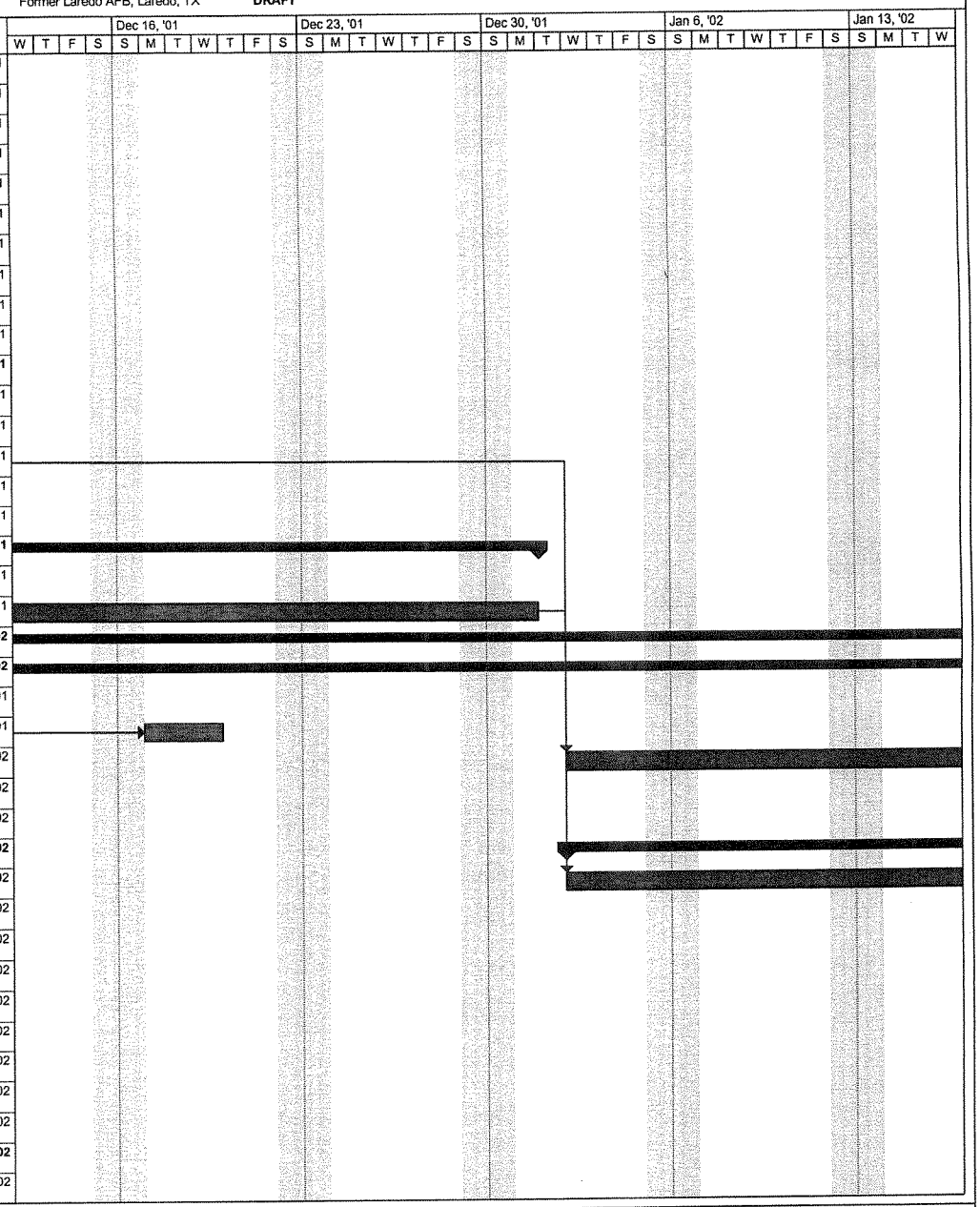

Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

1565

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
39	Submit Draft Technical Memoranda	1 day	Mon 9/16/02	Mon 9/16/02
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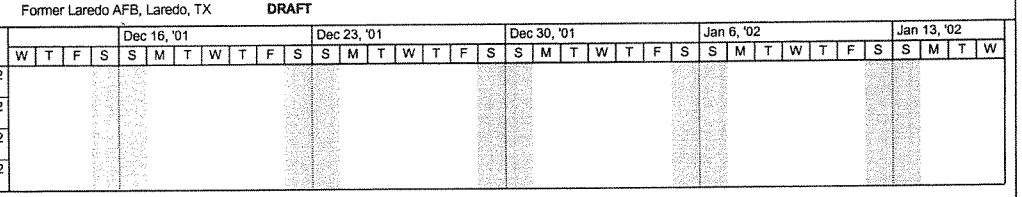

Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

350

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
1	**Planning Documents**	**96 days**	**Mon 6/18/01**	**Mon 10/29/01**
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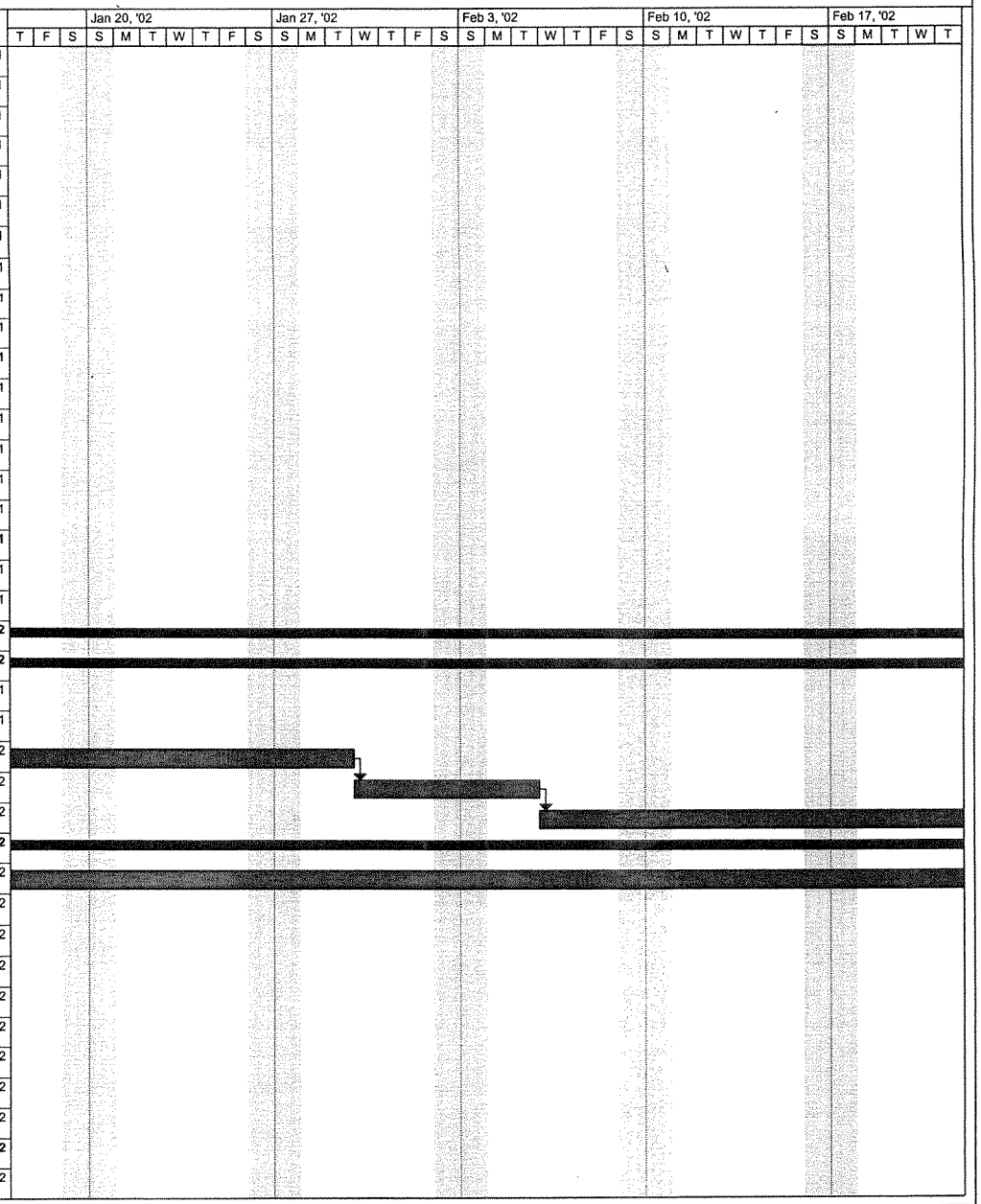

Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

550

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
39	Submit Draft Technical Memoranda	1 day	Mon 9/16/02	Mon 9/16/02
40	USACE/TNRCC Review of Draft Memoranda	15 days	Tue 9/17/02	Mon 10/7/02
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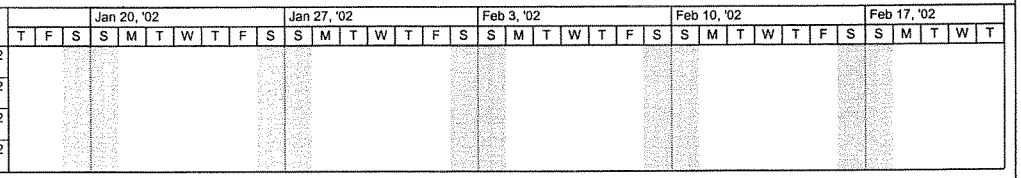

Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

4065

Former Laredo AFB, Laredo, TX **DRAFT**

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1	**Planning Documents**	**96 days**	**Mon 6/18/01**	**Mon 10/29/01**
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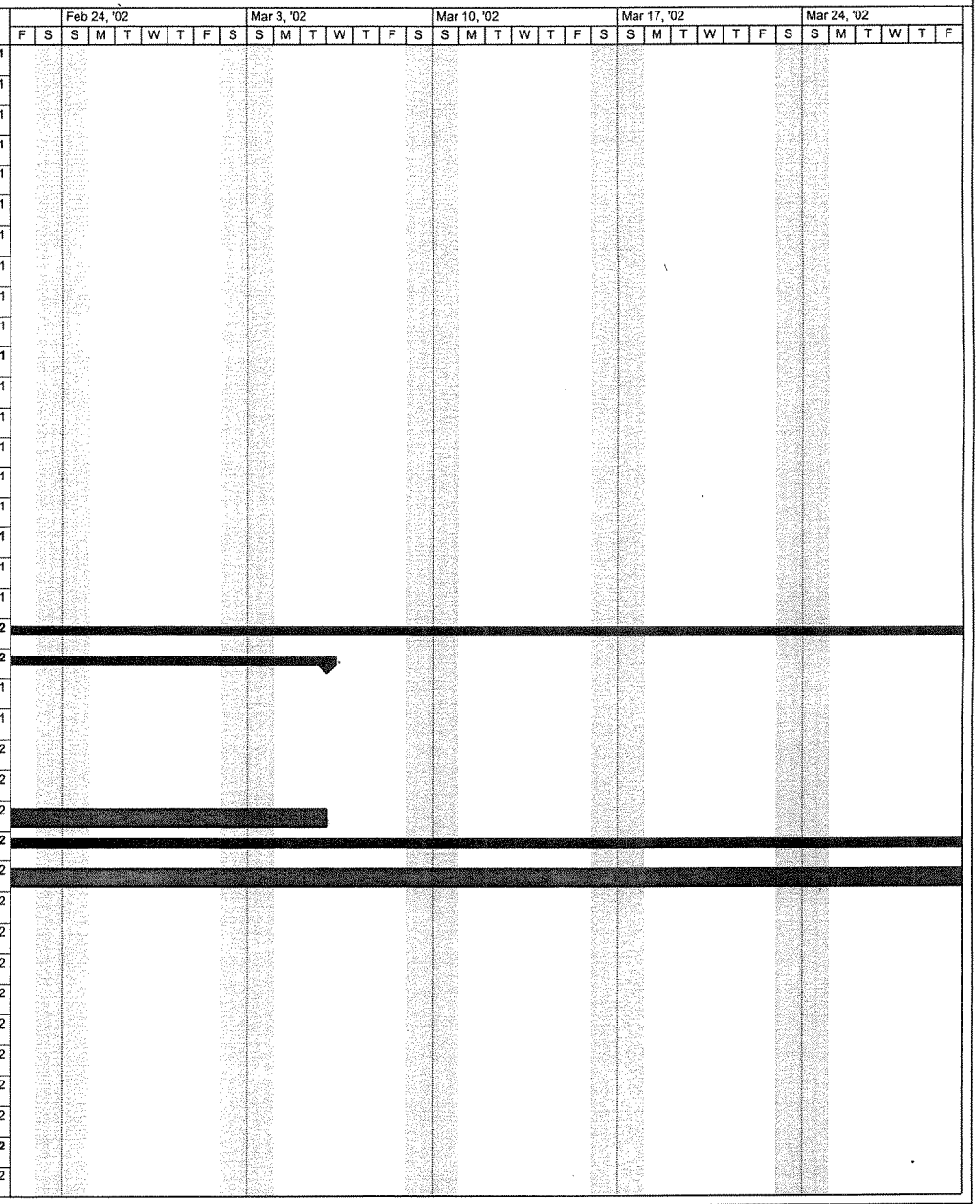
29	Submit Draft Report	1 day	Mon 4/8/02	Mon 4/8/02
30	USACE/TNRCC Review of Draft Report	20 days	Tue 4/9/02	Mon 5/6/02
31	Address Comments/Complete Draft Final Repo	34 days	Tue 5/7/02	Fri 6/21/02
32	Submit Draft Final Report	1 day	Mon 6/24/02	Mon 6/24/02
33	USACE/TNRCC Review of Draft Final Report	15 days	Tue 6/25/02	Mon 7/15/02
34	Address Comments/Complete Final Report	19 days	Tue 7/16/02	Fri 8/9/02
35	Submit Final Report	1 day	Mon 8/12/02	Mon 8/12/02
36	USACE Approval of Final Report	10 days	Tue 8/13/02	Mon 8/26/02
37	**Technical Memoranda**	**45 days**	**Tue 8/27/02**	**Mon 10/28/02**
38	Complete Draft Technical Memoranda	14 days	Tue 8/27/02	Fri 9/13/02

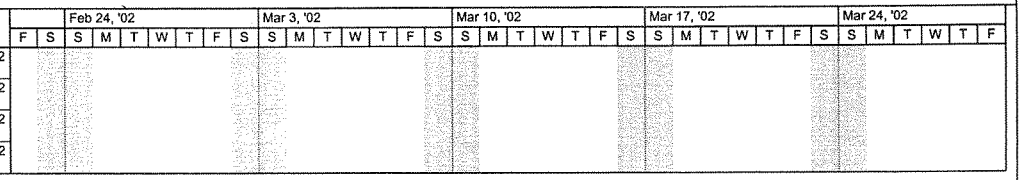


Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

5903

ID	Task Name	Duration	Start	Finish
39	Submit Draft Technical Memoranda	1 day	Mon 9/16/02	Mon 9/16/02
40	USACE/TNRCC Review of Draft Memoranda	15 days	Tue 9/17/02	Mon 10/7/02
41	Address Comments/Complete Final Technical	14 days	Tue 10/8/02	Fri 10/25/02
42	Submit Final Technical Memoranda	1 day	Mon 10/28/02	Mon 10/28/02



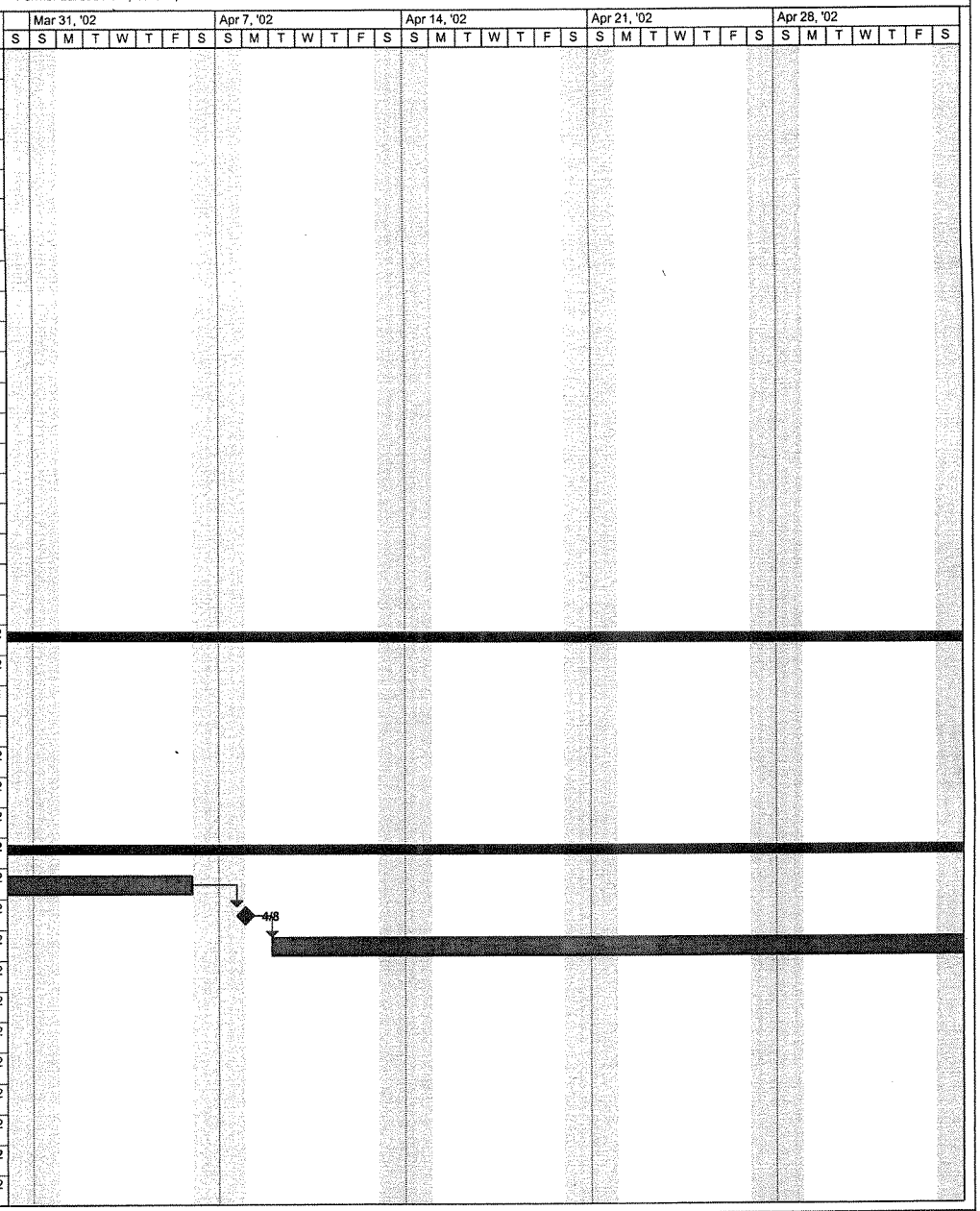
Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

5908

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
1	**Planning Documents**	**96 days**	**Mon 6/18/01**	**Mon 10/29/01**
2	Work Plan	20 days	Mon 6/18/01	Fri 7/13/01
3	Site Safety & Health Plan	20 days	Mon 6/18/01	Fri 7/13/01
4	Quality Assurance Project Plan	20 days	Mon 6/18/01	Fri 7/13/01
5	Field Sampling Plan	22 days	Mon 6/18/01	Tue 7/17/01
6	Submit Draft Planning Documents	1 day	Wed 7/18/01	Wed 7/18/01
7	USACE/TNRCC Review	31 days	Thu 7/19/01	Thu 8/30/01
8	Respond to Comments/Revise Planning Documents	16 days	Fri 9/14/01	Fri 10/5/01
9	Submit Final Planning Documents	1 day	Mon 10/8/01	Mon 10/8/01
10	USACE Approval of Final Planning Documents	15 days	Tue 10/9/01	Mon 10/29/01
11	**Field Activities**	**29 days**	**Tue 10/30/01**	**Fri 12/7/01**
12	Procure Subcontractors	9 days	Tue 10/30/01	Fri 11/9/01
13	Install Monitoring Wells	5 days	Mon 11/12/01	Fri 11/16/01
14	Collect Groundwater Samples	17 days	Thu 11/15/01	Fri 12/7/01
15	Collect Soil Samples	3 days	Mon 11/5/01	Wed 11/7/01
16	Survey Monitoring Wells	2 days	Mon 11/19/01	Tue 11/20/01
17	**Laboratory Analysis**	**45 days**	**Tue 10/30/01**	**Mon 12/31/01**
18	Procure Laboratory	9 days	Tue 10/30/01	Fri 11/9/01
19	Analyze samples	40 days	Tue 11/6/01	Mon 12/31/01
20	**Statistical Analysis and Report**	**244 days**	**Wed 11/21/01**	**Mon 10/28/02**
21	**Data Analysis**	**75 days**	**Wed 11/21/01**	**Tue 3/5/02**
22	Generate Well/Soil Logs	5 days	Wed 11/21/01	Tue 11/27/01
23	Generate Potentiometric Surface	3 days	Mon 12/17/01	Wed 12/19/01
24	QA Laboratory Results	20 days	Wed 1/2/02	Tue 1/29/02
25	Compile Laboratory Data	5 days	Wed 1/30/02	Tue 2/5/02
26	Statistical Analysis	20 days	Wed 2/6/02	Tue 3/5/02
27	**Background Study Report**	**169 days**	**Wed 1/2/02**	**Mon 8/26/02**
28	Complete Draft Report	68 days	Wed 1/2/02	Fri 4/5/02
29	Submit Draft Report	1 day	Mon 4/8/02	Mon 4/8/02
30	USACE/TNRCC Review of Draft Report	20 days	Tue 4/9/02	Mon 5/6/02
31	Address Comments/Complete Draft Final Repo	34 days	Tue 5/7/02	Fri 6/21/02
32	Submit Draft Final Report	1 day	Mon 6/24/02	Mon 6/24/02
33	USACE/TNRCC Review of Draft Final Report	15 days	Tue 6/25/02	Mon 7/15/02
34	Address Comments/Complete Final Report	19 days	Tue 7/16/02	Fri 8/9/02
35	Submit Final Report	1 day	Mon 8/12/02	Mon 8/12/02
36	USACE Approval of Final Report	10 days	Tue 8/13/02	Mon 8/26/02
37	**Technical Memoranda**	**45 days**	**Tue 8/27/02**	**Mon 10/28/02**
38	Complete Draft Technical Memoranda	14 days	Tue 8/27/02	Fri 9/13/02



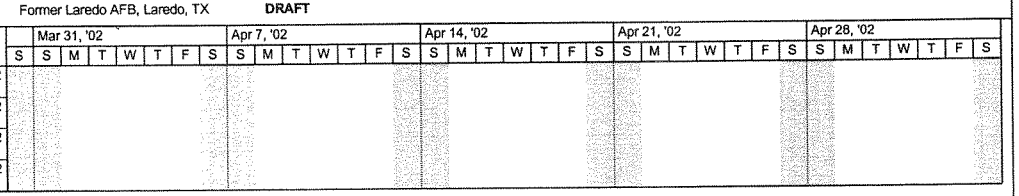
Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

1057

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
39	Submit Draft Technical Memoranda	1 day	Mon 9/16/02	Mon 9/16/02
40	USACE/TNRCC Review of Draft Memoranda	15 days	Tue 9/17/02	Mon 10/7/02
41	Address Comments/Complete Final Technical	14 days	Tue 10/8/02	Fri 10/25/02
42	Submit Final Technical Memoranda	1 day	Mon 10/28/02	Mon 10/28/02



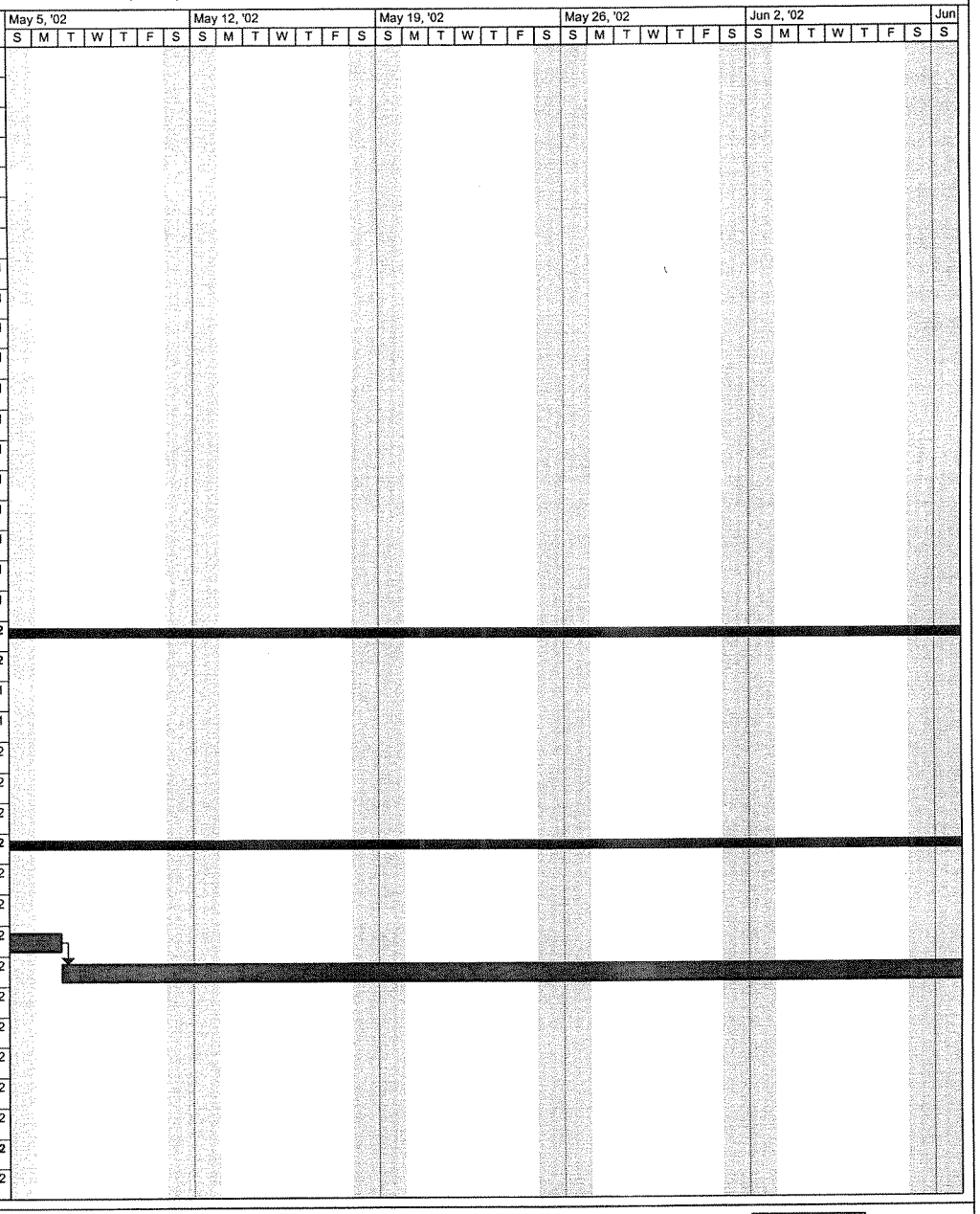
Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

5933

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
1	**Planning Documents**	**96 days**	**Mon 6/18/01**	**Mon 10/29/01**
2	Work Plan	20 days	Mon 6/18/01	Fri 7/13/01
3	Site Safety & Health Plan	20 days	Mon 6/18/01	Fri 7/13/01
4	Quality Assurance Project Plan	20 days	Mon 6/18/01	Fri 7/13/01
5	Field Sampling Plan	22 days	Mon 6/18/01	Tue 7/17/01
6	Submit Draft Planning Documents	1 day	Wed 7/18/01	Wed 7/18/01
7	USACE/TNRCC Review	31 days	Thu 7/19/01	Thu 8/30/01
8	Respond to Comments/Revise Planning Documents	16 days	Fri 9/14/01	Fri 10/5/01
9	Submit Final Planning Documents	1 day	Mon 10/8/01	Mon 10/8/01
10	USACE Approval of Final Planning Documents	15 days	Tue 10/9/01	Mon 10/29/01
11	**Field Activities**	**29 days**	**Tue 10/30/01**	**Fri 12/7/01**
12	Procure Subcontractors	9 days	Tue 10/30/01	Fri 11/9/01
13	Install Monitoring Wells	5 days	Mon 11/12/01	Fri 11/16/01
14	Collect Groundwater Samples	17 days	Thu 11/15/01	Fri 12/7/01
15	Collect Soil Samples	3 days	Mon 11/5/01	Wed 11/7/01
16	Survey Monitoring Wells	2 days	Mon 11/19/01	Tue 11/20/01
17	**Laboratory Analysis**	**45 days**	**Tue 10/30/01**	**Mon 12/31/01**
18	Procure Laboratory	9 days	Tue 10/30/01	Fri 11/9/01
19	Analyze samples	40 days	Tue 11/6/01	Mon 12/31/01
20	**Statistical Analysis and Report**	**244 days**	**Wed 11/21/01**	**Mon 10/28/02**
21	**Data Analysis**	**75 days**	**Wed 11/21/01**	**Tue 3/5/02**
22	Generate Well/Soil Logs	5 days	Wed 11/21/01	Tue 11/27/01
23	Generate Potentiometric Surface	3 days	Mon 12/17/01	Wed 12/19/01
24	QA Laboratory Results	20 days	Wed 1/2/02	Tue 1/29/02
25	Compile Laboratory Data	5 days	Wed 1/30/02	Tue 2/5/02
26	Statistical Analysis	20 days	Wed 2/6/02	Tue 3/5/02
27	**Background Study Report**	**169 days**	**Wed 1/2/02**	**Mon 8/26/02**
28	Complete Draft Report	68 days	Wed 1/2/02	Fri 4/5/02
29	Submit Draft Report	1 day	Mon 4/8/02	Mon 4/8/02
30	USACE/TNRCC Review of Draft Report	20 days	Tue 4/9/02	Mon 5/6/02
31	Address Comments/Complete Draft Final Repo	34 days	Tue 5/7/02	Fri 6/21/02
32	Submit Draft Final Report	1 day	Mon 6/24/02	Mon 6/24/02
33	USACE/TNRCC Review of Draft Final Report	15 days	Tue 6/25/02	Mon 7/15/02
34	Address Comments/Complete Final Report	19 days	Tue 7/16/02	Fri 8/9/02
35	Submit Final Report	1 day	Mon 8/12/02	Mon 8/12/02
36	USACE Approval of Final Report	10 days	Tue 8/13/02	Mon 8/26/02
37	**Technical Memoranda**	**45 days**	**Tue 8/27/02**	**Mon 10/28/02**
38	Complete Draft Technical Memoranda	14 days	Tue 8/27/02	Fri 9/13/02



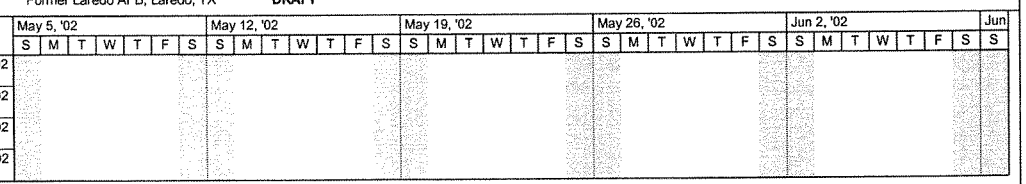
Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

5303

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
39	Submit Draft Technical Memoranda	1 day	Mon 9/16/02	Mon 9/16/02
40	USACE/TNRCC Review of Draft Memoranda	15 days	Tue 9/17/02	Mon 10/7/02
41	Address Comments/Complete Final Technical	14 days	Tue 10/8/02	Fri 10/25/02
42	Submit Final Technical Memoranda	1 day	Mon 10/28/02	Mon 10/28/02



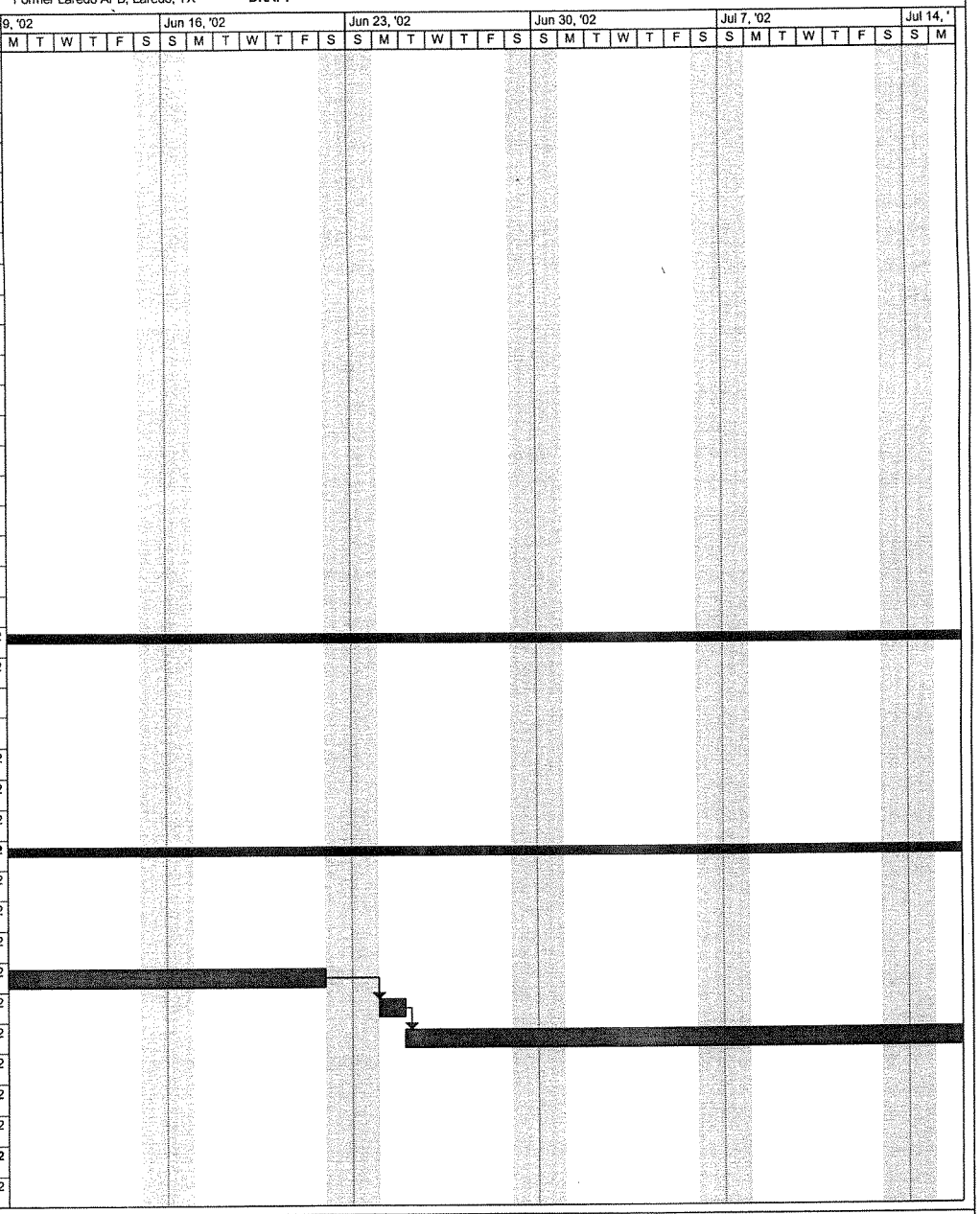
Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Progress | Milestone | Summary | Rolled Up Task | Rolled Up Milestone | Rolled Up Progress | Split | External Tasks | Project Summary

5910

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
1	**Planning Documents**	**96 days**	**Mon 6/18/01**	**Mon 10/29/01**
2	Work Plan	20 days	Mon 6/18/01	Fri 7/13/01
3	Site Safety & Health Plan	20 days	Mon 6/18/01	Fri 7/13/01
4	Quality Assurance Project Plan	20 days	Mon 6/18/01	Fri 7/13/01
5	Field Sampling Plan	22 days	Mon 6/18/01	Tue 7/17/01
6	Submit Draft Planning Documents	1 day	Wed 7/18/01	Wed 7/18/01
7	USACE/TNRCC Review	31 days	Thu 7/19/01	Thu 8/30/01
8	Respond to Comments/Revise Planning Documents	16 days	Fri 9/14/01	Fri 10/5/01
9	Submit Final Planning Documents	1 day	Mon 10/8/01	Mon 10/8/01
10	USACE Approval of Final Planning Documents	15 days	Tue 10/9/01	Mon 10/29/01
11	**Field Activities**	**29 days**	**Tue 10/30/01**	**Fri 12/7/01**
12	Procure Subcontractors	9 days	Tue 10/30/01	Fri 11/9/01
13	Install Monitoring Wells	5 days	Mon 11/12/01	Fri 11/16/01
14	Collect Groundwater Samples	17 days	Thu 11/15/01	Fri 12/7/01
15	Collect Soil Samples	3 days	Mon 11/5/01	Wed 11/7/01
16	Survey Monitoring Wells	2 days	Mon 11/19/01	Tue 11/20/01
17	**Laboratory Analysis**	**45 days**	**Tue 10/30/01**	**Mon 12/31/01**
18	Procure Laboratory	9 days	Tue 10/30/01	Fri 11/9/01
19	Analyze samples	40 days	Tue 11/6/01	Mon 12/31/01
20	**Statistical Analysis and Report**	**244 days**	**Wed 11/21/01**	**Mon 10/28/02**
21	**Data Analysis**	**75 days**	**Wed 11/21/01**	**Tue 3/5/02**
22	Generate Well/Soil Logs	5 days	Wed 11/21/01	Tue 11/27/01
23	Generate Potentiometric Surface	3 days	Mon 12/17/01	Wed 12/19/01
24	QA Laboratory Results	20 days	Wed 1/2/02	Tue 1/29/02
25	Compile Laboratory Data	5 days	Wed 1/30/02	Tue 2/5/02
26	Statistical Analysis	20 days	Wed 2/6/02	Tue 3/5/02
27	**Background Study Report**	**169 days**	**Wed 1/2/02**	**Mon 8/26/02**
28	Complete Draft Report	68 days	Wed 1/2/02	Fri 4/5/02
29	Submit Draft Report	1 day	Mon 4/8/02	Mon 4/8/02
30	USACE/TNRCC Review of Draft Report	20 days	Tue 4/9/02	Mon 5/6/02
31	Address Comments/Complete Draft Final Repo	34 days	Tue 5/7/02	Fri 6/21/02
32	Submit Draft Final Report	1 day	Mon 6/24/02	Mon 6/24/02
33	USACE/TNRCC Review of Draft Final Report	15 days	Tue 6/25/02	Mon 7/15/02
34	Address Comments/Complete Final Report	19 days	Tue 7/16/02	Fri 8/9/02
35	Submit Final Report	1 day	Mon 8/12/02	Mon 8/12/02
36	USACE Approval of Final Report	10 days	Tue 8/13/02	Mon 8/26/02
37	**Technical Memoranda**	**45 days**	**Tue 8/27/02**	**Mon 10/28/02**
38	Complete Draft Technical Memoranda	14 days	Tue 8/27/02	Fri 9/13/02



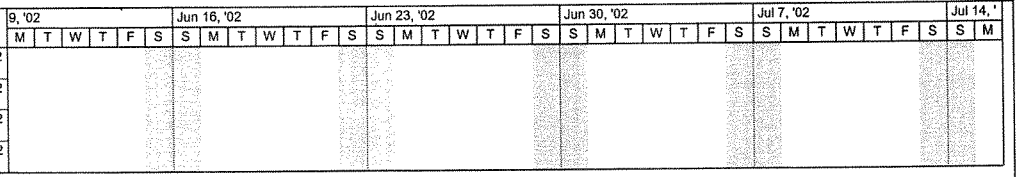
Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Progress | Milestone | Summary | Rolled Up Task | Rolled Up Milestone | Rolled Up Progress | Split | External Tasks | Project Summary

5911

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
39	Submit Draft Technical Memoranda	1 day	Mon 9/16/02	Mon 9/16/02
40	USACE/TNRCC Review of Draft Memoranda	15 days	Tue 9/17/02	Mon 10/7/02
41	Address Comments/Complete Final Technical	14 days	Tue 10/8/02	Fri 10/25/02
42	Submit Final Technical Memoranda	1 day	Mon 10/28/02	Mon 10/28/02



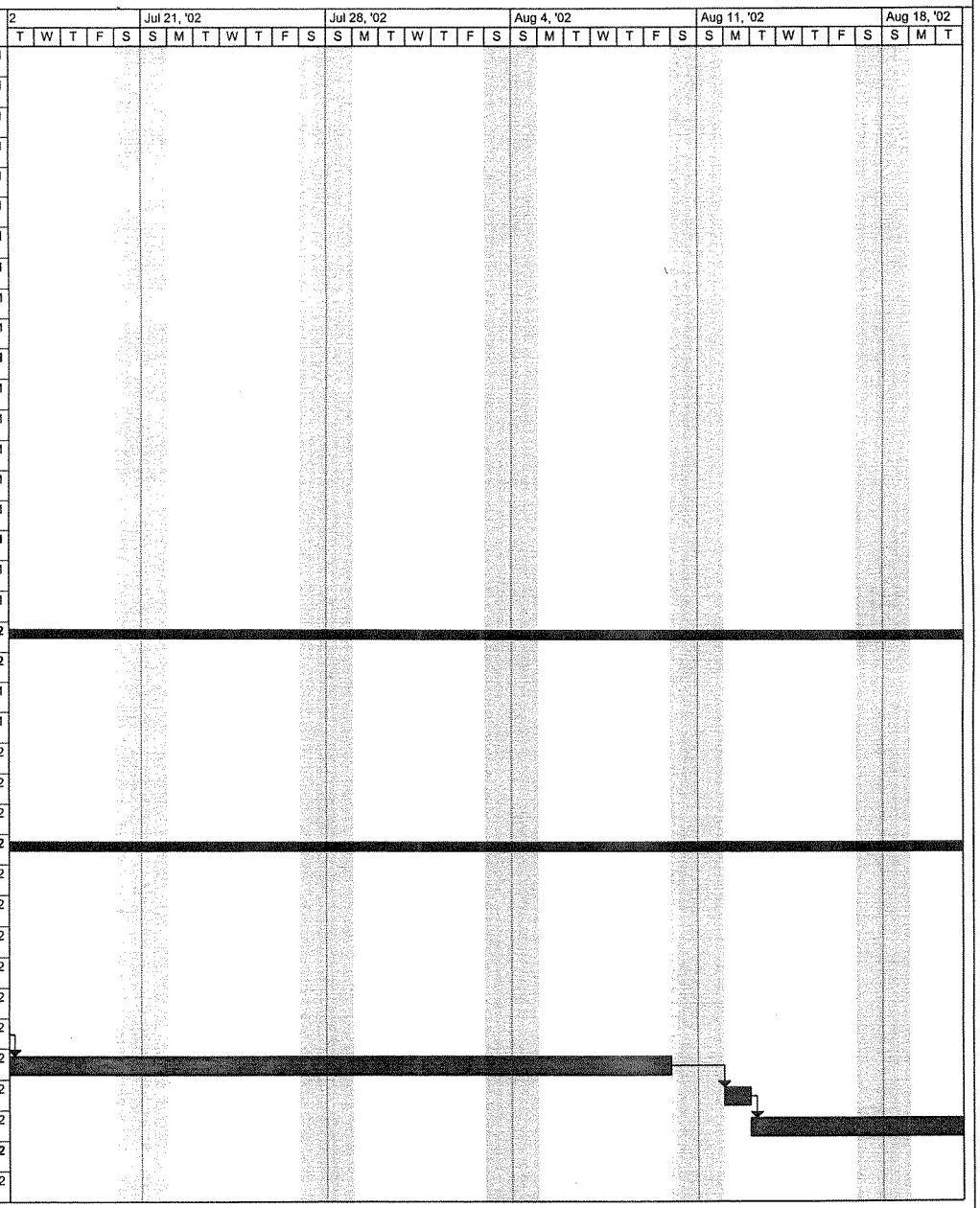
Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

5912

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
1	**Planning Documents**	**96 days**	**Mon 6/18/01**	**Mon 10/29/01**
2	Work Plan	20 days	Mon 6/18/01	Fri 7/13/01
3	Site Safety & Health Plan	20 days	Mon 6/18/01	Fri 7/13/01
4	Quality Assurance Project Plan	20 days	Mon 6/18/01	Fri 7/13/01
5	Field Sampling Plan	22 days	Mon 6/18/01	Tue 7/17/01
6	Submit Draft Planning Documents	1 day	Wed 7/18/01	Wed 7/18/01
7	USACE/TNRCC Review	31 days	Thu 7/19/01	Thu 8/30/01
8	Respond to Comments/Revise Planning Documents	16 days	Fri 9/14/01	Fri 10/5/01
9	Submit Final Planning Documents	1 day	Mon 10/8/01	Mon 10/8/01
10	USACE Approval of Final Planning Documents	15 days	Tue 10/9/01	Mon 10/29/01
11	**Field Activities**	**29 days**	**Tue 10/30/01**	**Fri 12/7/01**
12	Procure Subcontractors	9 days	Tue 10/30/01	Fri 11/9/01
13	Install Monitoring Wells	5 days	Mon 11/12/01	Fri 11/16/01
14	Collect Groundwater Samples	17 days	Thu 11/15/01	Fri 12/7/01
15	Collect Soil Samples	3 days	Mon 11/5/01	Wed 11/7/01
16	Survey Monitoring Wells	2 days	Mon 11/19/01	Tue 11/20/01
17	**Laboratory Analysis**	**45 days**	**Tue 10/30/01**	**Mon 12/31/01**
18	Procure Laboratory	9 days	Tue 10/30/01	Fri 11/9/01
19	Analyze samples	40 days	Tue 11/6/01	Mon 12/31/01
20	**Statistical Analysis and Report**	**244 days**	**Wed 11/21/01**	**Mon 10/28/02**
21	**Data Analysis**	**75 days**	**Wed 11/21/01**	**Tue 3/5/02**
22	Generate Well/Soil Logs	5 days	Wed 11/21/01	Tue 11/27/01
23	Generate Potentiometric Surface	3 days	Mon 12/17/01	Wed 12/19/01
24	QA Laboratory Results	20 days	Wed 1/2/02	Tue 1/29/02
25	Compile Laboratory Data	5 days	Wed 1/30/02	Tue 2/5/02
26	Statistical Analysis	20 days	Wed 2/6/02	Tue 3/5/02
27	**Background Study Report**	**169 days**	**Wed 1/2/02**	**Mon 8/26/02**
28	Complete Draft Report	68 days	Wed 1/2/02	Fri 4/5/02
29	Submit Draft Report	1 day	Mon 4/8/02	Mon 4/8/02
30	USACE/TNRCC Review of Draft Report	20 days	Tue 4/9/02	Mon 5/6/02
31	Address Comments/Complete Draft Final Repo	34 days	Tue 5/7/02	Fri 6/21/02
32	Submit Draft Final Report	1 day	Mon 6/24/02	Mon 6/24/02
33	USACE/TNRCC Review of Draft Final Report	15 days	Tue 6/25/02	Mon 7/15/02
34	Address Comments/Complete Final Report	19 days	Tue 7/16/02	Fri 8/9/02
35	Submit Final Report	1 day	Mon 8/12/02	Mon 8/12/02
36	USACE Approval of Final Report	10 days	Tue 8/13/02	Mon 8/26/02
37	**Technical Memoranda**	**45 days**	**Tue 8/27/02**	**Mon 10/28/02**
38	Complete Draft Technical Memoranda	14 days	Tue 8/27/02	Fri 9/13/02



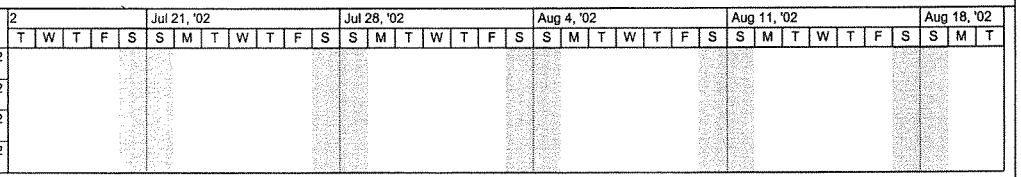
Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Progress | Milestone | Summary | Rolled Up Task | Rolled Up Milestone | Rolled Up Progress | Split | External Tasks | Project Summary

5913

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
39	Submit Draft Technical Memoranda	1 day	Mon 9/16/02	Mon 9/16/02
40	USACE/TNRCC Review of Draft Memoranda	15 days	Tue 9/17/02	Mon 10/7/02
41	Address Comments/Complete Final Technical	14 days	Tue 10/8/02	Fri 10/25/02
42	Submit Final Technical Memoranda	1 day	Mon 10/28/02	Mon 10/28/02



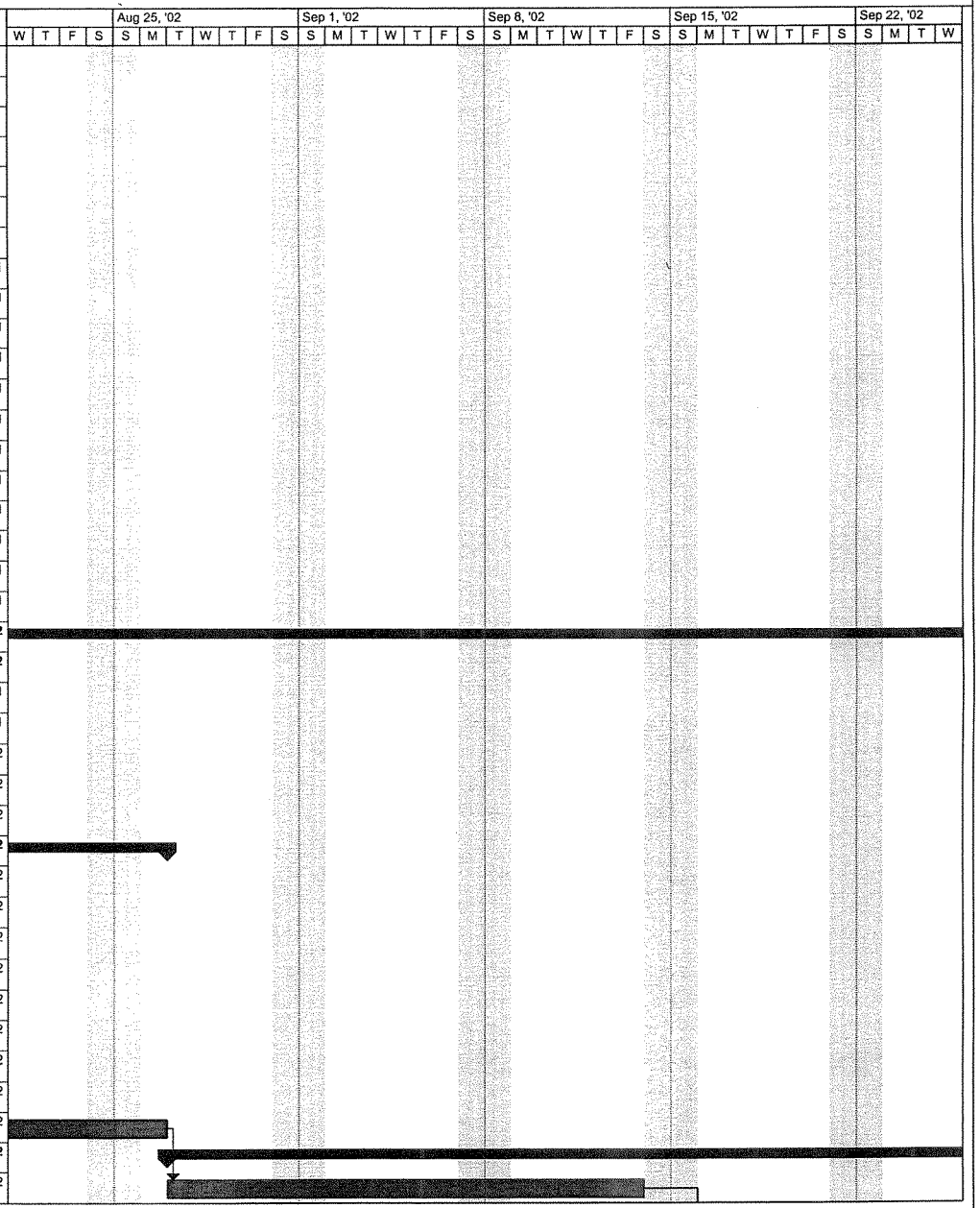
Project: Former Laredo AFB
Date: Thu 10/4/01

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

5914

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
1	**Planning Documents**	**96 days**	**Mon 6/18/01**	**Mon 10/29/01**
2	Work Plan	20 days	Mon 6/18/01	Fri 7/13/01
3	Site Safety & Health Plan	20 days	Mon 6/18/01	Fri 7/13/01
4	Quality Assurance Project Plan	20 days	Mon 6/18/01	Fri 7/13/01
5	Field Sampling Plan	22 days	Mon 6/18/01	Tue 7/17/01
6	Submit Draft Planning Documents	1 day	Wed 7/18/01	Wed 7/18/01
7	USACE/TNRCC Review	31 days	Thu 7/19/01	Thu 8/30/01
8	Respond to Comments/Revise Planning Documents	16 days	Fri 9/14/01	Fri 10/5/01
9	Submit Final Planning Documents	1 day	Mon 10/8/01	Mon 10/8/01
10	USACE Approval of Final Planning Documents	15 days	Tue 10/9/01	Mon 10/29/01
11	**Field Activities**	**29 days**	**Tue 10/30/01**	**Fri 12/7/01**
12	Procure Subcontractors	9 days	Tue 10/30/01	Fri 11/9/01
13	Install Monitoring Wells	5 days	Mon 11/12/01	Fri 11/16/01
14	Collect Groundwater Samples	17 days	Thu 11/15/01	Fri 12/7/01
15	Collect Soil Samples	3 days	Mon 11/5/01	Wed 11/7/01
16	Survey Monitoring Wells	2 days	Mon 11/19/01	Tue 11/20/01
17	**Laboratory Analysis**	**45 days**	**Tue 10/30/01**	**Mon 12/31/01**
18	Procure Laboratory	9 days	Tue 10/30/01	Fri 11/9/01
19	Analyze samples	40 days	Tue 11/6/01	Mon 12/31/01
20	**Statistical Analysis and Report**	**244 days**	**Wed 11/21/01**	**Mon 10/28/02**
21	**Data Analysis**	**75 days**	**Wed 11/21/01**	**Tue 3/5/02**
22	Generate Well/Soil Logs	5 days	Wed 11/21/01	Tue 11/27/01
23	Generate Potentiometric Surface	3 days	Mon 12/17/01	Wed 12/19/01
24	QA Laboratory Results	20 days	Wed 1/2/02	Tue 1/29/02
25	Compile Laboratory Data	5 days	Wed 1/30/02	Tue 2/5/02
26	Statistical Analysis	20 days	Wed 2/6/02	Tue 3/5/02
27	**Background Study Report**	**169 days**	**Wed 1/2/02**	**Mon 8/26/02**
28	Complete Draft Report	68 days	Wed 1/2/02	Fri 4/5/02
29	Submit Draft Report	1 day	Mon 4/8/02	Mon 4/8/02
30	USACE/TNRCC Review of Draft Report	20 days	Tue 4/9/02	Mon 5/6/02
31	Address Comments/Complete Draft Final Repo	34 days	Tue 5/7/02	Fri 6/21/02
32	Submit Draft Final Report	1 day	Mon 6/24/02	Mon 6/24/02
33	USACE/TNRCC Review of Draft Final Report	15 days	Tue 6/25/02	Mon 7/15/02
34	Address Comments/Complete Final Report	19 days	Tue 7/16/02	Fri 8/9/02
35	Submit Final Report	1 day	Mon 8/12/02	Mon 8/12/02
36	USACE Approval of Final Report	10 days	Tue 8/13/02	Mon 8/26/02
37	**Technical Memoranda**	**45 days**	**Tue 8/27/02**	**Mon 10/28/02**
38	Complete Draft Technical Memoranda	14 days	Tue 8/27/02	Fri 9/13/02



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Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks
Progress | Summary | Rolled Up Milestone | Split | Project Summary

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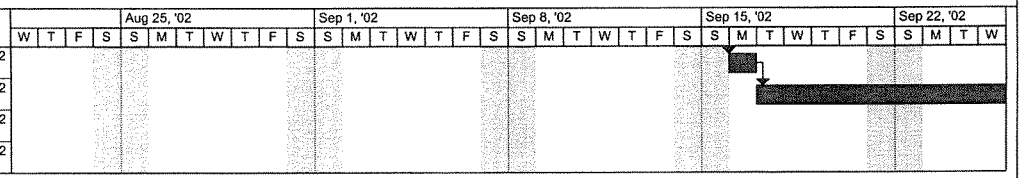
Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
39	Submit Draft Technical Memoranda	1 day	Mon 9/16/02	Mon 9/16/02
40	USACE/TNRCC Review of Draft Memoranda	15 days	Tue 9/17/02	Mon 10/7/02
41	Address Comments/Complete Final Technical	14 days	Tue 10/8/02	Fri 10/25/02
42	Submit Final Technical Memoranda	1 day	Mon 10/28/02	Mon 10/28/02



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Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
1	**Planning Documents**	**96 days**	**Mon 6/18/01**	**Mon 10/29/01**
2	Work Plan	20 days	Mon 6/18/01	Fri 7/13/01
3	Site Safety & Health Plan	20 days	Mon 6/18/01	Fri 7/13/01
4	Quality Assurance Project Plan	20 days	Mon 6/18/01	Fri 7/13/01
5	Field Sampling Plan	22 days	Mon 6/18/01	Tue 7/17/01
6	Submit Draft Planning Documents	1 day	Wed 7/18/01	Wed 7/18/01
7	USACE/TNRCC Review	31 days	Thu 7/19/01	Thu 8/30/01
8	Respond to Comments/Revise Planning Documents	16 days	Fri 9/14/01	Fri 10/5/01
9	Submit Final Planning Documents	1 day	Mon 10/8/01	Mon 10/8/01
10	USACE Approval of Final Planning Documents	15 days	Tue 10/9/01	Mon 10/29/01
11	**Field Activities**	**29 days**	**Tue 10/30/01**	**Fri 12/7/01**
12	Procure Subcontractors	9 days	Tue 10/30/01	Fri 11/9/01
13	Install Monitoring Wells	5 days	Mon 11/12/01	Fri 11/16/01
14	Collect Groundwater Samples	17 days	Thu 11/15/01	Fri 12/7/01
15	Collect Soil Samples	3 days	Mon 11/5/01	Wed 11/7/01
16	Survey Monitoring Wells	2 days	Mon 11/19/01	Tue 11/20/01
17	**Laboratory Analysis**	**45 days**	**Tue 10/30/01**	**Mon 12/31/01**
18	Procure Laboratory	9 days	Tue 10/30/01	Fri 11/9/01
19	Analyze samples	40 days	Tue 11/6/01	Mon 12/31/01
20	**Statistical Analysis and Report**	**244 days**	**Wed 11/21/01**	**Mon 10/28/02**
21	**Data Analysis**	**75 days**	**Wed 11/21/01**	**Tue 3/5/02**
22	Generate Well/Soil Logs	5 days	Wed 11/21/01	Tue 11/27/01
23	Generate Potentiometric Surface	3 days	Mon 12/17/01	Wed 12/19/01
24	QA Laboratory Results	20 days	Wed 1/2/02	Tue 1/29/02
25	Compile Laboratory Data	5 days	Wed 1/30/02	Tue 2/5/02
26	Statistical Analysis	20 days	Wed 2/6/02	Tue 3/5/02
27	**Background Study Report**	**169 days**	**Wed 1/2/02**	**Mon 8/26/02**
28	Complete Draft Report	68 days	Wed 1/2/02	Fri 4/5/02
29	Submit Draft Report	1 day	Mon 4/8/02	Mon 4/8/02
30	USACE/TNRCC Review of Draft Report	20 days	Tue 4/9/02	Mon 5/6/02
31	Address Comments/Complete Draft Final Repo	34 days	Tue 5/7/02	Fri 6/21/02
32	Submit Draft Final Report	1 day	Mon 6/24/02	Mon 6/24/02
33	USACE/TNRCC Review of Draft Final Report	15 days	Tue 6/25/02	Mon 7/15/02
34	Address Comments/Complete Final Report	19 days	Tue 7/16/02	Fri 8/9/02
35	Submit Final Report	1 day	Mon 8/12/02	Mon 8/12/02
36	USACE Approval of Final Report	10 days	Tue 8/13/02	Mon 8/26/02
37	**Technical Memoranda**	**45 days**	**Tue 8/27/02**	**Mon 10/28/02**
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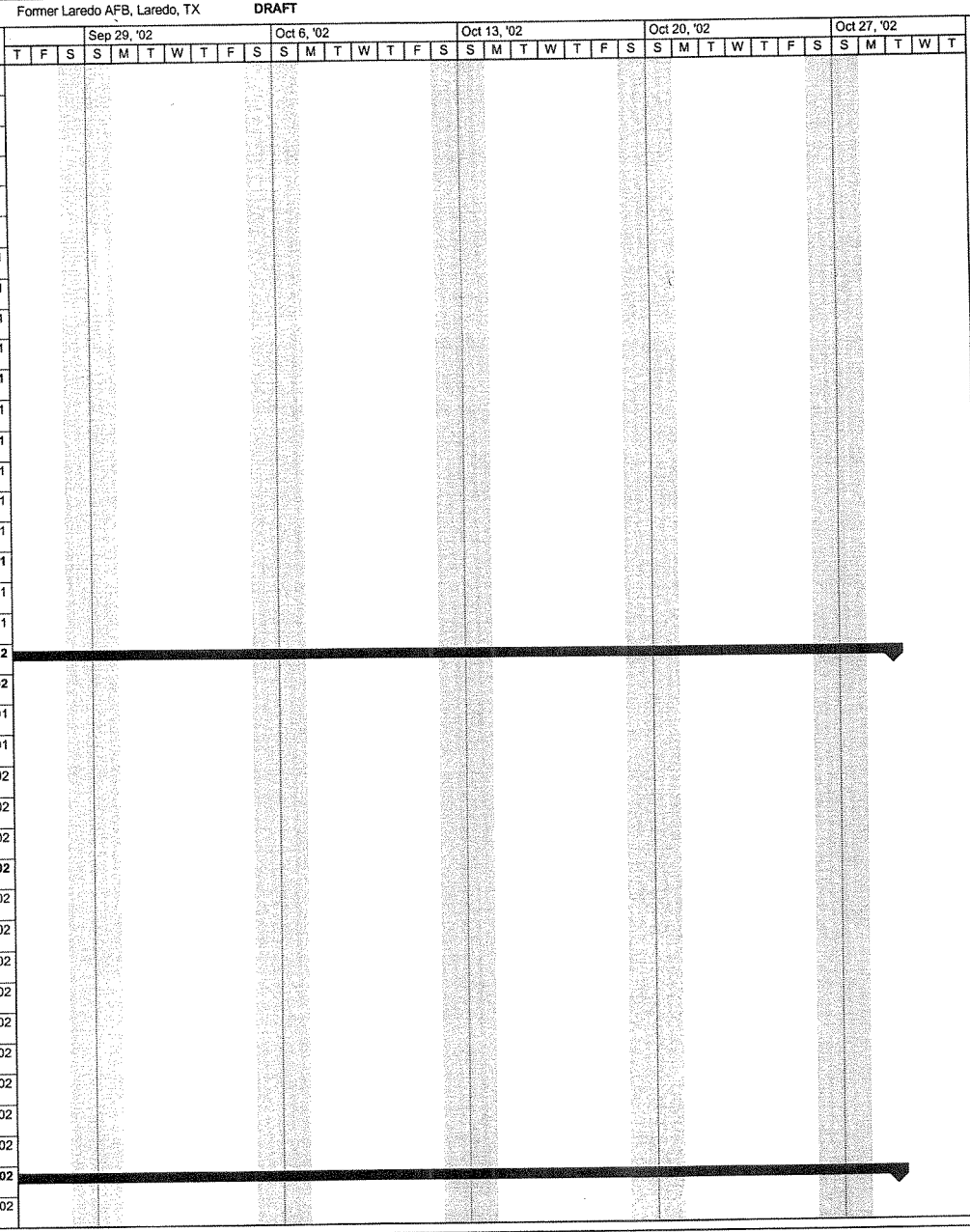

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Task | Progress | Milestone | Summary | Rolled Up Task | Rolled Up Milestone | Rolled Up Progress | Split | External Tasks | Project Summary

Former Laredo AFB, Laredo, TX **DRAFT**

ID	Task Name	Duration	Start	Finish
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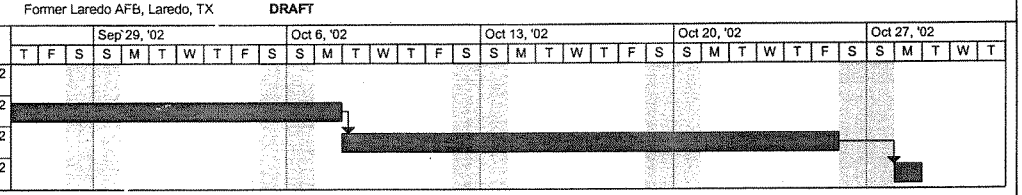

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Progress | Summary | Rolled Up Milestone | Split | Project Summary

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