Kerr-McGee Chemical, LLC
Oklahoma City, Oklahoma

Quality Assurance Project Plan for Installation of Groundwater Remedial System

Moss-American Site
Milwaukee, Wisconsin

23 June 1999

WESTON®
MANAGERS DESIGNERS/CONSULTANTS

QUALITY ASSURANCE PROJECT PLAN FOR INSTALLATION OF GROUNDWATER REMEDIAL SYSTEM MOSS-AMERICAN SITE MILWAUKEE, WISCONSIN

Prepared for

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Oklahoma City, Oklahoma

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QUALITY ASSURANCE PROJECT PLAN
FOR INSTALLATION OF GROUNDWATER REMEDIAL SYSTEM
MOSS-AMERICAN SITE
MILWAUKEE, WISCONSIN

June 1999

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LIST OF ACRONYMS/ABBREVIATIONS

bgs	below ground surface
BTEX	benzene, toluene, ethylbenzene, and total xylenes
CD	consent decree
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act (Superfund)
CPAHs	carcinogenic polynuclear aromatic hydrocarbons
COC	chain-of-custody
CRL	Central Regional Laboratory
cy	cubic yards
DQO	Data Quality Objective
DOT	Department of Transportation
FSM	Field Sample Manager/Custodian
FSP	field sampling plan
FSS	Field Services Section
FTL	field team leader
GC/MS	Gas Chromatograph/Mass Spectrometer
IATA	International Air Transport Association
IDW	Investigation-Derived Waste
KMC	Kerr-McGee Chemical LLC
MMSD	Milwaukee Metropolitan Sewerage District
MS/MSD	matrix spike/matrix spike duplicate
NEIC	National Enforcement Investigations Center
NPL	National Priorities List
PAH	polynuclear hydrocarbon
PE	performance evaluation
PID	photoionization detector
ppb	parts per billion
PPE	personal protective equipment
QA	quality assurance
QAPP	quality assurance project plan
QA/QC	quality assurance/quality control
QC	quality control
RCL	residual contaminant level
RI	remedial investigation
ROD	record of decision
RPD	relative percent difference
RPM	Remedial Project Manager
SHSC	Site Health and Safety Coordinator
SMC	sample management coordinator
SOP	standard operating procedure
SOW	Statement of Work
SVOC	semivolatile organic compound
TSS	total suspended solids
U.S. EPA	United States Environmental Protection Agency

LIST OF ACRONYMS/ABBREVIATIONS (Continued)

VOA	volatile organic analysis
VOC	volatile organic compound
WESTON	Roy F. Weston, Inc.

SECTION 1
INTRODUCTION

This QAPP has been prepared by Roy F. Weston, Inc. (WESTON®), on behalf of Kerr-McGee Chemical, LLC (KMC) for use during the construction of the groundwater remedial system at the Moss-American Site in Milwaukee, Wisconsin. The United States Environmental Protection Agency (U.S. EPA) requires that all environmental monitoring and measurement efforts mandated or supported by U.S. EPA participate in a centrally-managed quality assurance (QA) program. Any party generating data under this program has the responsibility to implement procedures to ensure that the precision, accuracy, completeness, and representativeness of its data are known and documented. To ensure the responsibility is met uniformly, each party must prepare a written Quality Assurance Project Plan (QAPP) covering each project it is to perform.

This QAPP presents the organization, objectives, functional activities, and specific Quality Assurance and Quality Control (QA/QC) activities associated with the implementation of the funnel-and-gate groundwater remedial system. This QAPP describes the specific protocols that will be followed for sampling, sample handling and storage, chain-of-custody (COC), and laboratory and field analysis for soil characterization and verification sampling as well as for water generated during the construction activities.

All QA/QC procedures will be in accordance with applicable professional technical standards, U.S. EPA, government regulations and guidelines, and specific project goals and requirements. This QAPP has been prepared in accordance with all U.S. EPA QAPP guidance documents; in particular, the *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans* (QAMS-005/80), *Region V Content Requirements for QAPPs* (U.S. EPA, 1989), the *U.S. EPA Region V Model QAPP* (U.S. EPA, Revision 1, May 1996) and the *U.S. EPA Requirements for QAPPs for Environmental Data Operation* (EPA QA/R-5).

SECTION 2
PROJECT DESCRIPTION

2.1 SITE BACKGROUND

The Moss-American site is the location of a former wood-preserving facility that treated railroad ties with a creosote and fuel oil mixture. The facility was operated from 1921 to 1976 and was closed after being acquired by KMC.

The U.S. EPA, pursuant to Section 105 of the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA), placed the Moss-American site on the National Priorities List (NPL) in 1983. The U.S. EPA conducted a remedial investigation/ feasibility study (RI/FS) for the Moss-American site and issued the corresponding RI/FS report in 1990.

A Consent Decree (CD) incorporating the Statement of Work (SOW) was signed by KMC in 1991. This CD directs KMC to lead in developing and implementing the remedial design and remedial action plan for the Moss-American site. KMC implemented a pre-design phase to further evaluate site conditions and to complete various engineering studies before remedial design/remedial action (RD/RA) was initiated.

In 1995, KMC initiated groundwater and soil remediation at the site by designing, installing, and operating a free product recovery and removal system. The system extracts free phase creosote from subsurface soil and groundwater. Additional free product will be treated through excavation and thermal treatment of the source soil. The free product recovery and removal system will be dismantled and removed prior to the implementation of both the soil and groundwater remedies. Some components of the system may be salvaged and utilized as part of the groundwater remedy.

2.2 SITE LOCATION

The Moss-American site is located in the northwestern section of the City of Milwaukee, County of Milwaukee, State of Wisconsin, at the southeast corner of the intersection of Brown Deer and Granville Roads, at 8716 Granville Road. The Moss-American site was defined in the CD as consisting of the following areas:

- The former Moss-American wood-preserving plant site.
- Approximately 5 miles of the Little Menomonee River.
- Portions of the Little Menomonee floodplain.

The Little Menomonee River flows through the eastern portion of the former wood-preserving plant site and continues through the Milwaukee County Parkway to its confluence with the Menomonee River, about 5 miles south of the site. Milwaukee County owns 51 acres of the former wood-preserving plant site, this parcel is undeveloped, recreational-use, park land. Most of the County property is located in the 100-year floodplain of the Little Menomonee River. Union Pacific, formerly Chicago and Northwestern Transportation Company, owns 23 acres of the site. Union Pacific uses its parcel as a loading and storage area for automobile transport (industrial use).

The site is located in a moderately populated suburban area of mixed light industrial, commercial, residential, and recreational uses. Population in the nearby area is estimated to be approximately 2,036 persons per square mile. A general location map of the site is presented as Figure 2-1.

2.3 SITE CONDITIONS

2.3.1 Geological and Hydrogeological Conditions

To assist the design of remedial measures, WESTON has evaluated all available stratigraphic and hydrogeological data collected during the RI, the remedial pre-design investigation, and all subsequent related investigations. This evaluation has been used to characterize the depositional

history of the site and the stratigraphy that has been encountered. The results of WESTON's evaluation are presented in the following subsections.

2.3.1.1 Geological Conditions

The surficial and near-surface soil encountered at the site varies depending on location, and consists of soil deposited by man-made, lacustrine, fluvial, and glacial processes. In the vicinity of former wood-preserving plant's main process area, now occupied by the Union Pacific automobile transport operation, gravel and asphalt cover the ground surface. East of this area and west of the Little Menomonee River, varying thicknesses of clay, aggregate, topsoil, and natural vegetation cover the ground surface.

Throughout much of the area on the west side of the river, the surficial soil is underlain by man-made fill deposits that range from 2 to 5 feet thick. This fill typically consists of gravel, cinders, wood chips, railroad ties, and other miscellaneous debris.

Deposits that are derived from lacustrine, fluvial, and glacial processes underlie the fill material. These deposits range in thickness from approximately 13 feet on the western, topographically higher portions of the site to approximately 9 feet on the topographically lower portions of the site, nearer to the river (Little Menomonee floodplain). These deposits become even thinner (approximately 5 feet thick) in the immediate vicinity of the river, where the underlying glacial till occurs at a higher elevation and the river channel has eroded through these deposits. It is this sequence of deposits that comprise the shallow groundwater-bearing zone.

The occurrence and thickness of each type of deposit within the groundwater-bearing zone is dependent on depth and distance from the river. In the topographically higher portions of the site, these deposits range from low to moderate permeability silt, silty clay, and sandy silt to moderate to high permeability sand and silty sand. These deposits have been interpreted as being the result

of the erosion and subsequent re-deposition of glacial till by lacustrine processes (reworked glacial till).

Nearer to the river, the deposits within the groundwater-bearing zone consist of the low to moderate permeability sediments described above and more permeable deposits consisting of sand, silt, sand and gravel, and silty sand. While the low to moderate permeability sediments appear to have been deposited as described above, the underlying, more permeable sediments have been interpreted to be the result of overbank flood deposition and alluvial channel deposition.

The depositional history for these sediments can be further defined by interpreting their occurrence and relative depth. Specifically, ascending from the river floodplain to the topographically higher portions of the site, there appears to be at least two areas where the high permeability deposits seem to be associated with drainage channels. The high permeability deposits resulted from erosion of the glacial till surface and subsequent deposition by post-glacial surface water drainage. However, on the river floodplain these deposits have been interpreted as being the result of alluvial channel deposition that may be associated with periods of higher, post-glacial flow within the Little Menomonee River.

The deposits that comprise the groundwater-bearing zone are underlain by a dense, very fine-grained, silty clay glacial till. Based on the results of geotechnical tests completed on soil samples collected from within the glacial till deposit, this is a highly impermeable sediment that has an average vertical hydraulic conductivity of 2×10^{-7} centimeters per second (cm/s). Based on the measured geotechnical parameters and observations made during the RI, the glacial till acts as an impermeable barrier that separates the shallow groundwater-bearing zone from thin groundwater-bearing layers of sandy sediment that were encountered within the glacial till at depths ranging from approximately 20 to 40 feet below the surface of the glacial till deposit.

2.3.1.2 Hydrogeological Conditions

As previously discussed, the available data indicates that the shallow groundwater-bearing zone that underlies the site occurs within low to high permeability lacustrine and alluvial deposits that range in thickness from approximately 5 to 13 feet, and that this zone occurs on top of a dense, impermeable, glacial till. As a result of the impermeable glacial till, groundwater flow within the shallow zone is restricted to this zone and is controlled by site topography. Groundwater elevation measurements collected in the shallow groundwater-bearing zone since 1988 indicate that the flow of the shallow groundwater on the western side of the Little Menomonee River is consistently eastward, from the topographically higher portions of the site towards the river. This indicates that under current-day surface water and groundwater flow conditions, groundwater consistently flows from the site and discharges to the river; therefore, the Little Menomonee River is a gaining river.

Slug tests completed on intermediate and deep groundwater monitoring wells installed during the RI indicate that the deeper groundwater-bearing zones occurring within the impermeable glacial till have hydraulic conductivities ranging from 1×10^{-5} to 1×10^{-6} cm/s. Because of the discontinuous occurrence of these deeper groundwater-bearing layers, the relatively thin occurrence of these beds (maximum thickness of approximately 5 feet), and the low hydraulic conductivity of these layers, these deeper sediment beds do not appear to represent a continuous water-bearing deposit capable of transmitting abundant quantities of groundwater. In addition, a review of historical groundwater elevation measurements taken from the shallow/intermediate/deep monitoring well clusters shows that there is an upward vertical hydraulic gradient between these deeper beds and the shallow groundwater-bearing zone, supporting the contention that the groundwater within the shallow zone does not migrate downward to the intermediate and deeper zones.

2.3.2 Nature and Extent of Soil Contamination

Soil

Shallow soil at the Moss-American site consists of topsoil, fill, gravelly fill, silt, silty sand, and silty clays. The shallow soil (typically between the ground surface and 10 feet below ground surface [bgs]) at certain locations of the site has been impacted by residues from past wood-preserving operations. This soil contains varying concentrations of polynuclear aromatic hydrocarbons (PAHs); the primary constituents of concern (COCs) in creosote. Other COCs at the site include volatile organic compounds (VOCs) namely benzene, toluene, ethylbenzene, and xylenes (BTEX).

The RI (CH2M HILL, 1990) and extensive pre-design phase soil investigations provided data that define the extent of soil contamination at the Moss-American site. Figure 2-1 of the FSP graphically illustrates the locations and extent of site soil requiring excavation and treatment based on the criteria discussed in the next section. The areas from which soils would require excavation and treatment during the installation of the groundwater remedial system are identified as Areas T1, T2, and T3 in Figure 2-1. Figure 2-1 of the FSP also illustrates the location of the funnel-and-gate groundwater remedial system and the extent of soil requiring excavation in the immediate vicinity of the groundwater remedial system. Soil from Areas T1 and T2 would require excavation and treatment due to the presence of free product, PAHs, and VOCs in the subsurface soil. Soil from Area T3, however, would require excavation and treatment only due to the presence of PAHs and VOCs. The depth of the soil requiring excavation from Areas T1, T2, and T3 ranges from 1 to 10 feet bgs.

In addition to soil generated due to excavation of Areas T1, T2, and T3, soil will be generated due to installation of Gates TG1 through TG6, installation of monitoring and free-product recovery wells, and installation of an underground piping system. This soil will require appropriate management consistent with the requirements of the amended ROD.

Water

Contaminated water due to infiltration of groundwater or precipitation entering the excavations, or precipitation which comes in contact with the contaminated soil will be generated during the construction activities. This water could potentially contain PAHs and VOCs and would have to be appropriately managed.

2.4 CLEANUP LEVELS

2.4.1 Cleanup Levels for Soil

Based on the Amended Record of Decision (ROD) for the site, impacted soil requiring excavation and treatment (using thermal desorption) is defined as:

- Soil that contains free product.
- Soil that exceeds a benzo(a)pyrene equivalent residual contaminant level (RCL) of 78 mg/kg for total carcinogenic polycyclic aromatic hydrocarbons (CPAHs).
- Soil that exceeds the generic migration to groundwater cleanup standards for fluorene and benzo(a)pyrene under Table 1 of WDNR Publication RR-519-97 (Soil Cleanup Levels for Polynuclear Aromatic Hydrocarbons [PAHs] Interim Guidance).
- Soil that exceeds the naphthalene concentration of 100 mg/kg. [Please note that the 100 mg/kg value for naphthalene is neither a new standard nor a new RCL. It represents a value supported by U.S. EPA and KMC at this time that would facilitate attainment of acceptable groundwater naphthalene concentrations in the future.]
- Soil that exceeds generic migration to groundwater standards for benzene, toluene, ethylbenzene, and xylene (BTEX) as presented in NR 720.19.

The amended ROD also requires that soil that exceeds direct contact risk levels for total CPAH (benzo[a]pyrene equivalent) concentrations corresponding to specific land uses (i.e., recreational, industrial, etc.) be appropriately capped. Since the land use for the site has not yet been finalized,

use of a direct contact value for a specific land use may not be justifiable. Nevertheless, for the purposes of this QAPP a residential land use for the entire site has been assumed. Consequently, in accordance with the amended ROD, soil that exceeds the direct contact values of 1.9 mg/kg for total CPAHs (BAP equivalent) in areas outside the 100-year floodplain and 15 mg/kg for total CPAHs (actual) in areas within the 100-year floodplain will be ultimately addressed in a manner consistent with the amended ROD. The direct contact value for soil associated with areas outside the 100-year floodplain may change if an alternative land use (i.e. other than residential land use) is established for the site.

2.4.2 Cleanup Levels for Water

Contaminated water resulting from infiltration of groundwater or precipitation entering the excavations, or precipitation which comes in contact with the contaminated soil will be collected, treated and discharged to Milwaukee Metropolitan Sewerage District's (MMSD's) sanitary sewer system. Contaminated water from the excavations as well as from the asphalt storage used for storing soil requiring treatment will be pumped to two 10,000 gallon aboveground storage tanks (ASTs).

Water collected in the tanks will be pre-treated with a portable water treatment system to meet MMSD's discharge requirements. After treatment, the water will be transferred to tanker trucks for transportation and discharge to the sanitary sewer located along Granville road.

Prior to discharge, all samples will be analyzed for parameters that will satisfy MMSD's discharge requirements. Parameters will include volatile organic compounds (VOCs), PAHs, total metals including cadmium, copper, lead, mercury, nickel, silver, and zinc, cyanide, oil and grease, and total suspended solids (TSS). All analytical methods will be in accordance with the requirements of MMSD.

2.5 PROJECT OBJECTIVES AND SCOPE

2.5.1 Introduction

The QAPP describes the policy, organization, functional activities, and QA/QC protocols necessary to obtain data of sufficient and known quality for use as intended in implementation of the funnel-and-gate groundwater remedy for the site. The objective of the QAPP is to establish standard procedures so that the integrity, accuracy, precision, completeness, and representativeness of the samples are maintained and the required objectives of the amended ROD are achieved.

2.5.2 Project Objectives

The sampling program has been designed to characterize the excavated soil for appropriate management, to confirm that soil requiring treatment has been removed from the excavations, and to verify that water generated during the remedial activities meets the MMSD's sanitary sewer discharge standards.

2.5.3 Specific Objectives

The objectives of the field investigations at Moss-American are to characterize the soil generated during the construction of the groundwater treatment system. During construction, soil will be generated due to the following activities:

- Excavation of Areas T1, T2, and T3.
- Installation of treatment gates G1 through G6.
- Installation of groundwater monitoring and free-product extraction wells.
- Installation of underground piping.

All excavated soil would require appropriate management that is consistent with the cleanup standards and remedial alternatives established in the amended ROD. Consequently, all excavated soil will be classified as:

- Soil that will require treatment
- Soil that will not require treatment but exceeds the generic migration to groundwater cleanup standard of 0.4 mg/kg for naphthalene.
- Soil that will not require treatment but will require an appropriate cover consistent with the amended ROD requirements (i.e. soil that exceeds the direct contact values for total CPAHs).
- Clean soil (i.e. soil that meets all of the site cleanup standards).

2.6 SAMPLE NETWORK DESIGN AND RATIONALE

Section 2 of the Field Sampling Plan (FSP) (Appendix A) describes the sample network design and rationale for sample locations.

2.7 PARAMETERS TO BE TESTED AND FREQUENCY

Table 2-1 of the FSP (Appendix A) presents sample matrices, analytical parameters, and frequencies of sample collection.

2.8 DATA QUALITY OBJECTIVES (DQOs)

Data quality objectives (DQOs) are required for all environmental data collection activities. DQOs are statements of the quality of data needed to support a specific decision or an action. Data quality is defined in terms of the study objectives, rather than in terms of equipment or equipment analysis method characteristics. The DQOs must address the hypotheses that are to be proved or disproved and the necessary quality to support or defend the results obtained.

The DQO process is a series of planning steps based on the scientific method that is designed to ensure that the type, quality, and quantity of environmental data used in decision making are appropriate for the intended application.

DQOs are qualitative and quantitative statements derived from outputs of each step of the DQO process that:

- Clarify the study objective.
- Define the most appropriate type of data to collect.
- Determine the most appropriate conditions from which to collect the data.

The DQOs are then used to develop a scientific and resource-effective sampling design.

The DQO process allow decision makers to define their data requirements and acceptable levels of decision during planning before any data are collected. DQOs are based on the seven-step process described in EPA QA/G-4 (Sept. 1994) document.

The seven-step DQO process was used to establish DQOs. The seven steps are similar for each media of interest (soil and water), so the seven-step process needs to be applied only once.

1. STATE THE PROBLEM

Previous environmental investigations and response actions, as summarized in sections 2.1 and 2.3.2 have confirmed the presence of CPAHs and BTEX in the soil and the groundwater.

Soil

Contaminated soil will be generated during the installation of the groundwater treatment system. In order to segregate and manage all excavated soils in accordance with the requirements of the amended ROD, soils generated during the implementation of the groundwater remedy will require appropriate characterization.

Verification sampling will be required to verify that all soil requiring treatment has been removed from open excavations. Excavations subject to verification sampling will include excavations that will result from the excavation of Areas T1, T2, and T3 and installation of Treatment Gates TG1 through TG6.

Water

During the installation of the groundwater treatment system, contaminated water will be generated due to infiltration of groundwater or precipitation entering the excavations and due to precipitation that may come in contact with the contaminated soil staged on the asphalt pad. This water will require appropriate management.

2. IDENTIFY THE DECISION

Soil

All excavated soil must be further characterized to select the proper action required (i.e. thermal treatment, backfill onsite with an appropriate cover, clean soil). The decision to continue excavation of soil from Areas T1, T2, and T3 and Gates TG1 through TG6 will be dependent on the outcome of verification sampling. Excavation of soil requiring treatment will stop if the analytical results of the verification samples prove that all soil requiring treatment has been removed. Excavation of soil will continue if analytical results of the verification samples prove otherwise.

Water

Water will be treated prior to discharge to the MMSD sanitary sewer system. Water not meeting the MMSD criteria will undergo a second round of treatment prior to discharge.

3. IDENTIFY INPUTS TO THE DECISION

Soil

The soil will be characterized as follows:

- Soil that will require treatment
- Soil that will not require treatment but exceeds the generic migration to groundwater cleanup standard of 0.4 mg/kg for naphthalene.
- Soil that will not require treatment but will require an appropriate cover consistent with the requirements of the amended ROD (i.e. soil that exceeds the direct contact values for total CPAHs).

- Clean soil (i.e., soil that meets all of the site cleanup standards).

Based on the amended ROD for the site, soil requiring excavation and treatment (using thermal desorption) is defined as:

- Soil that contains free product.
- Soil that exceeds a benzo(a)pyrene equivalent residual contaminant level (RCL) of 78 mg/kg for total carcinogenic polycyclic aromatic hydrocarbons (CPAHs).
- Soil that exceeds the generic migration to groundwater cleanup standards for fluorene and benzo(a)pyrene under Table 1 of WDNR Publication RR-519-97 (Soil Cleanup Levels for Polynuclear Aromatic Hydrocarbons [PAHs] Interim Guidance).
- Soil that exceeds the naphthalene concentration of 100 mg/kg.
- Soil that exceeds the generic migration to groundwater for benzene, toluene, ethylbenzene, and xylene (BTEX) as presented in NR 720.19.

Water

Water will be characterized either as meeting or as not meeting MMSD's discharge standards.

4. DEFINE THE STUDY BOUNDARIES

The boundaries of this investigation are based on past RI and predesign studies and include areas necessary to implement the funnel-and-gate groundwater remedy system for the site. Clean up objectives are based on the amended ROD.

Soil

This QAPP covers soil that is anticipated to be generated as a result of the following activities:

- Excavation of Areas T1, T2, and T3.
- Installation of treatment gates G1 through G6.

- Installation of groundwater monitoring and free-product extraction wells.
- Installation of underground piping.

The soil parameters for analysis are based on past studies and include PAHs and BTEX

Water

Contaminated water is expected to be generated due to infiltration of groundwater or precipitation entering the excavations, and due to precipitation that comes in contact with the contaminated soil. This water will require collection, treatment and disposal.

5. DEVELOP A DECISION RULE

Soil

Soils that will require treatment will be staged on the existing asphalt pad and will undergo treatment in a low thermal temperature desorption (LTTD) system at a future date. Soil that will not require treatment but will exceed the generic migration to groundwater cleanup standard of 0.4 mg/kg of naphthalene and direct contact values for total CPAHs (BAP equivalent or otherwise) will be staged in the area shown in Figure 2-1 for future management consistent with the requirements of the amended ROD. Clean soil (i.e. soil that meets all the soil cleanup standards) will be used as backfill material.

The decision to continue excavation of soil from Areas T1, T2, and T3 and Gates G1 through G6 will be dependent on the outcome of verification sampling. Excavation of soil requiring treatment will stop if the analytical results of the verification samples prove that all soil requiring treatment has been removed. Excavation of soil will continue if analytical results of the verification samples prove otherwise.

Water

Water that will meet MMSD's sanitary sewer discharge standards will be discharged to MMSD's sanitary sewer system. Water that does not meet MMSD's discharge standards will undergo a second round of treatment prior to discharge to MMSD's sanitary sewer system.

6. SPECIFY LIMITS ON DECISION ERRORS

Data will be collected with the lowest level of uncertainty possible. All laboratory data will be reviewed for compliance with established methods and U.S. EPA guidelines for acceptability.

7. OPTIMIZE THE DESIGN FOR OBTAINING DATA

Soil

In order to characterize the excavated soil, analytical results of the samples will be compared to the appropriate soil cleanup standards established for the site. Results of this comparison will be used to classify the soil as previously stated. Soils that will require treatment will be staged on the existing asphalt pad and will undergo treatment in a low thermal temperature desorption (LTTD) system at a future date. Soil that will not require treatment but will exceed the generic migration to groundwater cleanup standard of 0.4 mg/kg of naphthalene and direct contact values for total CPAHs (BAP equivalent or otherwise) will be staged in the area shown in Figure 2-1 for future management consistent with the requirements of the amended ROD. Clean soil (i.e., soil that meets all the soil cleanup standards) will be used as backfill material.

Water

In order to determine if treated water meets MMSD's discharge standards, analytical results of the treated water will be compared to the MMSD discharge criteria. Results of this comparison will determine if the treated water meets MMSD's discharge standard. If the treated water meets the discharge standards, than discharge of the water can occur. Water that does not meet MMSD discharge standards will undergo a second round of treatment.

2.9 PROJECT SCHEDULE

The overall schedule for the Moss-American site activities is presented in Figure 2-2.

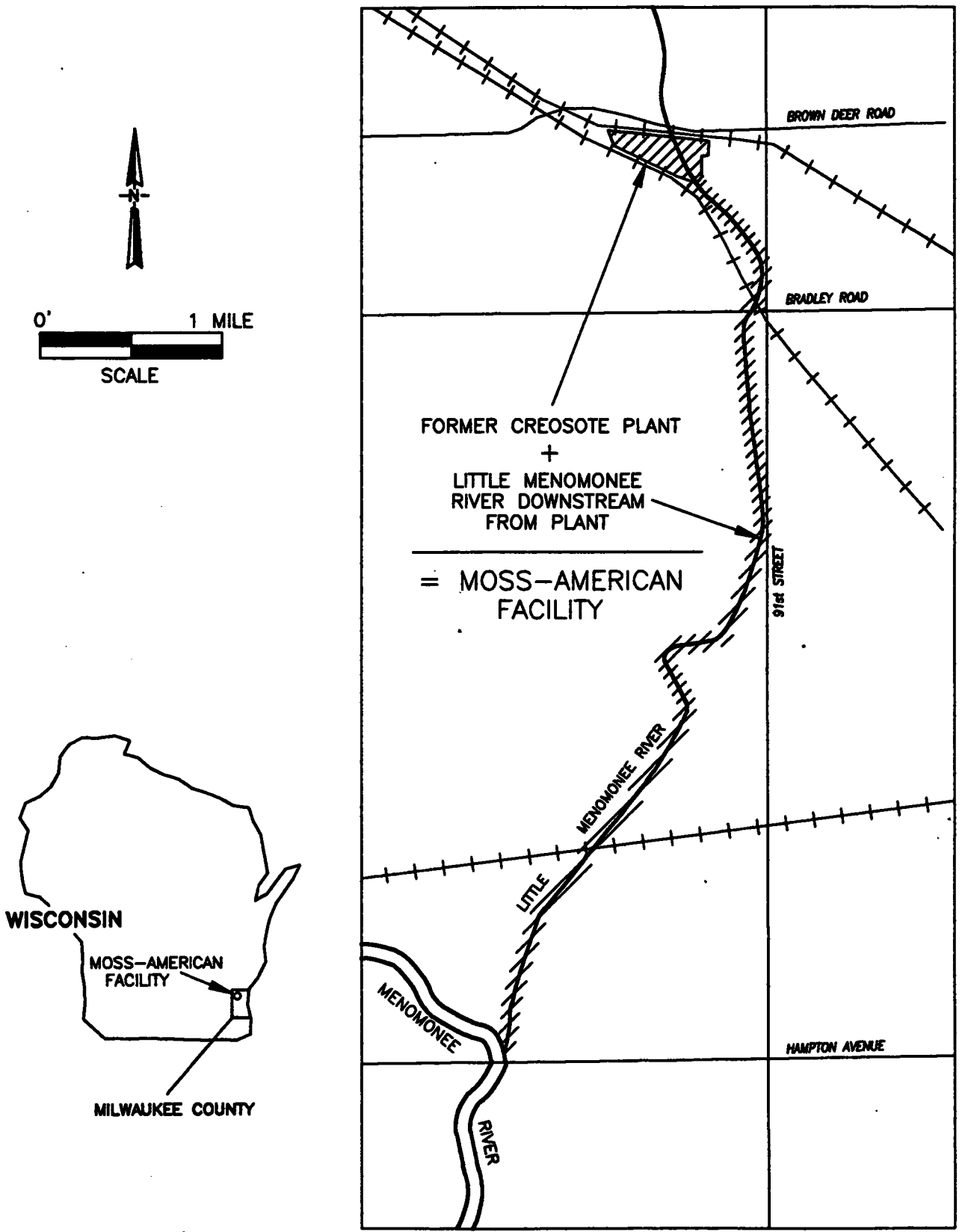

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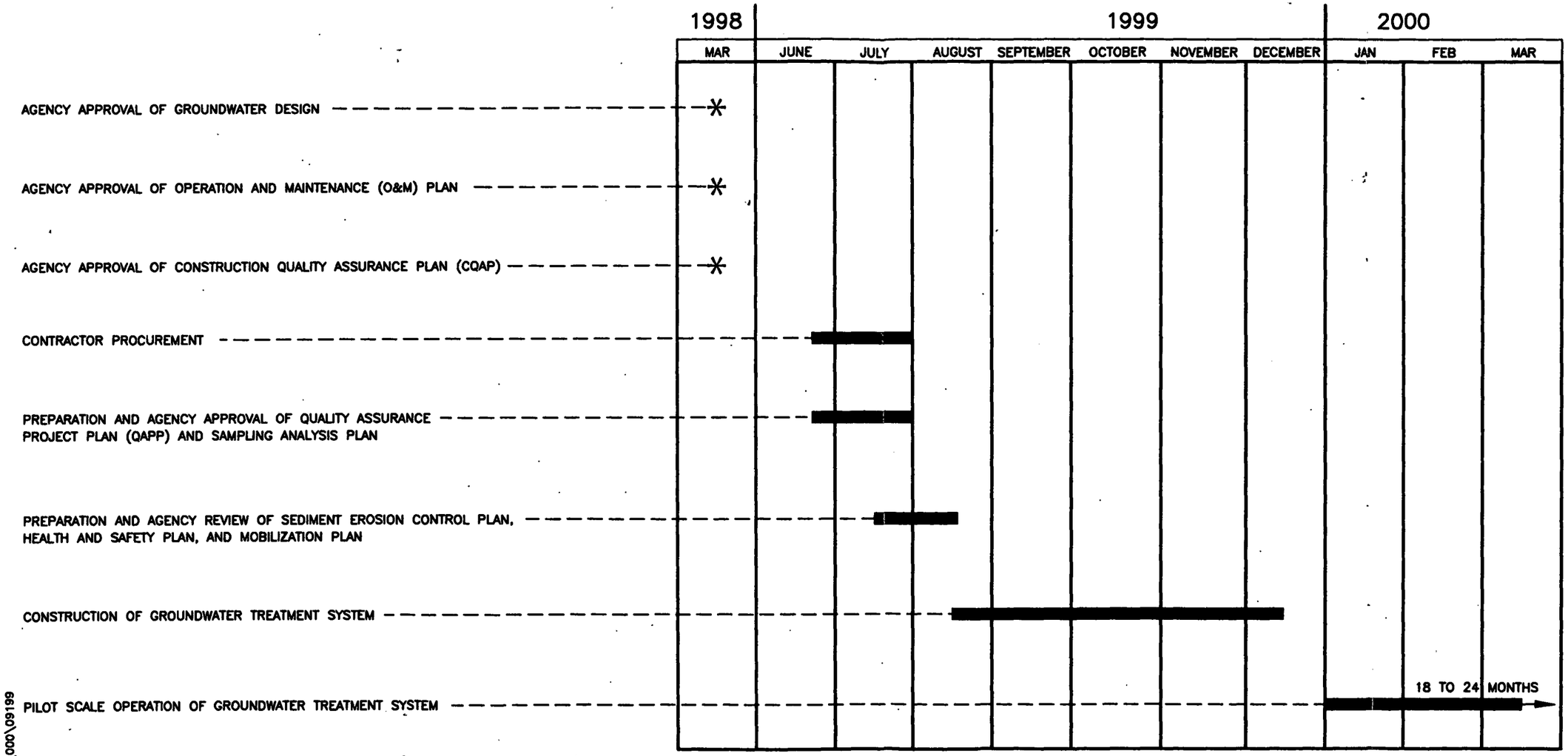
FIGURE 2-1

WESTON MANAGERS DESIGNERS/CONSULTANTS

Three Hawthorn Parkway
Vernon Hills, Illinois
60061

FACILITY LOCATION MAP

MOSS-AMERICAN SITE
Milwaukee, Wisconsin



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* – U.S. EPA APPROVAL

(REVISED ON 22 JUNE 1999)

WESTON MANAGERS DESIGNERS/CONSULTANTS

Three Hawthorn Parkway
Vernon Hills, Illinois
60061

FIGURE 2-2

ANTICIPATED PROJECT SCHEDULE FOR
FUNNEL/GATE DESIGN AND CONSTRUCTION
MOSS – AMERICAN SITE
Milwaukee, Wisconsin

SECTION 3

PROJECT ORGANIZATION AND RESPONSIBILITY

This section presents overall project organization and responsibilities, lines of communication, responsibilities for specific quality assurance tasks, and responsibilities for field and laboratory operations.

Key personnel responsibilities in four specific areas (project management, QA, field operations, and laboratory operations) are discussed in the following subsections. Figure 3-1 presents the overall project organization chart.

3.1 PROJECT MANAGEMENT

Operational responsibilities involving execution and direct management of the technical and administrative aspects of this project have been assigned as follows:

WESTON/KMC Project Managers—Mr. Tom Graan is the WESTON Project Manager. Mr. Keith Watson is the KMC Project Manager. The Project Managers are responsible for implementing the project objectives, and have the authority to commit the resources necessary to meet the project objectives and requirements. The Project Managers primary function is to ensure that the technical, financial, and scheduling objectives are achieved successfully. The WESTON Project Manager will coordinate with the KMC Project Manager, the WESTON Project Director, the U.S. EPA RPM, and the WDNR Project Manager on the following issues.

- Coordination and management of project personnel.
- Project scheduling.
- Coordination and review of required deliverables.
- General QA of field activities.

WESTON/KMC Project Director —Mr. Kurt Stimpson is the WESTON Project Director. The Project Director has overall responsibility for all tasks performed under this QAPP. The Project Director is responsible for ensuring that the project meets all U.S. EPA, WDNR, and KMC objectives and quality standards. He is also responsible for ensuring that all work is executed in accordance with the U.S. EPA's technical directives. The WESTON Project Director is responsible for assigning and monitoring the functions and responsibilities of the WESTON Project Manager. In addition, he will commit the necessary resources and personnel to meet the objectives of this investigation.

U.S. EPA Remedial Project Manager —Mr. Russ Hart is the U.S. EPA RPM for this project. Mr. Hart has overall responsibility for all phases of the Moss-American funnel-and-gate groundwater remedy system.

U.S. EPA Field Services Section (FSS) Quality Assurance Reviewer--The U.S. EPA Region V Superfund FSS will be responsible to review and provide comments to the U.S EPA RPM for all QAPPs.

Wisconsin DNR Project Manager—Mr. Gary Edelstein is the Wisconsin Department of Natural Resources project manager. His overall responsibility is to review project documents, monitor the progress of the Moss-American activities, and serve as a liaison between the state and the U.S EPA in order to ensure that all activities address state requirements and are executed in accordance with state regulations and/or project-specific agreements.

3.2 QUALITY ASSURANCE

All aspects regarding the implementation of the funnel-and-gate groundwater remedial system at the site are subject to review by the WESTON Project Director and/or Project Manager, the KMC Project Manager, and approval by the U.S. EPA. Primary responsibility for all QC activities at the

site is held by the WESTON Project Manager. The specific QA tasks and responsibilities are summarized below.

3.2.1 Final Review/Approval of the Quality Assurance Project Plan

WESTON

QA activities for the Moss-American site will be performed by the WESTON Project Director and/or Project Manager. The WESTON Project Director, WESTON Project Manager, and KMC Project Manager will review the Moss-American QAPP prior to submitting the document to the U.S. EPA.

U.S EPA Region V

The U.S. EPA Region V Superfund Division, Field Services Section (FSS) Quality Assurance Reviewer reviews all QAPPs. They shall provide recommendations for approval to the U.S. EPA Region V RPM. The WDNR Project Manager will also be provided with the opportunity to review and comment on the QAPP.

3.2.2 Validation of Analytical Data

All analytical data will be validated by trained WESTON validation personnel in accordance with specifications outlined in Section 9 of this QAPP.

3.2.3 Performance and Systems Audits

Field Audits

- External field audits of Moss-American site activities may be conducted by the U.S. EPA Region V any time during the field operations. These audits may or may not be announced at the discretion of the U.S. EPA Region V. External audits will be conducted according to the field activity information presented in the QAPP.

- Internal field audits are the primary responsibility of the WESTON Project Director and/or Project Manager and KMC Project Manager, as applicable. These audits will verify that all established procedures are being followed. Internal field audits will be conducted at least once at the beginning of the site sample collection activities.

Laboratory Audits

- External laboratory audits may be conducted by the U.S. EPA Region V any time during the laboratory activities. These audits may or may not be announced and are at the discretion of the U.S. EPA Region V. External audits will be conducted according to the laboratory method information presented in the QAPP.

- Internal laboratory audits are the primary responsibility of Lancaster Laboratories and the KMC Project Manager, as applicable. These audits will verify that all established procedures are being followed. Internal laboratory audits are further defined in the Lancaster Laboratories QAPP, Section 12 (Appendix B).

3.2.4 Final Assessment of Quality Assurance Objectives

WESTON's Project Director and/or Project Manager, along with the U.S EPA Region V RPM will jointly assess the validated data to determine whether the QA objectives have been met.

3.2.5 Internal Quality Assurance Review and Approval of Reports, Standard Operating Procedures (SOPs), and Field Activities

Responsibilities for internal QA review and approval of reports, SOPs and field activities are as follows:

- The WESTON Project Director and Project Manager are responsible for reviewing all necessary reports and procedures that can affect the data quality for planned site activities.

- The WESTON Project Director and Project Manager are responsible for auditing the implementation of the QA program (as outlined in the QAPP) to ensure conformance with WESTON's, KMC, WDNR, and U.S. EPA's project requirements.

- The WESTON Field Team Leader (FTL) shall report the status of the field QA program to the WESTON Project Director and/or Project Manager on a regular basis during field activities.

- The WESTON Project Director and Project Manager shall provide QA technical assistance to the field and project staff during the QA plan's development and field implementation.

3.2.6 Evidence Audits of Field Records

Internal evidence audits of field records shall be the responsibility of the WESTON Project Director and/or Project Manager. External evidence audits of field records are the responsibility of U.S. EPA Region V.

3.2.7 Approval of Laboratory Analytical Procedures

The U.S. EPA Region V must approve all laboratory procedures. Internally, the KMC Project Manager will review and approve analytical procedures.

3.3 FIELD OPERATIONS

The WESTON field team shall operate under the direction of the WESTON Project Manager. The field team's activities oversight of soil excavation, sample collection, field measurements, sample packaging, sample shipment, and sample COC preparation. Within the field team, there will be a minimum of three specific roles:

- FTL—Responsible for the management of the field team and the supervision of all field activities in the absence of the WESTON Project Manager.

- Site Health and Safety Coordinator (SHSC)—Responsible for the implementation of the Health and Safety Plan. Will perform health and safety monitoring and ensure compliance with all health and safety requirements for the Moss-American site.

- **Field Sample Manager (FSM)**—Manages the custody of all samples from the time they are collected to when they are shipped. Is responsible for ensuring that all sample management and documentation procedures are implemented correctly.

For health and safety and QA reasons, a minimum of two field personnel will be present at all times during sampling activities. Depending on the schedule for the field sampling activity, the WESTON Project Manager will evaluate the need for additional personnel. When necessary, the FTL may also perform in the capacity of the SHSC. To the extent practicable, the FSM will not be given any additional responsibilities other than field samples. All personnel will be given the title of field sampler in order to encourage full utilization of all personnel at all times. The field samplers will collect samples and decontaminate equipment. In the absence of the WESTON Project Manager, the FTL will provide QA of field activities.

3.4 LABORATORY OPERATIONS

All laboratory analyses for samples collected as part of the activities at Moss-American are anticipated to be performed by Lancaster Laboratories. The WESTON Project Manager is responsible for initiating and scheduling all analysis. The WESTON Project Manager will coordinate with the FTL in executing all laboratory arrangements. The organization and key responsibilities within Lancaster Laboratories are discussed in the following subsections.

Lancaster Laboratories

Lancaster Laboratories (Lancaster, Pennsylvania) is anticipated to provide all of the soil and groundwater analysis required during implementation of the funnel-and-gate groundwater remedy system. An overview of the Lancaster Laboratories laboratory organization chart is presented in the laboratory QAPP (Appendix B, Section 4).

Lancaster Laboratories Project Manager - Ms. Kay Hildy is the laboratory Project Manager for this project. She is responsible for coordinating all sampling with the WESTON and KMC Project

Managers. She is also responsible for implementing the required laboratory methods, and she has the authority to commit the resources necessary to meet the project analytical requirements.

Lancaster Laboratories Quality Assurance/Quality Control (QA/QC) - Ms. Kathleen Loewen is the Lancaster Laboratories QA/QC Manager. Ms. Loewen has the overall responsibility to evaluate the adherence to policies and to assure that systems are in place to produce the level of quality defined in the QAPP.

Lancaster Laboratories Organics Manager - Ms. Michele Turner is the volatile organics manager. Ms. Turner is responsible for the supervision of the volatile organic departments. Ms. Turner is also in charge of sample flow, analysis, data review, and reporting of the final results.

Lancaster Laboratories Nonvolatile Organics Manager - Dr. Jon Kauffman is the nonvolatile organics manager. Dr. Kauffman is responsible for the supervision of the semivolatile, pesticide and organic extraction departments. Dr. Kauffman is also in charge of sample flow, analysis, data review, and reporting of the final results.

Lancaster Laboratories Metals and Data Packages Manager - Ms. Ramona Goss is the metals and data packages manager. Ms. Goss is responsible for the supervision of the metals department. Ms. Goss is also in charge of sample flow, analysis, data review, and reporting of final results.

Lancaster Laboratories Instrumental Water Quality and Water Quality Group Leader - Mr. Erik Frederiksen is the group leader for the water quality group. He is in charge of sample flow, analysis, data review, and reporting of final results.

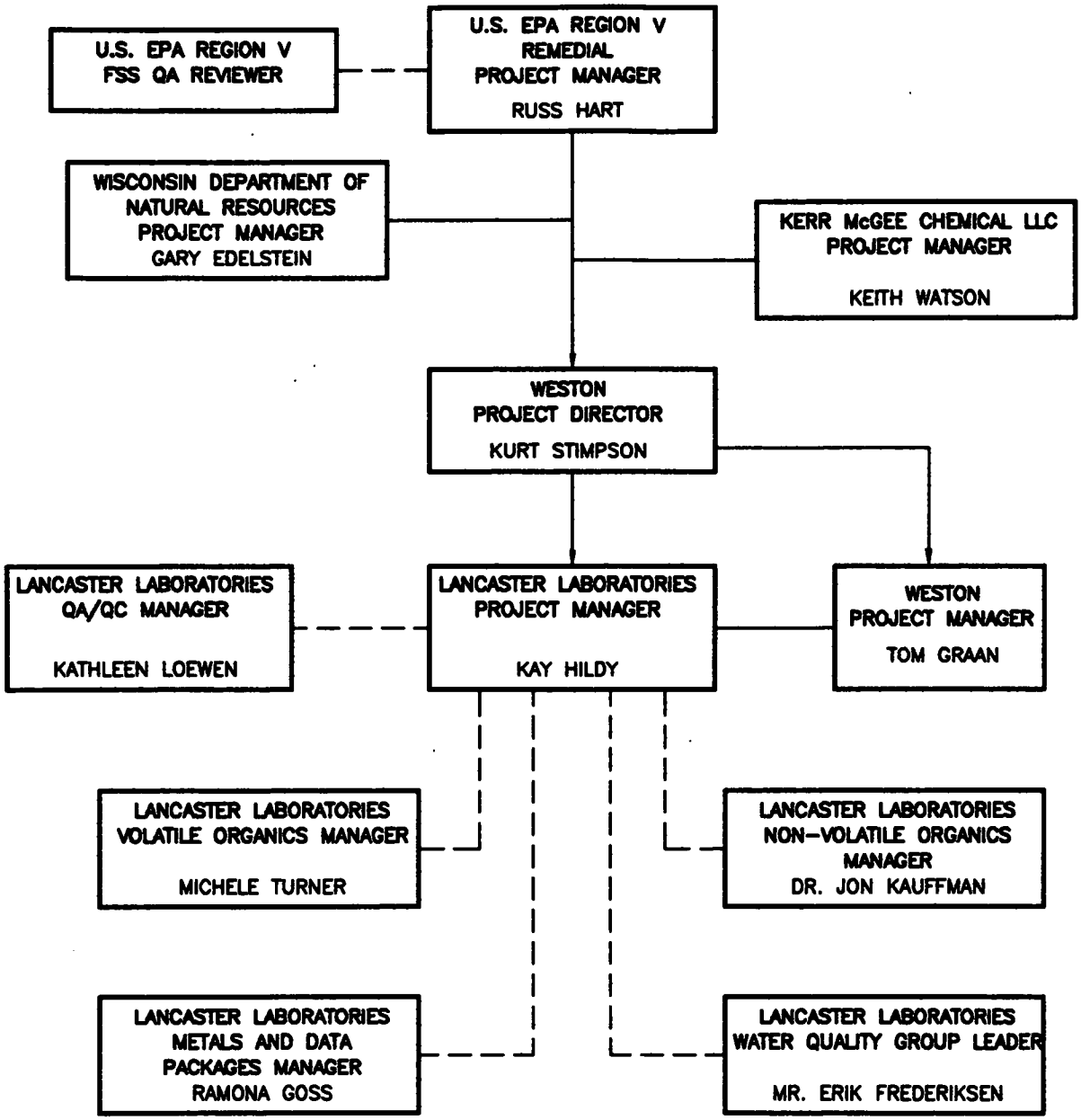

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FIGURE 3-1

WESTON MANAGERS DESIGNERS/CONSULTANTS

Three Hawthorn Parkway
Vernon Hills, Illinois
60061

PROJECT ORGANIZATION CHART
MOSS-AMERICAN SITE
Milwaukee, Wisconsin

SECTION 4

QUALITY ASSURANCE OBJECTIVE FOR MEASUREMENT DATA

The overall QA objective is to develop and implement procedures for field sampling, COC, laboratory analysis, and reporting that will provide results which are legally defensible. Specific procedures for sampling, COC, laboratory instrument calibration, laboratory analysis, reporting of data, internal QC audits, preventive maintenance of field equipment, and corrective action are described in other sections of this QAPP. The purpose of this section is to address the specific objectives for accuracy, precision, completeness, representativeness, and comparability of reported data from all analytical laboratories. QA objectives for field measurements are also discussed in this section.

4.1 LEVEL OF QUALITY CONTROL EFFORT

Field blank, trip blank, field duplicate, and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling program. Field and trip blanks consisting of ultra pure water (laboratory grade) will be submitted to the analytical laboratories to assess the quality of the data resulting from the field sampling program. Field blank samples are analyzed to check for procedures at the site that may cause sample contamination. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Field duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes are performed in duplicate and are known as matrix spike/matrix spike duplicate (MS/MSD) samples. MS/MSD samples are designated and collected for organic analyses only. For inorganic analyses, spike/duplicate samples are designated and collected.

The general level of the QC effort will be one field duplicate and one field blank for every 10 or fewer investigative samples (i.e., a 10 percent frequency). However, field blanks will only be

collected for water samples and will consist of ultra pure water (laboratory grade). No field blanks will be collected for soil samples because the U.S. EPA Region V Central Regional Laboratory (CRL) discourages the use of water for soil samples. One volatile organic analysis (VOA) trip blank, consisting of ultra pure water (laboratory grade), will be included along with each shipment container of aqueous volatile organic compound (VOC) samples.

MS/MSD and spike/duplicate samples are investigative samples on which additional analyses are performed. One MS/MSD and spike/duplicate sample will be collected/designated for every 20 or fewer investigative samples per sample matrix (e.g., soil and water). Soil MS/MSD samples require no extra volume. Soil inorganic spike/duplicate samples require no extra volume. Aqueous MS/MSD samples must be collected at triple the volume for VOCs and double the volume for PAHs. Aqueous spike/duplicate samples require double the normal volume for total metals and cyanide. Field blanks, trip blanks, and field duplicate samples will not be used as MS/MSD or spike/duplicate samples.

The specific level of field QC for samples collected as part of the sampling activities for the Moss-American site is summarized by sample matrix and parameter in Table 2-1 of the FSP. Sampling procedures are specified in the FSP.

All internal laboratory QA/QC will be in accordance with the requirements specified in each laboratory SOP (Appendix C).

4.2 ACCURACY, PRECISION, AND SENSITIVITY OF ANALYSIS

The fundamental QA objective with respect to accuracy, precision, and sensitivity of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols. The accuracy and precision requirements are specified in the laboratory QAPP (Appendix B) and laboratory SOPs (Appendix C).

Water generated during the installation of groundwater treatment system will not undergo screening in the field. Soil will be screened using a PID as one method to help determine potential contamination and will also be used for health and safety screening purposes. The PID SOP is located in Appendix E.

4.3 COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that the laboratory will provide data meeting QC acceptance criteria for 90 percent or more of all samples tested using the specified analytical methods. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness} = \frac{\text{Number of valid data}}{\text{Number of samples collected For each parameter analyzed}} \times 100\%$$

Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling network was designed to provide data representative of conditions at the site. The rationale of the sampling network is discussed in detail in the FSP (Appendix A). Representativeness will be satisfied by ensuring that the FSP is followed, proper sampling techniques are used, proper analytical procedures are followed, and holding times of the samples are not exceeded in the laboratory. Representativeness will also be assessed by the analysis of field duplicate samples.

Comparability expresses the confidence with which one data set can be compared with another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data, as documented in the QAPP, are expected to provide comparable data.

SECTION 5

SAMPLING PROCEDURES

Sampling procedures are specified in the FSP, which is presented as Appendix A.

SECTION 6
SAMPLE CUSTODY

6.1 INTRODUCTION

Sample custody, or COC protocols will be as described in the *National Enforcement Investigations Center (NEIC) Policies and Procedures*, (U.S. EPA, 1985). This custody is in three parts: sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained in a secure area.

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

- In your possession.
- In your view, after being in your possession.
- In your possession, and you place it in a secured location.
- In a designated secure area.

6.2 FIELD CHAIN-OF-CUSTODY PROCEDURES

The key requirements for ensuring field chain of custody are summarized in this section. The specifics of sample handling and completion of sample documentation forms are detailed in Section 6 of the FSP (Appendix A).

The sample packaging and shipping procedures summarized below will ensure that samples arrive at the laboratory with the COC intact. The protocol for specific sample numbering using case numbers and traffic report numbers (if applicable) and other sample designations are included in the FSP (Appendix A).

6.2.1 Field Procedures

Field procedures are as follows:

(a) The field sampler is personally responsible for the care and custody of the samples until they are transferred to the field sample manager (FSM) or properly dispatched. As few people as possible should handle the samples.

(b) All bottles will be tagged with sample numbers and locations. The FSP defines the site-specific sample numbering system.

(c) Sample labels are to be completed for each sample using waterproof ink unless prohibited by weather conditions.

(d) WESTON's Project Manager (or his designee) will review all field activities to determine whether proper custody procedures were followed during the field work. He or she will notify the WESTON SMC and Project Manager of a breach or irregularity in COC procedures.

6.2.2 Field Logbooks/Documentation

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

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

The title page of each logbook will contain the following:

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

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

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

Samples will be collected following the sampling procedures documented in the FSP (Appendix A). The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume, and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description. The sample identification system is described in Section 4 of the FSP.

6.2.3 Transfer of Custody and Shipment Procedures

Transfer of custody and shipment procedures are as follows:

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

(b) Samples will be packaged for shipment and dispatched to the appropriate laboratory for analysis. A separate signed COC form will be enclosed in each sample shipment container. Shipping containers will be locked and secured with strapping tape and custody seals for shipment to the laboratory. A custody seal will be attached to the front right and back left of the shipment container. The custody seals will be covered with clear plastic tape. The shipment container will be strapped shut with strapping tape in at least two locations.

(c) Whenever samples are split with a source or government agency, a separate sample receipt will be prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency will request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this will be noted in the "received by" space.

(d) All shipments will be accompanied by the COC form identifying the contents. The bottom two forms will be retained by the sampler for return to the sampling office.

(e) If the samples are sent by common carrier, a bill of lading will be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers will not be required to sign off on the custody form as long as the custody forms are sealed inside the sample shipment container and the custody seals remain intact.

(f) Samples will be shipped in accordance with International Air Transport Association (IATA) requirements for air transport and in accordance with Department of Transportation requirements for ground transport.

6.2.4 Summary of Field Chain-of-Custody Procedures

The WESTON site field team will consist of the following:

- The FTL.
- The SHSC.
- The FSM.
- The Field Sampler.

There will be a minimum of two people in each field team. All members will be considered to be field samplers and may be involved in the actual sample collection. Depending on the magnitude of the field operations, the WESTON Project Manager will evaluate the need for additional personnel. When necessary, the FTL will also perform in the capacity of the SHSC. To the extent practicable, the FSM will not be given any additional responsibilities other than sometimes performing as a field sampler. If more than two people are in the field team, there may be personnel who are designated as only field samplers.

The FTL will have overall responsibility for ensuring the completion of all field activities in accordance with the QAPP and FSP. The FTL is the overall coordinator of sampling activities at the site and is the communication link between field team members and the WESTON Project Manager. The FTL will assign specific field duties to the team members based on input from the WESTON Project Manager.

The FSM will be responsible for preparing (and reviewing for accuracy and completeness) all sample paperwork such as COC forms, sample labels, and any other paperwork required for sample documentation. The FSM will also prepare all sample shipment information such as airbills. If the FSM requests assistance from other members of the field team in completing sample paperwork, the FSM will be responsible for reviewing and ensuring the accuracy and completeness of this paperwork before he/she encloses it in the sample shipment container. All members of the field team

may be involved in the actual sample packaging and shipment. The FSM is responsible for tracking all sample paperwork from the time of receipt until the completed paperwork is given to the WESTON Project Manager.

The FTL is responsible for maintaining the site logbook. The site logbook will contain notes made by the FTL on-site activities, including the tracking of the samples from the time of sample collection to the delivery of the samples to the shipping carrier. The names and function of all field team members will be listed in the logbook. During the course of sample collection activities, the FTL will document the times and dates of all sampling activities (e.g., who collected the samples, when and where the samples were collected, who delivered the samples to FSM, when the sample coolers were delivered to the shipping carrier) If the FSM was part of the sampling team this will be specifically noted. The FTL will note the names of the actual samplers for each station location along with the time, date, station location identifier and sample identifiers, etc.

The collected samples will be transported to the FSM by a member or members of the field team. If the sample locations are far apart, multiple samples may be collected prior to delivering them to the FSM. The FTL will ensure that any preservation requirements (e.g., keeping the samples cool) are implemented prior to the time that the samples are delivered to the FSM. To the extent practicable, the FSM will be in view of the sampling crew.

Upon receipt of the samples, the FSM will be responsible for ensuring that custody is transferred. The FSM will require the field team member delivering the samples to sign and date the COC form associated with the samples as relinquisher of the samples in the "relinquished by" area. The FSM will then sign the forms as the recipient. The signed forms will be the same forms that will accompany the samples to the laboratory. Prior to enclosing the forms in the shipment container, the FSM will sign the COC form to indicate he or she is relinquishing custody to the shipment carrier. If the forms are sealed in the shipment container with COC seals on the outside of the container, the shipment carrier will not sign the forms as the recipient. The FSM will be responsible for completing the remainder of all forms except as noted previously.

The team member delivering the samples will provide the FSM with the individual time of collection for each sample. All sample documentation shipped with the sample to the laboratory will become part of the evidence file for the samples. The field logbook will be maintained in the site file or in the custody of the FTL.

The FSM assumes custody of the samples once the FSM has signed the COC forms. If the FSM must leave the "staging area" (where sample preparation for shipment and documentation completion is performed), the samples will either be locked inside of the sampling team's vehicle/trailer, or will be secured in a sample shipment container with custody seals. The custody seals will be inspected by the FSM upon return to the staging area to ensure they are intact. These practices will be followed whenever necessary to maintain custody of the samples in the field and will be logged into the site logbook.

6.3 LABORATORY CHAIN-OF-CUSTODY PROCEDURES

The purpose of laboratory COC procedures is to document the history of sample containers and labels, including sample extracts or digestates. The associated records should provide traceability from the time of preparation of sample containers, through collection, shipment, analysis, and disposal of the sample. Items under custody will be:

- Maintained in the physical possession or view of the responsible party.
- Placed and/or stored in a designated secure area to prevent tampering. This secure area must be accessible only to authorized personnel.

Lancaster Laboratories sample custody procedures, sample log-in, sample storage and discard, and internal chain-of-custody documentation SOPs are included in section 7 of the laboratory QAPP (Appendix B).

6.4 FINAL EVIDENCE FILES CUSTODY PROCEDURES

WESTON is the custodian of the evidence file and maintains the contents of the evidence files for the Moss-American activities. WESTON maintains all relevant records, reports, correspondence, logs, field notebooks, pictures, subcontractor reports, and the data reviews in a secured, limited access area.

SECTION 7
CALIBRATION PROCEDURES AND FREQUENCY

This section describes procedures for maintaining the accuracy of all the instruments and measuring equipment which are used for conducting field tests and laboratory analyses. For any activity that influences data quality, all instruments and equipment should be calibrated prior to each day's use or on a scheduled periodic basis.

7.1 FIELD INSTRUMENTS/EQUIPMENT

Instrument and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that the accuracy and reproducibility of results are consistent with the manufacturer's specifications. WESTON further requires that field instruments be calibrated and maintained by trained personnel.

Equipment to be used during the field sampling will be examined to certify that it is in operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to ensure that all maintenance requirements are being observed. Field notes from previous sampling trips will be reviewed so that any prior equipment problem is not overlooked and all necessary repairs to equipment have been made.

Calibration of field instruments is governed by the specific SOP for the applicable field analysis method, and such procedures take precedence over the following general discussion. Calibration of field instruments will be performed at the intervals specified by the manufacturer or more frequently as conditions dictate. In the event that an internally calibrated field instrument fails to meet calibration/checkout procedures, it will be returned to the manufacturer for service.

Field instruments to be used during the Moss-American site field investigation include a photoionization (PID) for screening the soil and for personnel health and safety.

The calibration and checkout of field instruments will be performed prior to use each day. The calibration, checkout, and maintenance programs for each instrument are outlined in the respective SOPs presented in Appendix E, along with the procedures for field measurements.

All calibration performed in the field will be documented in the field logbook. A master calibration/maintenance file will be maintained by the WESTON FTL at the site office for each measuring instrument and will include at least the following information:

- Name of device or instrument calibrated.
- Device or instrument serial or identification (I.D.) number.
- Frequency of calibration.
- Results of calibration.
- Name of person performing the calibration.
- Identification of the calibration media (e.g., pH buffer solutions).

Tape measures used to locate sampling stations and to determine depths in boreholes or wells will be examined visually prior to each day of use to check for damage. Damaged tape measures will not be used.

7.2 LABORATORY INSTRUMENTS

The SOP for each analysis performed in the laboratory describes the calibration procedures, their frequency, acceptance criteria, and the conditions that will require recalibration. All calibration procedures and frequencies shall be in accordance with the laboratory's QAPP found in Appendix B and Laboratory SOPs found in Appendix C.

The laboratory maintains a sample logbook for each instrument which will contain the following information: instrument identification, serial number, date of calibration, analyst, calibration solutions run and the samples associated with these calibrations.

SECTION 8

ANALYTICAL PROCEDURES

This section describes the analytical procedures for all analyses to be conducted for the implementation of the funnel-and-gate groundwater remedy at the Moss-American site. Lancaster Laboratories of Lancaster, Pennsylvania will perform all of the analytical analysis for the soil and groundwater samples. Table 8-1 identifies the analytical method and laboratory detection limit for each soil and water parameter. Table 2-1 in the FSP (Appendix A) identifies the laboratory parameters for each medium to be sampled and the corresponding QC samples.

8.1 OFF-SITE LABORATORY ANALYTICAL SERVICES

Soil and groundwater samples will be analyzed for volatile organic and semivolatile organic or PAH constituents. The groundwater will also be analyzed for metals (cadmium, copper, lead, mercury, nickel, silver, and zinc), cyanide, oil and grease, and total suspended solids in order to meet the MMSD water discharge requirements. The analytical methods are consistent with the methods previously used at the site for the quarterly groundwater sampling and other phases of work. Lancaster Laboratories has also been involved with the analysis over the last several investigations. All analytical analyses will be in accordance with the protocols outlined in the respective laboratory SOPs in Appendix C.

8.2 FIELD SCREENING ANALYTICAL PROTOCOLS

The procedures associated with the field measurements with the PID are described in the SOPs presented in Appendix E.

Table 8-1

Analytical Methods and Project Detection Limits
Moss-American Site
Milwaukee, Wisconsin

Parameter	Soil Method	Soil Detection Limit (ug/kg)	Water Method	Water Detection Limit (ug/L)
Semivolatile Organics				
Benzo(a)anthracene	8310	3	625	10
Chyrsene	8310	11	---	---
Dibenzo(a,h)anthracene	8310	5	---	---
Benzo(b)fluoranthene	8310	2	---	---
Benzo(k)fluoranthene	8310	2	---	---
Benzo(g,h,i)perylene	8310	16	---	---
Benzo(a)pyrene	8310	3	625	10
Indeno(1,2,3-cd)pyrene	8310	11	---	---
Fluorene	8310	27	625	10
Napthalene	8310	270	625	10
Phenanthrene	---	---	625	10
Volatile Organics				
Benzene	8021B	20	8021B	1
Toluene	8021B	20	8021B	1
Ethylbenzene	8021B	20	8021B	1
Total Xylene	8021B	20	8021B	1
Methyl bromide	---	---	8021B	5
Methyl chloride	---	---	8021B	5
Metals				
Cadmium	---	---	200.7	10
Copper	---	---	200.7	25
Lead	---	---	200.7	100
Mercury	---	---	245.1	0.2
Nickel	---	---	200.7	50
Silver	---	---	200.7	20
Zinc	---	---	200.7	25
Other Analytical Parameters				
Cyanide	---	---	335.4	5
Oil and Grease	---	---	413.1	8,000
pH	---	---	150.1	10
Total Suspended Solids(TSS)	---	---	160.2	9,000

--- Analysis not required.

SECTION 9
INTERNAL QUALITY CONTROL CHECKS

This section describes the internal QC checks for field sample collection, field measurements, and laboratory analyses.

9.1 FIELD SAMPLE COLLECTION

The assessment of QC for field sampling will be made through the collection of field blank and field duplicate samples, in accordance with the applicable procedures and frequency described in Table 2-1 of the FSP.

9.2 FIELD MEASUREMENT

Assessment of field sampling precision and bias will be made through collection of field duplicates and field blanks in accordance with the applicable procedures described in the FSP at the frequency indicated in the Sampling and Analysis Summary .

9.3 LABORATORY ANALYSIS

QC checks for the analytical analysis are identified in the corresponding SOPs in Appendix C .

SECTION 10

DATA REDUCTION, VALIDATION, AND REPORTING

This section identifies responsibilities and procedures for data reduction, validation, and reporting for sample collection and laboratory services.

10.1 FIELD MEASUREMENTS AND SAMPLE COLLECTION

No field measurement data will be generated during background soil and groundwater collection activities. A PID will be used to help characterize potentially contaminated soil and will also be used for health and safety screening purposes.

10.2 LABORATORY SERVICES

10.2.1 Data Reduction

Raw analytical data generated in the laboratories is collected on printouts from the instruments and associated data system or manually in bound notebook. Analysts review data as it is generated to determine that the instruments are performing within specifications. This review includes calibration checks, surrogate recoveries, blank checks, retention time reproducibility, and other QC checks. If any problems are noted during the analytical run, corrective action is taken and documented. Each analytical run is reviewed by a chemist for completeness and accuracy prior to interpretation and data reduction. Results are reported as µg/L for water samples and µg/kg for solid samples. Soil samples are reported on an as received and a dry weight basis. Additional information regarding the laboratory protocol for data storage, security, and archiving is provided in the laboratory SOP in Appendix D.

10.2.2 Data Validation

Data validation will be performed by trained WESTON personnel. Validation for will be accomplished by comparing the contents of the data packages and QA/QC results to the requirements contained in the method SOP. Raw data such as chromatograms, mass spectra data reports, and data station printouts will be examined to ensure that reported results are accurate. The validation protocols for the data are based on the following guidelines:

- *U.S EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* —U.S. EPA, February 1994.
- *U.S EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* —U.S. EPA, February 1994.

10.2.3 Data Reporting

The analytical laboratory will prepare and submit full analytical and QC reports to WESTON for review. They will include the following (as applicable):

1. Narrative including statement of samples received, description of any deviations from the standard SOP procedures, explanation of qualifications regarding data quality, and any other significant problems encountered during analysis. QC frequency, overall performance and exceptions to QAPP criteria are included along with unresolved issues on sample collection, preservation, shipping, receipt, or identification.

2. Data on each sample are reported on a data form. Sample identifiers (both field and laboratory) are given along with collection and laboratory receipt dates. Sample results, units, and qualifiers are provided.

3. The results of QC samples (duplicates, MS/MSD, and method blanks) are reported.

4. All associated raw data for standards and samples.

5. Field and laboratory COC documentation pertaining to each sample delivery group analyzed.

SECTION 11

PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

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

QA system audits are conducted at least once during activities that may affect the integrity of the sampling program. The objectives of the systems audits are:

- To verify that a system of QC measures, procedures, reviews, and approvals is established for all activities that generate and process environmentally-related data.
- To verify that a system for project documentation (records, COC forms, analytical tags, logbooks, worksheets, etc.) is established.
- To verify documentation of the required QC reviews, approvals, and activity records (logbooks, worksheets, etc.).
- To identify nonconformance with the established system of QC measures, procedures, reviews, approvals, and documentation.
- To recommend corrective actions for identified nonconformance.
- To verify implementation of corrective action.
- To provide written reports of audits.

11.1 FIELD AUDITS

Internal audits of field activities at the site will be the responsibility of the WESTON Project Director and/or Project Manager. In the absence of both persons, the QA of field activities will be conducted by the designated FTL. Field audits will cover the following:

- Organization and responsibilities—To determine whether the QA organization is operational.
- Collection of samples—To ensure that written procedures are available and being followed.
- COC program—To ensure that the appropriate steps have been followed in the traceability of sample origin.
- Implementation of the operational procedures—To ensure that the appropriate QC checks are being made in the field and records are maintained of these checks.
- Equipment—To determine whether the specified equipment is available, calibrated, and in proper working order.
- Training—To ensure that sampling crews are adequately trained.
- Records—To ensure that recordkeeping procedures are operational and that field notebooks, log sheets, bench sheets, and tracking forms are properly prepared and maintained.
- Health and Safety Audit.

These audits will occur at the outset of the project to verify that all established procedures are followed. Follow-up audits will be conducted to ensure correction of any deficiencies that were previously identified and to verify that QA procedures are maintained throughout the project.

In addition, constant surveillance of field sampling and testing activities shall be performed by qualified technical personnel, as approved by the site manager. The Project Manager may conduct audits of site work procedures on an unscheduled basis.

External field audits may also be conducted by U.S. EPA Region V. These audits may or may not be announced and are at the discretion of the U.S. EPA Region V. External field audits will be conducted according to the field activity information presented in the QAPP.

11.2 LABORATORY AUDITS

11.2.1 Internal Laboratory Audits

The laboratory quality assurance officer has overall responsibility for monitoring the internal QA/QC program.

System audits are conducted on each department at Lancaster Laboratories by members of the Quality Assurance Department on a minimum quarterly basis. The audits may include checks on methodology, reagent preparation, equipment calibration and maintenance, quality control results, and training of personnel. The results of the audits and corrective action, where necessary, are communicated to laboratory personnel and management by means of a written report.

Performance audits consist of both intralaboratory and interlaboratory check samples. QC samples from commercial suppliers are analyzed quarterly to assess laboratory accuracy including a double blind program. The laboratory also participates in a number of interlaboratory performance evaluation studies which involve analysis of samples with concentrations of analytes that are known to the sponsoring organization, but unknown to the laboratory. Lancaster Laboratories has participated in the U.S EPA Contract Laboratory Program which provides laboratory analysis in support of the Superfund Program. Part of maintaining this contract includes analysis of quarterly blind samples.

11.2.2 External Laboratory Audits

An external audits may be conducted by U.S EPA Region V or qualified KMC staff. An external audit may be conducted prior to the initiation of the sampling and analysis activities. These audits may or may not be announced and are at the discretion of the U.S. EPA.

External lab audits will include (but not be limited to) review of laboratory analytical procedures, laboratory on-site audits, and/or submissions of performance evaluation samples to the laboratory for analysis.

SECTION 12

PREVENTIVE MAINTENANCE PROCEDURES

This section describes the specific preventive maintenance procedures to be followed for field equipment and laboratory instruments.

12.1 FIELD EQUIPMENT/INSTRUMENTS

No field measurement data will be generated during background soil and groundwater collection activities. A PID will be used to help characterize potentially contaminated soil and will also be used for health and safety screening purposes. Specific preventive maintenance procedures for the PID are discussed in the SOP in Appendix E, and will be conducted in accordance with manufacturer's specifications.

Field instruments will be checked and calibrated daily before use. Calibration checks will be documented in the field logbook. The FTL will be responsible for implementing and documenting these procedures in the logbook.

Preventive maintenance will normally be conducted by a WESTON Equipment Store representative. Additional maintenance of equipment will be performed at the site, if necessary, by the field sampling personnel on an as-needed or an as-recommended basis. Backup instruments and equipment will be available on-site if deemed necessary, or will be within 1-day shipment to avoid delays in the field schedule.

12.2 LABORATORY INSTRUMENTS

The preventive maintenance program for Lancaster Laboratory will be in accordance with the laboratory's preventive maintenance procedures. In order to ensure timely production of data, Lancaster Laboratories schedules routine preventative maintenance of instruments based on

manufacturer's recommendations. Maintenance of the laboratory instruments is the responsibility of the technical group using the equipment in conjunction with an in-house equipment maintenance group. All preventative maintenance as well as maintenance performed as corrective action is recorded in the laboratory instrument logs. A laboratory QAPP for the Moss-American site is located in Appendix B. Laboratory SOPs are presented in Appendix C.

SECTION 13

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

This section describes the specific routine procedures to be followed to assess data precision, accuracy, and completeness for field measurements and laboratory results.

13.1 FIELD MEASUREMENTS

No field measurement data will be generated during background soil and groundwater collection activities. A PID will be used to help characterize potentially contaminated soil and will also be used for health and safety screening purposes.

13.2 LABORATORY DATA

Laboratory results will be assessed for compliance with required precision, accuracy, completeness, and sensitivity as discussed in the following subsections.

13.2.1 Precision

The degree of agreement between the numerical values of a set of duplicate samples performed in an identical fashion constitutes the precision of the measurement. Precision of laboratory analysis will be assessed by comparing the analytical results between MS/MSD samples for organic analysis and laboratory duplicate analyses for inorganic analysis. Precision will be reported as a relative percent difference (RPD) and will be calculated for each pair of duplicate analysis as follows:

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

Where:

% RPD = Relative percent difference.

S = First sample value (MS for organics and initial sample result for inorganics).

D = Second sample value (MSD for organics and method duplicate for inorganics).

13.2.2 Accuracy

Accuracy is the measure of a result to the accepted (or true) value. Accuracy of laboratory results will be assessed for compliance with the QC criteria that are described in Section 4 of the QAPP, using the analytical results of the method blanks, reagent/preparation blank, MS/MSD samples, field blanks, and trip blanks.

Analytical accuracy is expressed as the percent recovery of an analyte that has been added to the sample or standard matrix (i.e., blank) at a known concentration before analysis. The percent recovery of matrix spike samples will be calculated as follows:

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

Where:

% R = Percent recovery

A = The total analyte concentration determined experimentally from the spiked sample.

B = The background level determined by separate analysis of the unspiked sample.

C = Amount of the spike added.

13.2.3 Completeness

The data completeness of laboratory analyses results will be assessed for compliance with the amount of data required for decision making. Data completeness for laboratory data will be calculated using the following equation:

$$\text{Completeness} = \frac{\text{number of valid data}}{\text{number of samples analyzed for each parameter}} \times 100\%$$

13.2.4 Sensitivity

The achievement of method detection limits depends on instrument sensitivity and matrix effects. Therefore, it is important to monitor the instrument sensitivity to ensure the data quality through constant instrument performance. The instrument sensitivity will be monitored through various means including the analysis of method blank, calibration check sample, and laboratory control samples.

SECTION 14
CORRECTIVE ACTIONS

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

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the WESTON Project Manager or his designee if the problem occurs in the field, or the Laboratory Manager or Laboratory Quality Assurance Officer if the problem occurs in the laboratory. It will be the laboratory manager's responsibility to notify the WESTON Project Manager and/or Project Director and inform them of any problems. Problems will be communicated to the KMC Project Manager and the U.S. EPA RPM by the WESTON Project Manager or his designee. Implementation of corrective actions will be confirmed in writing through the same channels.

Any nonconformance with the established QC procedures in the QAPP or FSP will be identified and corrected in accordance with the QAPP. The WESTON Project Manager or his designee will issue a Nonconformance Report for each nonconformance condition.

Corrective actions will be implemented and documented in the field logbook. No staff member will initiate corrective action without prior communication of findings through the proper channels. Corrective actions will be defined by the auditor and implemented to the satisfaction of the WESTON Project Manager. If corrective actions are insufficient, work may be stopped by a stop-work order issued by the U.S. EPA RPM , the WESTON Project Director, the WESTON Project Manager or the KMC Project Manager.

14.1 SAMPLE COLLECTION/FIELD MEASUREMENTS

Technical staff and project field personnel will be responsible for reporting all suspected technical or QA nonconformance or suspected deficiencies of any activity or issued document by reporting the situation to the FTL or his designee. The FTL will be responsible for assessing the suspected problem, consulting with the WESTON Project Manager on the problem and anticipated change, and implementing the change. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the WESTON Project Manager.

The WESTON Project Manager will be responsible for informing the WESTON Project Director, the KMC Project Manager, the U.S. EPA RPM, and the WDNR Project Manager of the problem. The WESTON Project Manager will be responsible for ensuring that corrective action for nonconformance is initiated by:

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

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

Corrective action for field measurements may include:

- Repeating the measurement to check the error.
- Checking all proper adjustments for ambient conditions such as temperature.
- Checking instruments' batteries.
- Checking instruments' calibration.
- Re-calibration.
- Replacing the instrument or measurement device.
- Stopping work (if necessary).

All changes will be evaluated based on their potential to affect the quality of the data. The WESTON Project Manger has ultimate responsibility for all site activities. In this role, the Project Manager at times is required to adjust the site programs to accommodate site-specific needs. When it becomes necessary to modify a program, the responsible FTL notifies the WESTON Project Manager of the anticipated change and implements the necessary changes. The WESTON Project Manager or his designee must approve all changes verbally or in writing prior to field implementation by the FTL. The WESTON Project Director, the KMC Project Manager, the U.S. EPA RPM, and the WDNR will be notified when any field changes are made.

All problems and corrective actions will be documented in a field logbook by the FTL. No field team member will start corrective action without prior communication of findings through the proper channels. The action taken during the period of deviation will be evaluated in order to determine the significance of any departure from established program practices and action taken. If corrective actions are insufficient, work may be stopped by the FTL following instructions from the U.S. EPA RPM , WESTON Project Director and/or Manager, or the KMC Project Manager.

14.2 LABORATORY ANALYSES

For all analytical work, corrective action will be implemented as specified in the laboratory QAPP and applicable SOPs (Appendix B and C). Corrective action may include:

- Re-analyzing the samples, if holding time criteria permits.
- Resampling and analyzing.
- Evaluating and amending sampling procedures.
- Evaluating and amending analytical procedures.
- Accepting the data and acknowledge the level of uncertainty.

Re-sampling, if necessary, will be conducted if any laboratory problems occur. The U.S. EPA RPM will be notified, as appropriate. The U.S. EPA RPM must also recognize any delay in the project schedule as a result of the re-sampling effort or laboratory errors.

SECTION 15

QUALITY ASSURANCE REPORTS TO MANAGEMENT

The WESTON Project Director and/or Project Manager will audit the implementation of this QAPP. These reviews will include an assessment of data quality, and the results of systems and performance audits as appropriate. These reviews are done to ensure that problems, if any, identified during the sampling and analysis are investigated, and corrective actions are taken properly. The preparation of a QA Report is not anticipated, except as necessitated by problems arising during the execution of project activities. Any QA report prepared by WESTON for the Moss-American site will be submitted to the WESTON Project Director, the KMC Project Manager, and the U.S. EPA RPM. The final project report will include QA information, regardless of whether or not QA problems are observed.

SECTION 16

REFERENCES

Roy F. Weston, Inc. (Weston), 1998. Final (100 Percent) Design for Groundwater Remedial System. March 1998.

U.S. EPA. 1998. Amendment to the Record of Decision for the Moss-American Site, Milwaukee, Wisconsin.

U.S. EPA. 1996. *U.S. EPA Region V Model Quality Assurance Project Plan.* Region V. May 1996.

U.S. EPA. 1994a. *Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses.* EPA Data Review Work Group. February 1994.

U.S. EPA. 1994b. *Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses.* EPA Data Review Work Group. February 1994.

U.S. EPA. 1992. *EPA Requirements for QAPPs for Environmental Data Operation.* EPA QA/R-5. January 1992.

U.S. EPA. 1985. *NEIC Policies and Procedures.* EPA—33019-78-DDI-R. Revised June 1985.

U.S. EPA. 1980. *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans.* QAMS-005/80.

APPENDIX A

FIELD SAMPLING PLAN

FIELD SAMPLING PLAN
FOR INSTALLATION OF GROUNDWATER REMEDIAL SYSTEM
MOSS-AMERICAN SITE
MILWAUKEE, WISCONSIN

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SECTION 1
INTRODUCTION

This document presents the Field Sampling Plan (FSP) for the remedial activities that will be performed as part of the funnel-and-gate groundwater remedial system installation at the Moss-American Site in Milwaukee, Wisconsin (hereafter referred to as the facility). Environmental media to be sampled includes soil and water. Soil sampling will be performed to determine the chemical characteristics of the excavated soil and to confirm that all soil requiring treatment has been removed from the excavations. Water sampling will be conducted to ensure that water removed from the excavations meets the appropriate standards for discharge to Milwaukee Metropolitan Sewerage District's (MMSD) sanitary sewer system.

Specifically, this FSP addresses:

- Sampling plan rationale.
- Number and type of samples to be collected for analysis.
- Procedures for collection of environmental samples.
- Sampling personnel responsibilities.
- Sample identification.
- Sample containers and preservation.
- Sample packaging and shipment.
- Chain of custody.
- Documentation of sampling activities.
- Quality assurance and quality control (QA/QC) of field sampling.

SECTION 2
SAMPLING DESIGN AND RATIONALE

This section discusses the sampling program that will be employed during the construction of the groundwater remedial system at the Moss-American Site. A layout of the groundwater remedial system is shown in Figure 2-1.

2.1 SAMPLING DESIGN AND RATIONALE

The sampling program has been designed to characterize the excavated soil for appropriate management, to confirm that soils requiring treatment have been removed from the excavations, and to verify that water generated during the remedial activities meets Milwaukee Metropolitan Sewerage District's (MMSD's) sanitary sewer discharge standards. Table 2-1 summarizes the anticipated sampling effort. Details of the sampling program are discussed in the following subsections.

2.1.1 Characterization Sampling

Characterization sampling will be necessary to segregate the soils that are generated during the construction of the groundwater remedial system. During construction, soil will be generated due to the following activities:

- Excavation of Areas T1, T2, and T3.
- Installation of Treatment Gates TG1 through TG6.
- Installation of groundwater monitoring and free-product extraction wells.
- Installation of underground piping.

Soils other than clean soil would require management that is consistent with the cleanup standards and remedial alternatives established in the Amended Record of Decisions (ROD) for

the site. Consequently, all excavated soil will be classified as:

- Soil that will require treatment.
- Soil that will not require treatment but exceeds the generic migration to groundwater cleanup standard of 0.4 mg/kg for naphthalene.
- Soil that will not require treatment but will require an appropriate cover consistent with the ROD requirements (i.e. soil that exceeds the direct contact values for total CPAHs).
- Clean soil (i.e. soil that meets all the soil cleanup standards).

Soils that would require treatment would be excavated primarily from Areas T1, T2, and T3. Excavated soil that will require treatment will include:

- Soil that contains free product.
- Soil that exceeds a benzo(a)pyrene equivalent residual contaminant level (RCL) of 78 mg/kg for total carcinogenic polycyclic aromatic hydrocarbons (CPAHs).
- Soil that exceeds the generic migration to groundwater cleanup standards for fluorene and benzo(a)pyrene under Table 1 of WDNR Publication RR-519-97 (Soil Cleanup Levels for Polynuclear Aromatic Hydrocarbons [PAHs] Interim Guidance).
- Soil that exceeds the naphthalene concentration of 100 mg/kg. [Please note that the 100 mg/kg value for naphthalene is neither a new standard nor a new RCL. It represents a value supported by U.S. EPA and KMC at this time that would facilitate attainment of acceptable groundwater naphthalene concentrations in the future.]
- Soil that exceeds generic migration to groundwater standards for benzene, toluene, ethylbenzene, and xylene (BTEX) as presented in NR 720.19.

In addition to the soil requiring treatment, debris (i.e., oversized material, railroad ties), and soil that exceeds various land use based direct contact values for total CPAHs (benzo(a)pyrene equivalent or actual concentrations depending on the location outside or within the floodplain, respectively) would also be generated during the construction activities. The amended ROD

requires that soil that exceeds direct contact risk levels for total CPAH concentrations corresponding to specific land uses (i.e., recreational, industrial, etc.) be appropriately capped. Since the land use for the site has not yet been finalized, use of a direct contact value for a specific land use may not be justifiable. Nevertheless, for the purposes of this QAPP a residential land use for the entire site has been assumed. Consequently, in accordance with the amended ROD, soil that exceeds the direct contact values of 1.9 mg/kg for total CPAHs (BAP equivalent) in areas outside the 100-year floodplain and 15 mg/kg for total CPAHs (actual) in areas within the 100-year floodplain will require capping consistent with the requirements of the amended ROD. The direct contact value for soil associated with areas outside the 100-year floodplain may change if an alternative land use (i.e., other than residential land use) is established for the site. Soils that do not require treatment, do not exceed the generic migration to groundwater cleanup standard of 0.4 mg/kg for naphthalene, and do not exceed the direct contact values for total CPAHs will be considered clean.

Debris as well as soil that contains free-product will be immediately transferred to the existing asphalt storage pad. It is assumed that soil containing free-product will require treatment. Therefore, this soil will undergo neither preliminary screening nor characterization sampling. The rest of the excavated soil will undergo preliminary screening with a photoionization detector (PID). In addition, visual and olfactory observations will be used to screen the excavated soil for contamination. Based on the results of the field screening, excavated soil will be segregated into soil that appears contaminated and soil that appears clean. These soils will be stored in separate stockpiles and will undergo further characterization.

As approved in the Final (100 Percent) Design for Groundwater Remedial System (Weston, 1998), samples will be collected at a frequency of one grab sample per 200 cy of stockpiled soil. It is estimated that approximately 20,000 cubic yards of soil would be generated during the construction of the groundwater treatment system. Of this volume, 10,200 cubic yards of soil would be excavated from Areas T1, T2, and T3 and 9300 cubic yards would be generated during the installation of Treatment Gates TG1 through TG6. The rest of the soil (approximately 500

cubic yards) would be generated due to activities such as installation of wells and piping. This excavation volume also includes a 25 percent swell factor. Based on the expected volume of 20,000 cubic yards, approximately 100 characterization samples would be collected. In addition, appropriate quality control and quality assurance (QA/QC) samples will be collected. The total number of samples including the QA/QC samples and the sampling frequencies are shown in Table 2-1. Please note that the total number of samples are based on certain assumptions and will vary with the volume of the excavated soil as well as with the volume of soil exhibiting the presence of free product.

All samples will be analyzed for polyaromatic hydrocarbons (PAHs), and for benzene, ethylbenzene, toluene, and xylene (BTEX). All analytical methods will be in accordance with the methods specified in the Quality Assurance Project Plan (QAPP).

In order to characterize the excavated soil, analytical results of the samples will be compared to the appropriate soil cleanup standards established for the site. Results of this comparison will be used to classify the soil as stated previously. Soil that will require treatment will be staged on the existing asphalt pad and will undergo treatment in a low thermal temperature desorption (LTTD) system at a future date. Soil that will not require treatment but will exceed the generic migration to groundwater cleanup standard of 0.4 mg/kg of naphthalene and direct contact values for total CPAHs (BAP equivalent or otherwise) will be staged in the area shown in Figure 2-1 for future management consistent with the requirements of the amended ROD. Clean soil (i.e., soil that meets all the soil cleanup standards) will be used as backfill material.

2.1.2 Verification Sampling

Verification sampling will be conducted to verify that all soil requiring treatment has been removed from open excavations. Excavations subject to verification sampling will include excavations that will result from the excavation of Areas T1, T2, and T3 and installation of Treatment Gates TG1 through TG6.

As approved in the Final (100 Percent) Design for Groundwater Remedial System (Weston, 1998), soil samples from the sidewalls and floors of excavations associated with Areas T1, T2, and T3 will be collected at a frequency of one grab sample per 50 linear feet. One grab sample each from the sidewalls and floors of excavations associated with Treatment Gates TG1 through TG6 will be collected. All samples collected from the excavation sidewalls will be located at approximately two-thirds of the depth of the excavation. This sampling strategy will be modified in instances where excavation of Areas T1, T2, and T3 and Gates TG1 through TG6 overlap. Figure 2-1 indicates the approximate locations of the verification samples. As shown in Figure 2-1, approximately 84 verification samples would be collected. In addition, appropriate Quality Control and Quality Assurance (QA/QC) samples will be collected. The total number of samples including the QA/QC samples and the sampling frequencies are shown in Table 2-1. Please note that the total number of samples are based on certain assumptions and will vary with the size of excavations associated with Areas T1, T2, and T3.

All soil samples will be analyzed for PAHs and BTEX to confirm that all soil requiring treatment has been excavated. All analytical methods will be in accordance with the methods specified in the QAPP.

2.1.3 Water Sampling

Contaminated water resulting from infiltration of groundwater or precipitation entering the excavations, or precipitation which comes in contact with the contaminated soil will be collected, treated and discharged to MMSD's sanitary sewer system. Contaminated water from the excavations as well as from the asphalt storage used for storing soil requiring treatment will be pumped to two 10,000 gallon aboveground storage tanks (ASTs). These ASTs are part of the existing free-product recovery system. It is estimated that approximately 8 gpm (11,520 gpd) will enter a typical excavation (approximately 65 feet by 55 feet) via groundwater infiltration. In addition, assuming a 25-year 24-hour storm event results in approximately 4.5 inches of

precipitation, the volume of precipitation within the excavation would be approximately 9,700 gallons. Thus, the total volume of water requiring storage would be 21,200 gallons. Although this volume exceeds the total capacity of the ASTs, it is assumed that the excavation water will be pumped and treated continuously during the excavation activities. In addition, to the extent practicable, each excavation or a portion of the excavation will be backfilled at the end of the work day. Based on this assumption, the capacity of the ASTs would be adequate for storing both the groundwater infiltration and the precipitation from a 25-year 24-hour storm event. However, if additional storage is required, portable storage tanks will be used.

Water collected in the tanks will be treated with a portable water treatment system to meet MMSD's discharge requirements. After treatment, the water will be transferred to tanker trucks for transportation and discharge to the sanitary sewer located along Granville road.

The exact duration of excavation activities is currently unknown. Nevertheless, it has been assumed that it would take approximately one month to complete all excavation activities. Based on this assumption, approximately 660,000 gallons of water would be generated during the construction of the groundwater treatment system. Based on the frequency of one grab sample per 10,000 gallons of treated water, approximately 66 water samples would be collected. In addition, appropriate QA/QC samples will be collected. The total number of samples including the QA/QC samples and the associated sampling frequencies are shown in Table 2-1. Please note that the total number of samples are based on certain assumptions and will vary with the duration of excavation activities and consequently, on the volume of water generated.

Prior to discharge, all samples will be analyzed for parameters that will satisfy MMSD's discharge requirements. Parameters will include volatile organic compounds (VOCs), PAHs, total metals including cadmium, copper, lead, mercury, nickel, silver, and zinc, cyanide, oil and grease, and total suspended solids (TSS). All analytical methods will be in accordance with the requirements of the MMSD.

Table 2-1

Summary of Sampling Effort
Moss-American Site
Milwaukee, Wisconsin

Sample Matrix	Laboratory Parameters	Characterization/ Verification Samples			Field Duplicate Samples			Field Blank Samples			Matrix Spike/Matrix Spike Duplicate Samples			Matrix Total[1]
		No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	
Soil	BTEX[2]	184	1	182	19	1	19	--	--	--	10	1	10	203
	PAHs[3]	184	1	182	19	1	19	--	--	--	10	1	10	203
Water	VOCs[4]	66	1	66	7	1	7	7	1	7	7	1	7	87
	PAHs[3]	66	1	66	7	1	7	7	1	7	7	1	7	87
	Total Metals	66	1	66	7	1	7	7	1	7	7	1	7	87
	Total Mercury	66	1	66	7	1	7	7	1	7	7	1	7	87
	Total Cyanide	66	1	66	7	1	7	7	1	7	7	1	7	87
	TSS[5]	66	1	66	7	1	7	7	1	7	7	1	7	87
	Oil & Grease	66	1	66	7	1	7	7	1	7	7	1	7	87
	pH	66	1	66	7	1	7	7	1	7	7	1	7	87

1 – Matrix Total includes 100 characterization samples, 84 verification samples, and 19 field duplicates samples. It does not include trip blank or matrix spike/matrix spike duplicate (MS/MSD) samples.
2 – Benzene, Toluene, Ethylbenzene, Total Xylenes.
3 – Polynuclear Aromatic Hydrocarbons.
4 – Volatile Organic Analytes.
5 – Total Suspended Solids.

Note: Trip blank samples will be included in each shipment of aqueous VOA samples. MS/MSD samples are not additional samples, MS/MSD samples are characterization/verification samples that are to undergo a MS/MSD analysis.

SECTION 3
FIELD SAMPLE COLLECTION PROCEDURES

3.1 SAMPLE COLLECTION PROTOCOLS

The following subsections discuss the procedures that will be employed at the Moss-American Site for collection of samples from environmental media.

3.1.1 Soil Sampling Protocol

Soil samples for characterization sampling will be collected from soil stockpiles. Soil verification samples from the excavation sidewalls and floors will be collected directly from the excavator bucket.

All soil samples will be grab samples. Soil samples will be collected using dedicated and disposable sampling equipment (i.e., scoops, gloves, mixing bowls, etc.), and placed into precleaned sample containers provided by the laboratory. BTEX samples will be collected first followed by PAH samples.

3.1.2 Water Sampling Protocol

Samples of treated water will be collected for characterization analysis. Samples will be collected from a sampling port and placed directly into the precleaned sample containers. Samples designated to undergo analysis for VOCs will be collected first. VOC sample containers will be filled at an angle to minimize the potential for volatilization of organic compounds. If bubbles appear after the bottle is capped, a new preserved VOC container will be used to collect the sample. After the VOC samples have been collected, the remaining parameters will be collected in decreasing order of sensitivity (i.e., semi-volatiles, metals, cyanide, oil and grease and TSS).

3.2 FIELD QUALITY CONTROL SAMPLES

Four types of quality control (QC) samples will be collected during the sampling activities:

- Field duplicates.
- Field blanks.
- Matrix spike/matrix spike duplicates.
- Trip blanks.

The purpose for collection of each QC sample is explained in Section 4 of QAPP. The specific level of QC effort for the Moss-American Site is summarized in Table 2-1, and the procedures for collection of the QC samples is detailed in the following subsections.

3.2.1 Field Duplicate Samples

Field duplicate samples will be collected at a frequency of 1 duplicate sample per 10 characterization/verification samples collected for both soil and water matrices. Duplicate samples will be collected identically to the characterization/verification samples of the same matrix. Field duplicate samples will be analyzed for the same constituents as their characterization/verification counterparts. At each duplicate soil sampling location, sufficient sample media for both the characterization/verification and duplicate samples will be collected. The duplicate sample will be collected concurrently with the characterization and/ or verification sample.

3.2.2 Field Blank Samples

Field blanks will be collected at a frequency of 1 field blank sample per 10 characterization/verification samples collected. Field blanks will be collected by filling the sampling containers with ultra-pure (laboratory grade) water at the treated water sampling point.

All sample handling, volume, packaging, and preservation techniques used for the characterization water samples will be applied to the field blank samples. The field blank samples will be analyzed for the same parameters as their characterization water sample counterparts, in accordance with the same analytical methodologies. The field blank samples will be documented and identified as such on all sample documentation.

3.2.3 Matrix Spike/Matrix Spike Duplicate Samples

Matrix spike/matrix spike duplicate (MS/MSD) samples will be collected at a frequency of 1 MS/MSD sample per 20 characterization/verification samples collected. NOTE: MS/MSD samples are not additional samples, these are characterization/verification samples designated to undergo MS/MSD analysis. Therefore, all sample collection procedures used for collecting MS/MSD samples will be identical to the procedures used for collecting characterization/ verification sample collection. No additional sample volume is required for soil MS/MSD samples. Each MS/MSD sample will be identified on the chain-of-custody form and will be shipped to the analytical laboratory for all scheduled analyses. Trip blanks and field duplicate samples will not be used as MS/MSD samples.

3.2.4 Trip Blanks

One trip blank will be enclosed in each sample shipment container in which aqueous VOC samples are included. All sample handling, volume, packaging, and preservation techniques used for the characterization water samples will be applied to the trip blank samples. The trip blanks will be obtained by pouring ultra-pure water directly into the pre-preserved sample containers in a laboratory-type environment prior to beginning of a sampling event or . The trip blanks will then be subsequently placed in a sample shipment container. The trip blank samples will be documented and identified as such on all sample documentation. The trip blank samples will be analyzed for VOCs in accordance with the same analytical methodology as the characterization water samples.

3.3 DECONTAMINATION REQUIREMENTS

Where possible, dedicated and disposable sampling equipment will be employed for sample collection; however, if reusable digging/sampling equipment is used, the reusable equipment will be decontaminated between collection of each sample according to the procedures outlined in Table 3-1.

3.4 ANALYTICAL METHODS

Section 8 of the QAPP discusses the analytical methodology by which the soil and water samples will be analyzed.

Table 3-1

Standard Decontamination Protocol for Field Equipment
Moss-American Site
Milwaukee, Wisconsin

STEP 1	-	Scrub equipment thoroughly with a soft-bristle brush in a low-sudsing detergent solution. (Phosphate-free detergent will be used)
STEP 2	-	Rinse equipment with tap water by submerging and/or spraying.
STEP 3	-	Rinse equipment with solvent (isopropanol) by spraying until dripping; retain rinsate*.
STEP 4	-	Rinse equipment by spraying with deionized water until dripping.
STEP 5	-	Place equipment on polypropylene or aluminum foil and allow to air-dry.
STEP 6	-	Wrap equipment in polypropylene or aluminum foil for handling and/or storage until next use.

* - All isopropanol rinsate will be collected and containerized in a drum or equivalent vessel, staged onsite with other wastes, and properly disposed of at a licensed facility following the completion of all fieldwork.

SECTION 4
SAMPLE NUMBERING SYSTEM

All samples for analysis, including QC samples, will be given unique sample numbers. A listing of sample numbers, cross-referenced to chain-of-custody and shipment documents, will be maintained in the sample handling logbook.

Two identification numbers will be used for each soil and water sample, a WESTON project sample number and an analytical laboratory sample identifier.

The WESTON project sample number, which highlights the sample matrix and location, will be used for presentation of the data in memoranda and reports. The laboratory identifier is assigned by the laboratory custodian at the time of sample receipt and is the primary means of tracking a sample through the laboratory.

4.1 PROJECT SAMPLE NUMBERING SYSTEM

The project sample numbers will be composed of three components, which are described below:

- Project Identifier. A three-character designation will be used to identify the facility for which the samples will be collected. For this project, it will be MA3. MA stands for Moss-American Site, and the numerical designation (1, 2, 3...) refers to the phase of the project.

- Sample Type and Location. A two-character type code (SS for soil and TW for treated water) will be the prefix for the sample designating the sample media.

For samples collected from the stockpiled soil, the two-character matrix code will be followed by a one-character, two-digit code, i.e., C01. The character identifies the type of stockpile the soil sample was collected from (C = clean soil stockpile, S = stained soil stockpile). NOTE: Soil samples will **not** be collected from the stockpiled soil exhibiting the presence of free product, as this soil will be assumed to require treatment. The two digit code refers to the stockpile number

(01, 02, 03, ...99). For QC samples, the stockpile code will be followed by "D" for field duplicate sample and by "M" for matrix spike/matrix spike duplicate sample.

For verification soil samples, this code will be followed by a one-digit, one-character (i.e., 1F or 2W) code that indicates the excavation the sample was collected from (1 = Area T1, 2 = Area T2, and 3 = Area T3) and where in the excavation the sample was collected from (F = excavation floor and W = excavation sidewall). This code will be followed by a coordinate (i.e., 500N-1400E) that indicates where the sample was collected from with respect to the north-east coordinate system established during the pre-design activities. For QC samples, the coordinate code will be followed by "D" for field duplicate sample and by "M" for matrix spike/matrix spike duplicate sample. (It should be noted that all field duplicate samples will be submitted "blind" to the laboratory. Only field personnel will be acquainted with the sample nomenclature system.)

For water samples, the two-character matrix code will be followed by a one-character, six-digit code (i.e., N010199). The character will be either N or S, and indicates whether the sample was collected from the north (N) or south (S) treated water holding tank. The six-digit code indicates the date (day, month, and year) the sample was collected on (i.e., 030599 refers to 3 May 1999). For duplicate and MS/MSD water samples, the date code will be followed by a D or M, respectively. For trip blank samples, the N or S code will be replaced with TB and the date code would indicate the day the sample was prepared.

- Sequence. For soil samples, a two-digit number will be used to indicate the first, second, third, etc., sample collected at a given location during a phase of the project. For water samples, the sequence code would indicate the sample number on a daily basis (i.e., 02 would indicate the second sample on a given day).

Some examples of the project sampling number system are as follows.

Soil Samples Collected for Characterization Sampling

- MA3-SSS05 reads as:
  - Moss-American Site, phase 3.
  - Soil sample collected from Stained soil Stockpile number 5.
- MA3-SSS05D reads as:
  - Duplicate of above sample numbering example.
- MA3-SSS05M would be the sample identifier if the first sample example was designated as an MS/MSD sample.

Soil Samples Collected from Verification Sampling

- MA3-SS3W-200N-1000E-01 reads as:
  - Moss-American Site, phase 3.
  - Soil sample collected from the Area T3 excavation sidewall.
  - Grid coordinates 200N-1000E.
  - First sample at this location.
- MA3-SS3WD-200N-1000E-01 reads as:
  - Duplicate of first sample numbering example.
- MA3-SS3WM-200N-1000E-01 would be the sample identifier if the first sample numbering example was designated as an MS/MSD sample.

Treated Water Samples

- MA3-TW-N020700-01 reads as:
  - Moss-American Site, phase 3.
  - Treated water sample.
  - Collected from the north holding tank on 2 July 2000.
  - First sample of the day collected from the north holding tank.

- MA3-TW-N040700D-01 reads as:
  - Duplicate of above sample numbering example.
- MA3-TW-N040700M-01 would be the sample identifier if the first sample example was designated as an MS/MSD sample.
- MA3-TW-TB040700-01 would be the first trip blank sample prepared on 4 July 2000.

4.2 LABORATORY SAMPLE IDENTIFIER

Upon arrival at the laboratory, the WESTON batch number will be recorded by the laboratory custodian/sample log-in person on the chain-of-custody form and on the bottle label using a permanent marker. The laboratory will provide a sample identifier for tracking throughout the analytical process. This number will be identified on all data reports and will be cross-referenced to the WESTON project number.

SECTION 5
SAMPLE HANDLING

5.1 SAMPLE CONTAINERS AND SAMPLE PRESERVATION

All soil samples are expected to be low to moderate hazard level. Table 5-1 lists the required sample containers, sample volumes, sample preservation requirements, and holding times associated with all parameters and media applicable to the Moss-American Site sampling activities during the installation of the funnel-and-gate groundwater treatment system.

5.2 SAMPLE PACKAGING AND SHIPMENT

Following sample collection, the exteriors of all sample containers will be wiped clean with a moist cloth. The filled sample containers will not be sprayed with water during decontamination since water could contact the sample if the container is not tightly sealed. In preparation for shipment to the analytical laboratory, all samples will be packaged in accordance with the following procedures:

- Each sample container will be checked to ensure that the container lid is securely tightened.
- Each sample container will be checked to ensure that the sample label has been securely affixed to the container and completely/correctly filled out with the appropriate sample ID number, sample date, sample time of collection, and analytical parameters as a minimum requirement.
- Each container will be placed in a separate zip-lock bag and the bag securely closed (eliminating most of the air from within the bag prior to sealing the bag).
- The samples will be placed in a cooler lined with a large polyethylene bag. Enough vermiculite or equivalent absorbent material will be packed around the samples to minimize the possibility of container breakage. The temperature will be maintained at 4°C with cold packs or ice, sealed in plastic bags. The remaining space in the cooler will be filled with additional packing material. Upon completion of packing the cooler, the large polyethylene bag will be sealed.

- The completed chain-of-custody form identifying the contents of the sample shipment container will be placed in a large zip-lock bag and taped to the inside lid of the shipment container (the sampler's copy of the form will first be removed).

- The cooler lid will be closed and sealed shut with strapping tape. If the cooler has a drain port, it will also be sealed shut with tape. Two chain-of-custody seals will be placed across the seam between the cooler lid and base. The seals will be placed in a staggered configuration (either front left side and back right side or vice versa). This will ensure that if the cooler is opened by unauthorized persons, the custody seal will break and indicate intrusive action. The custody seals will be covered with waterproof tape to prevent accidental damage during shipment.

- The shipment airbill will be affixed to the top of the cooler. It will identify the shipper's and recipient's names and addresses. A WESTON mailing label will also be affixed to the top of the cooler and will contain the same information as the airbill in case the airbill becomes detached from the cooler during shipment.

- "This Side Up" arrows will be placed on the four sides of the shipment container.

- All samples will be shipped within 24 hours of collection. All samples will be shipped via overnight delivery.

Sample handling, packaging, and shipment activities are the responsibility of the assigned WESTON Field Sample Manager (FSM); however, all field samplers will assist as necessary. The FSM will provide the WESTON Field Team Leader (FTL) with the retained copies of the chain-of-custody forms and airbills. The FTL will be responsible for updating the WESTON Project Manager on sample management activities. The FTL will also be responsible for contacting the Lancaster Laboratory Project Manager or his/her designee and informing him/her of each shipment of samples. At a minimum, the FTL will provide the following information:

- Site name.
- Number of samples shipped.
- Number of coolers shipped.
- Date samples were shipped.
- Date samples should be received.
- Shipment airbill number(s).

Table 5-1

Required Sample Volume, Containers and Sample Preservation
Moss-American Site
Milwaukee, Wisconsin

Sample Matrix	Analysis	No. of Containers	Container Type	Preservatives	Holding Time
Soil	PAH	1	16-oz. clear glass wide-mouth (Teflon-lined cap)	Cool, 4°C	14 days to extract; analyze within 40 days of extracting
	BTEX	3	5 gram Encore sampler	Cool, 4°C. Lab must add preservative within 48 hours.	14 days.
Treated Water	VOC	2	40-mL vials	HCL to pH <2 Cool, 4°C	14 days
	PAH	2	1-liter amber glass (Teflon-lined lid)	Cool, 4°C	7 days to extract; analyze within 40 days of extracting
	Total Metals	1	1-liter HDPE	HNO_3 to pH <2 Cool, 4°C	6 months
	Total Mercury	1	1-liter HDPE	HNO_3 to pH <2 Cool, 4°C	28 days
	Total Cyanide	1	1-liter HDPE	NaOH, pH >12 Cool, 4°C	14 days
	Oil and Grease	1	32-oz. clear glass, wide-mouth (Teflon-lined lid)	H_2SO_4 to pH<2 Cool, 4°C	28 days
	TSS	1	1-liter HDPE	Cool, 4°C	7 days
	pH	1	1-liter HDPE	Cool, 4°C	24 hours

Notes: No additional soil volume is required for analysis of MS/MSD (organics) or spikes and duplicates (inorganics) with the exception of soil volatiles. Double volume will be required for each soil volatile MS/MSD. Aqueous MS/MSD samples will require triple the normal volume for VOAs and double the normal volume for PAHs. Spike and duplicates for aqueous inorganic samples will require double the normal volume. One trip blank will accompany each shipment of aqueous VOA samples. Trip blanks will be collected in two 40-ml glass vials. No trip blanks will be collected for soil samples or inorganic or extractable analyses. Percent moisture (water content) for soil volatile organic analyses will be determined from the sample volume collected for analysis of PAHs.

SECTION 6
SAMPLE DOCUMENTATION AND TRACKING

6.1 FIELD RECORDS

Field observations and other information pertinent to the collection of samples will be recorded in the field. All entries will be made in a bound logbook with black or blue ink. Logbooks will be identified by unique sequential numbers. The data to be recorded for each sample will include date, time (military time reference), sample number, sample location, and name of the person(s) collecting the sample. In addition, general information will be recorded in the logbook daily, including personnel present at the site, level of protection being worn, and weather. Photographs will be taken and logged to document sampling activities.

6.2 FIELD CHAIN-OF-CUSTODY PROCEDURES

Field chain-of-custody procedures are discussed in Subsection 6.2 of the QAPP.

6.3 SAMPLE DOCUMENTATION FORMS

The primary form of sample documentation for the Moss-American Site sampling activities is the Lancaster Laboratory chain-of-custody form (also referred to as the Analysis Request/ Environmental Services Chain of Custody). In addition, as previously mentioned, chain-of-custody seals and sample container labels will be utilized. An example Lancaster Laboratory Chain of Custody Form and sample label are provided in section 7 of the laboratory QAPP (Appendix B). The important protocols associated with each of these is summarized below:

Chain-of-Custody Form

- Each shipment cooler will be accompanied by a chain-of-custody form(s) documenting contents. The information on the chain-of-custody form will include project sample identification numbers; sample matrix; sample collection date; analysis required; type and number of sample containers per sample; and preservatives (if any).

- Carrier service does not need to sign the form if the chain-of-custody seals remain intact. The airbill number and the chain-of-custody seal numbers should be written on the chain-of-custody form.

- Every sample in the associated cooler will be documented on the chain-of-custody form.

- The facility name and associated project work order number will also be written on the chain-of-custody form.

- The FTL or his/her designee will sign and date the chain-of-custody form as relinquisher of the samples.

Custody Seals

- Two seals per shipping container are used to secure the lid and provide evidence that samples have not been tampered with. All seals will be pre-numbered. Each set of seal numbers will be recorded on the chain-of-custody form.

- The seals will be covered with clear tape after being affixed to the shipping container to prevent inadvertent damage during transport.

- The seal numbers will be recorded on the enclosed chain-of-custody form(s) and in the field log book.

- Seals will be used on all shipping containers containing facility samples.

Sample Bottle Labels

- Each sample container will have a sample label affixed to its outer surface.

- Each sample label will contain the WESTON project sample number, the date of sample collection, the analytical requirements, and the time of sample collection.

- All information on the sample label will be checked with the information on the chain-of-custody form to confirm accuracy and consistency between documents.

Once the FSM has turned over the sample paperwork to the FTL, it is the responsibility of FTL to maintain all the paperwork and to be able to account for all forms at the end of field work.

SECTION 7
SAMPLING TEAM ORGANIZATION

The Moss-American Site field team organization is presented in Subsection 3.3 of the QAPP.

SECTION 8
SAMPLE CONTAINER PROCUREMENT

Sample containers will be procured from the analytical laboratory. All bottle lot numbers associated with each sample collected during the Moss-American Site sampling program will be recorded. The laboratory SOP for sample containers is provided in Appendix D.1.

It will be assured that the sample containers used for the Moss-American Site sampling activities do not contain target organic and inorganic contaminants exceeding the levels specified in the aforementioned document. Specifications for the bottles will be verified by checking certified statement and analytical results for each bottle lot, and will be documented on a continuing basis. This data will be maintained in the project evidence file and will be available, if requested, for U.S. EPA review.

Corrective actions will be taken as soon as a problem is identified. This will be accomplished either by discontinuing the use of a specific bottle lot, requesting the laboratory for new bottles, resampling the suspected samples, validating the data taking into account that the contaminants could have been introduced by the laboratory (i.e., common lab solvents, sample handling artifacts, etc.) or could be a bottle QC problem, so as to make an educated determination of whether the bottles and hence the data are still usable, etc., whichever is appropriate.

SECTION 9
MANAGEMENT OF SAMPLING-DERIVED WASTES

For purposes of this FSP, wastes derived from the sampling of environmental media are defined as any by-product of the field activities that is known or suspected to be contaminated with hazardous substances. The performance of field activities will produce waste products such as decontamination rinsate and disposable sampling and personal protective equipment (PPE).

In order to collect isopropanol rinsate from decontamination of any reusable sampling equipment, if applicable, a portable or temporary decontamination pad will be set up on-site. Isopropanol rinsate will be pumped from the decontamination pad, collected, containerized in Department of Transportation (DOT) approved containers, and disposed of at an off-site, licensed facility.

All disposable PPE and sampling equipment will be containerized in DOT-approved containers for storage until completion of the field activities. All storage containers will be appropriately labeled and stored on-site in a secure staging area until disposed of off-site at a licensed facility in accordance with the U.S. Environmental Protection Agency (U.S. EPA) document *Management of Investigation-Derived Wastes During Site Inspections* (U.S. EPA, 1991).

APPENDIX B

LANCASTER LABORATORIES QAPP

LABORATORY QUALITY ASSURANCE PLAN

Kerr-McGee Chemical Corp.
Móss-American Site
Milwaukee, Wisconsin

August 26, 1993
Revised June 14, 1999

Lancaster Laboratories
A division of Thermo Analytical Inc.

1. Laboratory Quality Assurance Plan

This document provides the laboratory portion of the response to EPA's "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" QAMS-005/80, Sections 5.1 - 5.16 as revised December 29, 1980, and EPA-600/4-83-004, February 1983. Guidance was also obtained from "Preparation Aids for the Development of Category 1 Quality Assurance Project Plans," Office of Research and Development, USEPA, EPA/600/8-91/003, February 1991.

As much as possible, the procedures in this document have been standardized to make them applicable to all types of environmental monitoring and measurement projects. However, under certain site-specific conditions, all of the procedures discussed in this document may not be appropriate. In such cases it will be necessary to adapt the procedures to the specific conditions of the investigation.

Quality Assurance Officer: _____

3. Project Description

This quality assurance project plan provides specific quality assurance and quality control procedures involved in the generation of data of acceptable quality and completeness. Tests will be performed according to the analytical methodology set forth in the USEPA SW-846 3rd Edition, Update III, 1996. SW-846 provides specific analytical procedures to be used and defines the specific application of these procedures. Proven instruments and techniques will be used to identify and measure the concentrations of volatiles and PAH compounds. The laboratory will employ state-of-the-art GC/MS, HPLC, and/or GC procedures to perform all organic analyses, including all necessary preparation for analysis. Wet Chemical analyses will be performed according to Methods for the Chemical Analysis of Water and Wastes, USEPA 600/4-79-020 and will use appropriate instrumentation. The client is responsible for providing specifics on the project site.

*Test Methods for Evaluating Solid Waste - Physical/Chemical Methods. SW-846 (3rd Edition, Update III, September 1996).

4. Project Organization

The objectives of the laboratory Quality Assurance Program are to establish procedures which will ensure that data generated in the laboratory are within acceptable limits of accuracy and precision, to ensure that quality control measures are being carried out, and to ensure accountability of the data through sample and data management procedures. To this end, a Quality Assurance Department has been established. The Quality Assurance Officer reports directly to the President of Lancaster Laboratories and has no direct responsibilities for data production, thus avoiding any conflict of interest.

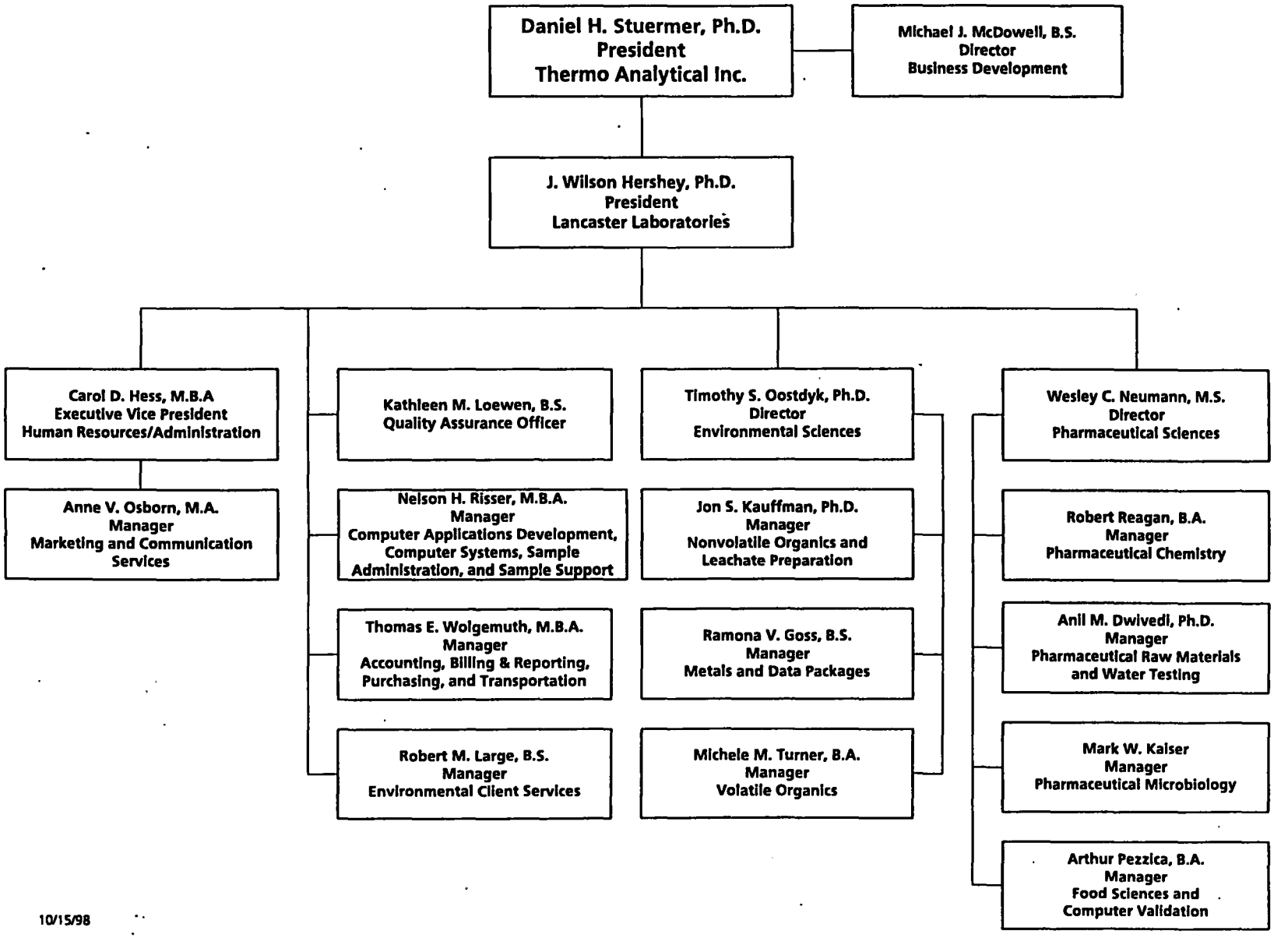
The attached organizational charts show key managerial personnel. Resumes of key individuals may be found in the enclosed Qualification Manual.

The Sample Administration Group will be responsible for receiving samples, signing the external chain-of-custody, checking sample condition, assigning unique laboratory sample identification numbers, and initiating internal chain-of-custody forms. Sample Support personnel will be responsible for assigning storage locations, checking and adjusting preservation, homogenizing the sample as needed, and sample discard.

Group Leaders listed in each technical area are responsible for performing laboratory analyses, quality control as specified in the methods, instrument calibration, and technical data review. Data is reported using a computerized sample management system, which tracks sample progress through the laboratory and generates client reports when all analyses are complete. Quality control data is entered onto the same system for purposes of charting and monitoring data quality.

The Quality Assurance Department is responsible for reviewing quality control data, conducting audits in the laboratory and reporting findings to management, maintaining current copies of all analytical methods, maintaining copies of computer code used to calculate and report results, submitting blind samples to the laboratory, and ensuring that appropriate corrective action is taken when quality problems are observed.

Data package deliverables are available upon request. The Quality Assurance Department reviews the contents of the deliverables for completeness and to be sure that all quality control checks were performed and met specifications. This step includes review of holding times, calibrations, instrument tuning, blank results, duplicate results, matrix spike results, surrogate results, and laboratory control samples (where applicable). Every attempt to meet specifications will be made, and any item outside of the specifications will be noted in the narrative. The laboratory will not validate data with regard to useability since this generally requires specific knowledge about the site.


Daniel H. Stuermer, Ph.D.
President
Thermo Analytical Inc.
Michael J. McDowell, B.S.
Director
Business Development
J. Wilson Hershey, Ph.D.
President
Lancaster Laboratories
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Accounting, Billing & Reporting,
Purchasing, and Transportation
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Leachate Preparation
Ramona V. Goss, B.S.
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Wesley C. Neumann, M.S.
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Robert Reagan, B.A.
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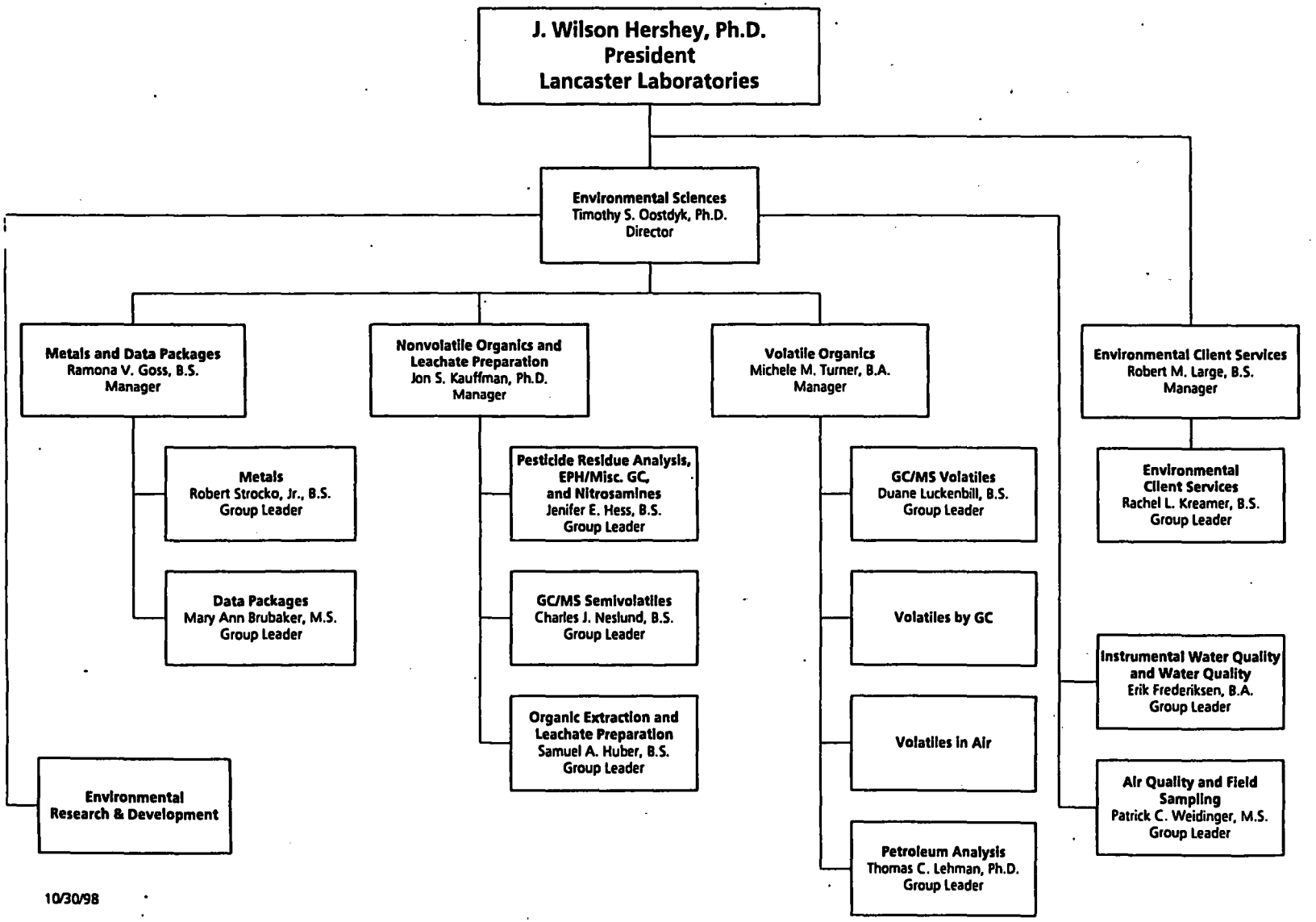
Anil M. Dwivedi, Ph.D.
Manager
Pharmaceutical Raw Materials
and Water Testing
Mark W. Kaiser
Manager
Pharmaceutical Microbiology
Arthur Pezzica, B.A.
Manager
Food Sciences and
Computer Validation

Environmental Sciences



10/30/98

5. QA Objectives For Measurement Data

Quality Assurance is the overall program for assuring reliability of monitoring and measurement data. Quality control is the routine application of procedures for obtaining set standards of performance in the monitoring and measurement process. Data quality requirements are based on the intended use of the data, the measurement process, and the availability of resources. The quality of all data generated and processed during this investigation will be assessed for Precision, Accuracy, Representativeness, Comparability, and Completeness. These specifications will be met through precision and accuracy criteria as specified in Section 11. Detection limits are presented in Section 9.

Precision - Precision is determined by measuring the agreement among individual measurements of the same property, under similar conditions. The laboratory objective is to equal or exceed the precision demonstrated for the applied analytical method on comparable samples. The degree of agreement is expressed as the relative percent difference (RPD%). Evaluation of the RPD% is based on statistical evaluation of past lab data or guidelines within the methods for organic and inorganic analyses. External evaluation of precision is accomplished by analysis of Standard Reference Material and interlaboratory performance data.

Accuracy - Accuracy is a measure of the closeness of an individual measurement to the true or expected value. Analyzing a reference material of known concentration or reanalyzing a sample which has been spiked with a known concentration/amount is a way to determine accuracy. Accuracy is expressed as a percent recovery (%R). Evaluation of the %R is based on statistical evaluation of past lab data or guidelines within the methods for organic and inorganic analyses.

Representativeness - Representativeness expresses the degree to which data accurately represents the media and conditions being measured. The representativeness of the data from the sampling site will depend on the sampling procedure. Sample collection is the responsibility of the client. Samples will be homogenized, if required, as part of the laboratory sample preparation. By comparing the quality control data for the samples against other data for similar samples analyzed at the same time, representativeness can be determined for this objective.

Comparability - Comparability conveys the confidence with which one set of data can be compared to another. The analytical results can be compared to other laboratories by using traceable standards and standard methodology and consistent reporting units. The Laboratory Quality Assurance Program documents internal performance, and the interlaboratory studies document performance compared to other laboratories.

Completeness - Completeness is a measure of the quantity of valid data acquired from a measurement process compared to the amount that was expected to be acquired under the measurement conditions. The completeness of an analysis can be documented by including in the data deliverables sufficient information to allow the data user to assess the quality of the results. Additional information will be stored in the laboratories archives, both hard copy and magnetic tape. Quality Assurance Standard Operating Procedures (SOPs) are in place to provide traceability of all reported results.

To ensure attainment of the quality assurance objectives, Standard Operating Procedures (SOPs) are in place detailing the requirements for the correct performance of laboratory procedures. The laboratory SOPs fall under five general categories:

1. Corporate Policy

2. Quality Assurance

3. Sample Administration

4. General Laboratory Procedures

5. Analytical (i.e., methods, standard preps., instrumentation)

All SOPs are approved by the QA Department prior to implementation. The distribution of current SOPs and archiving of outdated ones are controlled through a master file. Table 5-1 provides an index of QA SOPs in place in support of the Quality Assurance Objectives. These requirements are supplemented by the procedures in the laboratory and analytical SOPs.

Table 5-1	
Document #	Document Title
QA-101	Sample Collection
QA-102	Sample Log-in
QA-103	Sample Storage and Discard
QA-104	Internal Chain-of-Custody Documentation
QA-105	Analytical Methods Manual
QA-106	Validation and Authorization of Analytical Methods
QA-107	Analytical Methods for Nonstandard Analyses
QA-108	Subcontracting to Other Laboratories
QA-109	Laboratory Notebooks, Logbooks, and Documentation
QA-110	Reagents
QA-111	Instrument and Equipment Calibration
QA-112	Instrument and Equipment Maintenance
QA-113	Data Entry, Verification, and Reporting of Results from the Computerized Sample Management System (CSMS)
QA-114	Data Storage, Security, and Archiving
QA-115	Quality Control Records
QA-116	Investigation and Corrective Action of Unacceptable Quality Control Data
QA-117	Personnel Training Records and Curriculum Vitaes
QA-118	Quality Assurance Audits
QA-119	Proficiency Samples
QA-120	Documentation of Programming for the Sample Management System
QA-121	Quality Assurance Guidelines for Computers and Computerized Systems
QA-122	Investigation and Corrective Action Reporting for Laboratory Problems
QA-123	Missed Holding Time Reports
QA-124	External Audits
QA-125	Document Control
QA-126	Qualification and Validation Documentation for Laboratory Instrumentation and Equipment
QA-127	Handling of Client Technical Complaints (Investigations and Response)
QA-128	Compliance with Good Laboratory Practice (GLP) Regulations

6. Sampling Procedures

In order for meaningful analytical data to be produced, the samples analyzed must be representative of the system from which they are drawn. It is the responsibility of the client to ensure that the samples are collected according to accepted or standard sampling methods.

The laboratory will provide the appropriate sample containers, required preservative, chain-of-custody forms, shipping containers, labels, and seals. The majority of sample containers are purchased precleaned by the supplier. Any reused bottles are cleaned in-house following laboratory Standard Operating Procedures. Special containers with traceability documentation are available upon request. Because the laboratory does not stock this type of container, one month prior notice is required.

Each lot of preservative will be documented and checked for contaminants before use. The appropriate bottle will be preserved with the new preservative and filled with deionized water to represent a sample. A similar container (that does not contain preservative) will be filled with deionized water to be used as a blank check. Analysis results are documented for each preservative lot number.

Trip blanks will be prepared by the laboratory and accompany sample containers at the project required frequency. Analyte-free water will also be provided for field blanks.

A list of containers, preservatives, and holding times follows in Table 6-1.

Table 6-1
Sample Containers, Preservatives, and
Holding Times for Aqueous and Solid Samples

Fraction	Vol. Req. (mL) / Wt. Req. (g)	Container P=Plastic G=Glass	Preservation[a]	Holding Time[c] From Date of Collection Water	Soil
Volatiles BTEX	2 × 40 mL / 100 g	G	Cool, 4°C[b] pH <2 w/HCl	14 Days	14 Days
PAHs (8310)	2 × 1000 mL / 100 g	G (amber)	Cool, 4°C[b] $Na_2S_2O_3$	7 Days to extraction[d]	14 Days to extraction[d]
PAHs (8270) (625)	3 × 1000 mL / 100 g	G	Cool, 4°C[b] $Na_2S_2O_3$	7 Days to extraction[d]	14 Days to extraction[d]
pH	50 mL / 50 g	G	Cool, 4°C	Immediately	14 Days
BOD	1000 mL	G	Cool, 4°C	48 Hours	NA
COD	100 mL / 100 g	G	Cool, 4°C pH <2 w/H_2SO_4	28 Days	28 Days
Oil and Grease	2 × 1000 mL / 50 g	G	Cool, 4°C Preserved upon receipt	28 Days	28 Days
Ammonia Nitrogen	1000 mL / 100 g	P,G	Cool, 4°C pH <2 with H_2SO_4	28 Days	28 Days
Nitrate	50 mL / 20 g	P,G	Cool, 4°C	14 Days	14 Days
TKN	500 mL / 10 g	G	Cool, 4°C pH <2 with H_2SO_4	28 Days	28 Days

Table 6-1
Sample Containers, Preservatives, and
Holding Times for Aqueous and Solid Samples

Fraction	Vol. Req. (mL) / Wt. Req. (g)	Container P=Plastic G=Glass	Preservation[a]	Holding Time[c] From Date of Collection Water	Soil
Phosphorus	50 mL / 10 g	P,G	Cool, 4°C pH <2 with H_2SO_4	28 Days	28 Days
TSS	500 mL	P,G	Cool, 4°C	7 Days	NA
TOC	125 mL / 20 g	G	Cool, 4°C pH <2 with H_2SO_4	28 Days	28 Days
Metals	1000 mL / 100 g	P,G	HNO_3 to pH <2	6 Months Hg 28 days	6 Months Hg 28 days
Cyanide	5000 mL / 100 g	P,G	Cool, 4°C NaOH to pH >12 ascorbic acid	14 Days	14 Days

[a] pH Adjustment with acid/base is performed on water samples only.

[b] Sodium thiosulfate needed for chlorinated water samples

[c] Samples will be analyzed as soon as possible after collection. The times listed are the maximum times that samples will be held before analysis and still be considered valid.

[d] Analysis 40 days from extraction.

NOTE: For volatiles analysis, the container should be filled completely, with no headspace. **All sample containers, preservatives, and mailers will be supplied at no additional charge upon request, except for the special containers with traceability documentation. There is an additional charge for this type of container.**

7. Sample Custody

Samples are unpacked and inspected in the sample receipt area. At this time, the samples are examined for breakage and agreement with the associated client paperwork. The cooler temperatures will be checked upon receipt and recorded. As the samples are unpacked, the sample label information will be compared to the chain-of-custody record and any discrepancies or missing information will be documented. If necessary, the cooler will be closed and placed in cold storage until instructions and resolution of any discrepancies are received from the client.

A member of our Sample Administration Group will act as sample custodian for the project. To ensure accountability of our results, a unique identification number is assigned to each sample as soon as possible after receipt at the laboratory. When samples requiring preservation by either acid or base are received at the laboratory, the pH will be measured and documented, with the exception of samples designated for volatile analysis. Samples requiring refrigeration will be stored in our walk-in cooler which is maintained at 4° ± 2°C. The use of our computer system in tracking samples (by the Lancaster Laboratories sample # assignment) will control custody of the sample from receipt until the time of its disposal. The security system on our laboratory building allows us to designate the entire facility as a secure area since all exterior doors are either locked or attended. Therefore, hand-to-hand chain of custody is not part of our routine procedure, but is available upon request. If requested, hand-to-hand chain of custody will be provided as per attached SOP-QA-104. The laboratory chain of custody will begin with the preparation of bottles. The procedures for sample log-in, storage, and chain-of-custody documentation are detailed in the QA Standard Operating Procedures included in Section No. 7 (QA102, QA103, and QA104). Examples of sample labels and a custody seal are shown in Figure 7.1.

Figure 7.1

CLIENT If you do not have an account with us, results will not be released until payment is received.

SAMPLE INDENTIFICATION / LOCATION CL. RES:

COLLECTION INFORMATION

☐ COMPOSITE
☐ GRAB

DATE TIME BY:

TESTING REQUIRED PRESERVATIVE(S) ADDED

Lancaster Laboratories
2425 New Holland Pike, Lancaster, PA 17601-5994

LL1#

Sample Label (Field)

LL 1869683 DIS-000 26A 10/16/92
GRP-353016
EMP-210
00649-ABC MANUFACTURING, INC.
MW-4 GRAB WATER SAMPLE
SEMI-ANNUAL MONITORING PROJECT
COLLECTED ON 10/17/92 AT 1525 BY FRB
0219 0220 0516 1126

Sample Label (Laboratory)

Lancaster Laboratories
Where quality is a science

CUSTODY SEAL

2425 New Holland Pike, Lancaster, PA 17601-5994 (717) 656-2301

DATE:

SIGNATURE:

Laboratory Custody Seal

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SOP-QA-102.02
Supersedes Date: 03/14/97
Effective Date: APR 0 1 1998
Page 1 of 5

QUALITY ASSURANCE OPERATIONS MANUAL
Sample Log-In

Purpose:

In order to provide accountability of our results, protect client confidentiality, and to prevent sample loss/mix-up, a continuous and unique Lancaster Laboratories (LL) identification number is assigned to each sample upon laboratory receipt.

Scope:

This SOP will cover the procedure used to log client samples into the computerized sample management system (CSMS) after receipt. The Sample Administration Group is responsible for laboratory sample log-in. Sample Administration has procedures to define this sample entry process.

This procedure applies only to samples which are logged into and tracked by the CSMS. There are only a few cases where samples may not be tracked using the CSMS. These include samples which will be stored for a long period of time prior to analysis, (e.g., stability storage) or for special project samples that could be reported in a narrative research and development style report instead of our usual analytical reports. Written procedures for tracking samples not entered into the CSMS need to be developed by the technical department responsible for the project or analysis of those samples.

Personnel Training and Qualifications:

Training in sample log-in is performed in accordance with Sample Administration training procedures.

Procedure:

1. All samples received for testing shall be delivered to the Sample Administration Department immediately upon arrival at the laboratory.

2. The Sample Administration Department will be responsible for unpacking and organizing the samples.

3. Client correspondence relating to the samples shall also be transferred to the Sample Administration Department. This may include purchase orders, quotations, letters, phone logs, and Incoming Sample Activity Records (ISARs).

4. Personnel of the Sample Administration Group shall log the samples into the CSMS as soon as practical after receipt. Samples awaiting log-in are stored in temporary holding areas, at required temperature, to maintain the sample integrity. At the time of entry the computer will assign a unique Lancaster Laboratories' identification number to each sample. Samples can be received at the laboratory 7 days a week, 24 hours a day, 365 days of the year. Samples should be logged in on the same day as they are received, but there could be following exceptions:

   a. Samples received on a holiday will not be logged-in until the next normal work day. Samples received from 6 p.m. on Saturday through 11 p.m. on Sunday will be logged-in Sunday evening by third shift Sample Administration personnel.

   b. Samples submitted by clients which do not identify the type of testing to be performed or with unclear or incomplete paperwork documentation - Every effort will be made to contact the client on the same day of sample receipt. In this situation, the samples will be tracked in a hold database. The group of samples will be assigned a hold number. This database is maintained by the Sample Administration Group.

If same day sample log-in is not possible, all specified and appropriate storage requirements will be observed (e.g., refrigeration).

5. Upon assignment of a sample number, the CSMS will generate a label which shall be attached to the sample container. Every effort will be made as to not obscure the client label. The information on the sample label will include the LL sample number, the client name, the storage location, the analyses requested, a bottle code indicating container and preservative type, if applicable, a unique bar code (used for samples stored in Automated Sample Retrieval and Storage System locations [ASRS]), and any applicable notes to laboratory personnel.

6. Preservation, homogenization, and subsampling, if necessary, will be the responsibility of the Sample Support Group, or the testing laboratory. SOPs are in place within the group to define these procedures. A list of preservatives required for routine environmental analyses may be found in the *Environmental Schedule of Services*. A preservation, sulfate, and chlorine check shall be performed immediately after sample log-in for all applicable environmental samples.

7. After all above steps are performed, as required, samples shall be stored in an assigned storage location, taken to the laboratory for testing, or may temporarily be stored in SA until technical center picks up samples.

8. The next working morning, after sample log-in, a copy of an entry acknowledgment will print from the CSMS. The acknowledgment is a hard copy record of the sample entry. It will summarize, the LL sample number, the sample(s) submitted in an entry group, the test(s) to be performed, the client requesting the work, the account to be billed for the work, and the unique sample identifications assigned by the client. This acknowledgment is mailed to the client to confirm sample receipt and entry.

9. Another copy of the sample acknowledgment will print and be designated as the laboratory copy. This acknowledgment, in addition to client paperwork, will be audited by three levels of personnel after the entry process:

   a. **Sample Administration** will audit to assure that the entry corresponds to all entry documentation including client supplied paperwork and/or quotations.

   b. **Client Services** will audit to assure the entry is reflective of client documentation and that additional client/project requirements were communicated and taken into consideration. They will also verify that account and billing information is accurate.

   c. **Technical centers** will assure appropriate preparation and analysis set-up steps have been added to the entry. They will also verify that project and technical requirements have been taken into consideration from a technical point of view.

   Each reviewer will initial the top of the SA file copy of the acknowledgment to document their review. Additional copies of this acknowledgment can be made for laboratory personnel.

10. The LL sample number assigned to each sample shall be used to identify the sample in all laboratory records, including laboratory notebooks, instrument printouts, and laboratory final reports. The sample number will also be used to identify all additional containers of the sample which may be created during sample preparation and analysis. This will include subsamples, extracts, and digests.

Revision Log:

Initiated Date: 03/87

Ver. #	Effective Date	Change
00	02/15/96	Previous issue
01	03/14/97	Major changes are as follows: • Expanded upon the scope of the procedure • Added section about printing and auditing of the sample entry acknowledgment • Added Personnel Training and Qualification section • Removed specifics on how to document preservation checks
02	APR 0 1 1998	Major changes are as follows: • Corrected typo • Wording changes in Procedure section to clarify current operations

SOPQA102.DOC
031198

Prepared by: Kathy D. Wetzel Date: 3/23/98

Approved by: Kathleen J. Loewen Date: 3/23/98

Approved by: William Hensley Date: 3/24/98

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SOP-QA-103.01
Supersedes Date: 10/01/96
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QUALITY ASSURANCE OPERATIONS MANUAL

Sample Storage and Discard

Purpose:

Sample integrity can be compromised by improper storage conditions. The objective of this procedure is to prevent sample deterioration and mix-up prior to analysis. The computerized sample management system (CSMS) is used to assign storage locations to assist in the orderly storage of samples. Systems are also in place to ensure organized retrieval of samples for analysis and discard/return to client at an appropriate date.

Scope:

This procedure applies to all Lancaster Laboratories business units (Environmental Sciences, Food and Animal Health Sciences, Pharmaceutical Sciences). The content of this procedure will describe general systems that are in place for sample storage, retrieval, return, and discard. Additional procedures may be in place to describe specific storage operations and requirements within each business unit.

The scope of this procedure does not address the storage and monitoring of samples stored in stability chambers. Separate procedures are in place within the Pharmaceutical area to describe these operations.

The scope of this procedure also does not address the storage and discard of controlled substances. This is defined within SOP-LA-036, "Controlled Substance Handling Procedure."

Safety Precautions:

Refer to the corporate *Chemical Hygiene Plan* which provides safety information. Contact your supervisor if you have questions or concerns about a sample.

Personnel Training and Qualifications:

Personnel who handle client samples must be familiar with the requirements of this procedure.

Procedure:

A. Sample storage and transfer

1. Sample Administration will gather information into the CSMS at the time of sample entry about the approximate size of samples to be received in a group and the type of storage they require (e.g., refrigerator, freezer, or room temperature).

2. The CSMS will assign the storage location and record the length of time the samples must be retained after the analysis report has been issued.

3. Samples will be stored in the assigned storage location, when not in the laboratory area.

4. In the event that a sample location change is needed, a Sample Storage Custodian or designated analyst will access the appropriate CSMS program and choose a new location. After a successful change in location has occurred, the new location will be written on each Lancaster Laboratories sample label, or a new label will be reprinted and adhered to the sample. The sample will then be transferred to the new storage location.

5. Analysts requiring the use of a sample may determine its location by referring to a departmental sample status sheet, CSMS, or SA entry paperwork.

6. To prevent unnecessary deterioration of the samples, the contents needed for analysis shall be removed and the sample returned to storage with a minimum of delay.

B. Security of storage areas

There are varying degrees of additional security requirements for storage areas, which are in addition to the building security. This additional security may be driven by various regulatory agencies. The following are different levels of security which are in place at the laboratory.

1. Samples may be located in an individual laboratory area which is not locked. No additional security is required, in addition to the existing building security. Samples may only be removed from and returned to these locations by an analyst working in that area. Care shall be exercised in returning the samples to their appropriate location.

2. Locked storage areas are also available in many individual laboratory areas, in addition to the environmental sample support area. Access to these storage areas is limited to analysts who are responsible for testing the samples that are stored there. These areas are locked when the laboratories are unattended; keys or combinations are available only to members of the department where they are located. Again, care shall be exercised in returning the sample to its appropriate location.

3. Controlled access areas are attended by a sample custodian and are large areas used by more than one group. Samples stored in controlled access areas are coordinated by the sample custodian assigned to the area. Only appropriate personnel may access this controlled area. After analysis, samples are returned to this storage center and placed in their designated storage location.

   Pharmaceutical and Animal Health samples are stored in a controlled access area. Environmental and some Food samples which are also stored in a

separate controlled access area are tracked by an automated sample retrieval storage system (ASRS). Samples are barcoded in and out of this system to track retrieval, return, and disposal.

4. Forensic storage areas are locked and admission to these areas is permitted only to sample custodians. Most of the samples stored in these areas require chain-of-custody documentation as outlined in SOP-QA-104, "Internal Chain of Custody Documentation." Samples may not be removed from this area without signing a chain-of-custody form. A chain-of-custody record may also be kept for samples, at the request of the client, even if the samples are not for forensic purposes.

5. Security of the controlled substance samples storage area is addressed by SOP-LA-036.

C. Sample discard

1. When the retention time for sample storage has expired, a discard list will be generated from the CSMS. The retention dates are based upon client requirements or defaulted to a given number of days past the date when the final analysis report is generated, if no client requirement is given.

2. These samples will be removed from their assigned storage area by a sample custodian or analyst, and either disposed of or returned to the client.

3. Hazardous samples shall either be returned to clients, decontaminated, or disposed of at the direction of supervisory personnel.

D. Storage conditions

1. The temperature of each sample storage location requiring a temperature control should be checked during each normal working day by an assigned person responsible for the sample storage area. This information shall be recorded.

2. Temperature documentation can be recorded on logs posted on the outside of the unit or by computerized recording devices or chart recorders.

3. The following temperature ranges need to be maintained within storage units.

	Refrigerator Storage	Freezer Storage
Environmental	2° to 4°C	-10° to -20°C
Foods	2° to 6°C	-10° to -20°C
Pharmaceutical Animal Health	2° to 8°C	-10° to -20°C

NOTE:

Pharmaceutical Room Temperature Sample Storage (for Pharmaceutical, Animal Health, and GLP samples) is maintained at 15° to 30°C.

Foods also has storage conditions available at -40° ± 10°C and -80° ± 10°C.

4. If the temperature recorded does not fall within these ranges, corrective action needs to be taken.

5. Temperature monitoring documentation shall be recorded in ink and changes shall be made in accordance with the error correction procedure outlined in SOP-QA-109, "Laboratory Notebooks, Logbooks, and Documentation."

6. Temperature records must be reviewed by a second qualified person and this information must be permanently archived.

7. In the event that additional storage areas are needed as "overflow" storage, systems must be put into place before samples can be stored. These areas must also be monitored for acceptable storage conditions.

8. If a client requests storage conditions which are outside the temperature ranges defined above, arrangements will be made to accommodate the request, if possible.

Revision Log:

Initiated Date: 03/87

Ver. #	Effective Date	Change
00	10/01/96	Previous Issue
01	SEP 0 2 1997	Major changes are as follows:

- Expanded upon Scope, Purpose, and Procedure to include Pharmaceutical Sciences and Animal Health
- Added Personnel Training and Qualifications section
- Broke Procedure section down into sample storage and transfer, security of storage areas, sample discard, and storage conditions
- Incorporated Procedural Amendment #1
- Clarified the scope of this procedure does not apply to the storage of samples in our stability chambers or the storage of controlled substances

SOPQA103.DOC
081597

Prepared by: Kathy D Wetzel Date: 8/15/97

Approved by: Kathleen M Lowen Date: 8/18/97

Approved by: J. Graham Hershey Date: 8/19/97

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QUALITY ASSURANCE OPERATIONS MANUAL

Internal Chain-of-Custody Documentation

Purpose:

In order to demonstrate reliability of data which may be used as evidence in a legal case, required by a regulatory agency, or required by a client, an accurate written record tracing the possession of samples must be maintained from the time they are received at the laboratory until the last requested analysis is verified. The purpose of a chain of custody is to ensure traceability of samples while they are in the possession of the laboratory.

Scope:

This procedure describes the initiating and maintaining of chain-of-custody (COC) documentation for samples that require this level of traceability. It applies to all business units of Lancaster Laboratories (Environmental Sciences, Food and Animal Health Sciences, Pharmaceutical Sciences) when a client or regulatory agency requests an accurate written record tracing the possession of samples from the time they are received at the laboratory until the last requested analysis is verified. This procedure also applies to samples, especially environmental samples, which may be used as evidence is a legal case.

The first section of this procedure summarizes the COC requirements for food, animal health, and pharmaceutical samples. The second section summarizes the requirements for environmental COC samples.

Definition:

A sample is in custody if it is in any one of the following states:

1. In actual physical possession.
2. In view after being in physical possession.
3. Locked up so no one can tamper with it.
4. In a secured area, restricted to authorized personnel (e.g., in the ASRS system, pharmaceutical samples storage area).

Personnel Training & Qualifications:

Training for this procedure consists of reading SOP-QA-104. Supervisory review of all COC documentation should be done until the trainer is satisfied that proficiency has been achieved. Training of all laboratory personnel is the responsibility of the group leader. Documentation that this training has been completed must be kept in the training records.

Procedure:

A. Food, Animal Health, and Pharmaceutical procedure

1. General overview

a. Chain-of-custody documentation shall be kept upon the request of a client or regulatory agency. As with all analytical data, it is **extremely** important that this documentation be filled out completely and accurately with every sample transfer. **Everyone who handles the COC has the responsibility to check for documentation compliance to the point of their acquisition.** If changes need to be made to the form, they shall be made in accordance to the error correction

procedure addressed in SOP-QA-109, "Laboratory Notebooks, Logbooks, and Documentation." It is the responsibility of the person who made an error in documentation to correct the error.

b. For Food, Animal Health, and Pharmaceutical samples, internal COC documentation will begin when samples are received at the laboratory.

c. A member of the Sample Administration group will receive and unpack the samples. They will sign the client paperwork that accompanies the samples, if provided. If the samples were picked up by our Transportation department, the driver must also sign applicable paperwork to relinquish the samples to Sample Administration.

d. The Sample Administration group will track the custody of samples between receipt and entry into the computerized sample management system (CSMS) on the SA Receipt Documentation Log (Figure 2 attached). The client's sample designation will be used for identification purposes until a unique Lancaster Laboratories' number is assigned.

e. Samples will be entered into the CSMS as described in SOP-QA-102, "Sample Log-in."

f. After entry, samples are released by the Sample Administration group to be stored in other areas of the laboratory (e.g., Foods, Pharmaceutical specific storage areas). During this time, they may be accessed by several people in that area. Each of these people must note the specific sample numbers in their custody in addition to date, time, and reason for removal from storage. Chain-of-custody transfer documentation is recorded on a form which is given as Figure 6 of this procedure.

2. Documentation of custody changes

   a. An example of how to document changes in sample custody is shown as Figure 6 of this procedure. Each change of sample custody must be accurately documented in a consistent format. Entries documenting change of custody will use the following format:

      Signature: First initial, full last name, employee number
      Date: Month/day/year
      Time: Documented as military time
      Ink: Black ink is preferred, red ink and pencil are not acceptable

   b. When sample(s) are released from storage, they must be signed out by the analyst receiving them. The analyst must note the sample number(s) taken, the date and time received, and a note as to the reason why they were removed from storage (reason for change of custody).

   c. When an analyst returns sample(s) to storage, they must note the sample number(s) being returned in addition to time of return.

   d. If the custody of sample(s) is passed from one analyst to another, the following must be noted: the sample number(s) transferred, the reason for the transfer, in addition to the time and date of transfer. It is the responsibility of the analyst now in possession of the samples to ensure they are returned to the storage area.

   e. Each specific test that an analyst performs, in conjunction with the associated sample number(s), must be accurately documented by the analysts in the Reason for Change of Custody column before the samples are returned to the sample storage area.

3. Additional COC issues

   a. Chain-of-custody paperwork will remain with and accompany sample containers at all times.

   b. When sample analysis is complete, samples shall be returned to the storage area, in conjunction with the COC form.

   c. Original COC documentation will be maintained at the laboratory and filed in the sample storage area. COC documentation will be overchecked in conjunction with the sample data before a copy is sent to the client. The copy could either accompany the samples as they are returned to the client or it could be included within a client data package.

   d. All personnel who handle sample containers shall make every attempt to ensure that all changes of custody are accurately and completely documented. **Disciplinary action may be taken for employees who fail to comply with these important requirements.**

B. Environmental procedure

1. Initiation of COC documentation

   a. Chain-of-custody documentation shall be kept upon the request of the client or for any samples which are known to be involved in a legal dispute. As with all analytical data, it is **extremely** important that this documentation is filled out completely and accurately with every sample transfer. **Everyone who handles the COC has the responsibility to check for documentation compliance to the point of their acquisition.** If changes need to be made to the form, they shall be made in accordance to the error correction procedure addressed in SOP-QA-109. It will be the responsibility of the person who made an error in documentation to correct the error.

b. If requested by the client, the COC documentation will begin with the preparation of sampling containers. A form (Figure 1 attached) will be initiated by the person packing the bottle order for shipment to the client. If the delivery of containers is via Lancaster Laboratories Transportation department, the driver shall sign the form when they relinquish the bottles to the client. Drivers must also sign COC forms when they pick up samples for analysis.

c. When samples arrive at the laboratory for analysis, a member of the Sample Administration group will receive them and sign the external COC form that accompanies the samples, if provided. If the samples were picked up by our Transportation Department, the driver must sign the COC to relinquish the samples to Sample Administration.

d. The Sample Administration group will track the custody of samples between receipt and entry into the CSMS on the SA Receipt Documentation Log (Figure 2 attached). The client's sample designation will be used for identification purposes until a unique Lancaster Laboratories' number is assigned.

e. Samples will be entered into the Sample Management System as described in SOP-QA-102. Sample Administration will enter an analysis number for "Laboratory Chain of Custody" if requested. A lab note will print to inform analysts of the need for COC documentation. This note will also be automatically added to the sample labels.

2. Creating the internal COC

a. Sample Administration personnel shall initiate an internal Laboratory Chain of Custody form at the time of sample entry (Figure 3 attached) for each type of container in the sample group. A master list of all chains created will also be initiated for each sample group at the time of entry (Figure 4 attached). The samples will then be relinquished to a sample custodian who will store the samples in an assigned secure

location. This change of custody from sample entry to storage shall be documented on the chain, as well as any interim exchanges for rush analysis, preservation, homogenization, or temporary storage in the SA HOLD. The internal COC forms will then accompany the samples from storage to the laboratory for analysis.

b. If samples need to be checked out from the Sample Administration group before Lancaster Laboratories' numbers have been assigned to them, SA will be responsible for starting a COC form. They will note the available header information, the samples being relinquished (documented by the client sample designation), and the reason for transfer.

c. After sample entry, the original copy of the external client COC/analysis request form will be filed with Accounts Receivable, to be returned to the client with their invoice. Other copies of the external form will stay within SA to be filed within the client's paperwork file.

3. Documentation of custody changes

a. An example of how to document changes in sample custody is shown in Figures 3 and 5. Each change of sample custody must be accurately documented in a consistent format. All signatures documenting changes of custody will use the following format:

Signatures: first initial, full last name, employee number
Date: Month/day/year
Time: Documented as military time
Ink: Black ink is preferred, red ink and pencil are not acceptable

(1) When sample support releases samples to an analyst they must:

Note the sample number(s) released and sign the "Released By" column of the chain.

(2) When an analyst receives samples from sample support they must:

Sign the "Received By" column, note the date and time samples are received, and note the reason why they are taking the samples (reason for change of custody).

(3) When an analyst returns samples to sample support they must:

Note all sample numbers being returned, sign the released by column, and note time and date of return.

(4) When sample support receives samples from an analyst they must:

Sign the received by column and note the reason for sample transfer.

b. Sample handling should be kept to a minimum. Analysts requiring use of a sample will requisition it through the computer requisition program. During the hours when sample support is manned by sample custodians, a custodian will receive the computerized requisition and remove the sample from storage. The custodian will ensure that the bottle type listed on the COC form matches the bottle type being distributed. **It will be the shared responsibility of the analyst and sample custodian to insure that forms are signed, dated, and reason for sample transfer are recorded with each change of custody, as directed by Item (3)a. above.**

c. Each specific test that an analyst performed in conjunction with the associated sample number(s) must be accurately documented by the analyst before the samples are returned to a sample custodian in the sample storage area.

d. When an analyst requires the use of samples when a sample custodian will not be on duty, they must requisition samples earlier in the day or on the previous day. These samples and associated COCs will be pulled by a sample custodian and placed in the locked SA HOLD storage area. The sample custodian will note on the COC the change in transfer to the SA HOLD in addition to the time, date, and the sample numbers. The analyst picking up the samples will document the specific samples being checked out, record SA HOLD in the released by column, sign the received by column, note the time, date, and reason for transfer. When the analyst returns the samples to the SA HOLD, they must sign the samples back into the SA HOLD.

e. The following changes of custody will be handled in the following manner:

(1) Documentation is required for all shift changes. Signatures involving transfers from one shift to another shall be the responsibility of the analyst who originally acquired the samples from sample support.

(2) Occasionally, a sample container will be needed for analysis by an analyst in a department while it is in the custody of an analyst in another department. It will be the responsibility of the first person who received the sample to note on the COC the specific sample numbers requested by the second person and to sign the released by column. The second person will sign the received by column and note the time, date, and reason for sample transfer. After the second person is finished with the sample, the sample will be returned back to the first person or to the sample storage area.

(3) In situations where a sample group must be split between departments working on different analyses, a supplemental COC must be initiated by the Sample Support group. The

supplemental chain will be used to accompany that portion of the sample group which is needed by a second department, when another department has part of the sample group and the COC for the entire group. This supplemental COC will be created only when absolutely necessary to minimize paperwork and confusion. This chain must also be documented on the master list of chains initiated for the sample group.

(4) If COC samples are stored in other areas of the laboratory or in a specific department, they must be stored in a locked area. When samples are taken from a departmental storage area, the released by column of the COC is documented as "department XX storage." If samples are returned to this area when complete, the received by column will be noted as department XX storage.

4. Additional COC issues

a. Analysts in possession of samples shall remove the aliquot required for their analysis and return the samples to the Sample Support group with a minimum of delay. During this time of possession, samples must fall under the definition of sample custody.

b. If additional containers of the sample are created (e.g. subsamples, extracts, distillates, leachates, digests, etc.), an additional COC form must be created by the department if they do not document this information on the original COC form (Figure 5 attached). This form will be marked with the container type and will be initiated to accompany the new sample container. Each department in the lab has specifically designed COC forms which will be used if new containers are created. All changes of custody involving handling of new containers in the department (e.g. analysis, storage, vials on instruments, etc.) will be documented on the departmental specific COC form or on the original COC form. Any specific handling or documentation requirements for departmental chains can be described in a departmental SOP.

5. When sample analysis is complete

   a. After sample analysis, samples shall be returned to the Sample Support group as soon as possible. Original COC forms shall also be returned with the samples and this change of custody noted. At this time, it will be the responsibility of the Sample Support group to review the COC forms to ensure that all documentation on the forms is complete before they file the forms in their area. Sample custodians will not return a sample to its assigned storage location without signing the accompanying chain and performing this completeness check. All chains should either end with a note of "All Sample Consumed," "Discard," or "Storage" for the final reason of transfer.

   b. All completed COC forms for the original sample containers will be retained in files within Sample Support. The Data Package group will retrieve these forms so a copy can be included in the data package. All departmental created COC forms will be collected by the department's data package group so a copy can be included in the data package. These forms will not be returned to the Sample Support group since these sample containers will not be returned to the Sample Support group. The original copy of all COC forms will be retained on file by the laboratory.

   c. All personnel who handle sample containers shall make every attempt to ensure that all changes of custody are accurately and completely documented. **Disciplinary action may be taken for employees who fail to comply with these important requirements.**

   d. In the event that a signature or other information is inadvertently not recorded on a COC form, the Sample Support and Data Package groups, in conjunction with the technical centers, shall determine what information is missing by checking computer requisition records, raw data, or the sample support work schedule. The responsible party shall add the missing information or make the necessary correction at the

bottom of the COC form, in addition to noting the situation that caused the error in documentation. The person making this note needs to sign and date the information using the current date. Any errors in COC documentation that cause noncompliances must be noted in the case narrative of the sample data package. Examples of specific cases are on file in the Data Package Department.

Revision Log:

Initiated Date: 03/87

Ver. #	Effective Date	Change
00	12/01/95	Previous Issue
01	03/14/97	Major changes are as follows: • Training section added. • Examples of SA Receipt Documentation Log and Metals Locked Storage COC updated. • Section E.1., Option to end chain with "All Sample Consumed" added.
02	SEP 0 2 1997	Major changes are as follows: • Incorporated specific Procedure section for Food, Animal Health, and Pharmaceutical procedures. • Expanded upon the scope of the procedure.

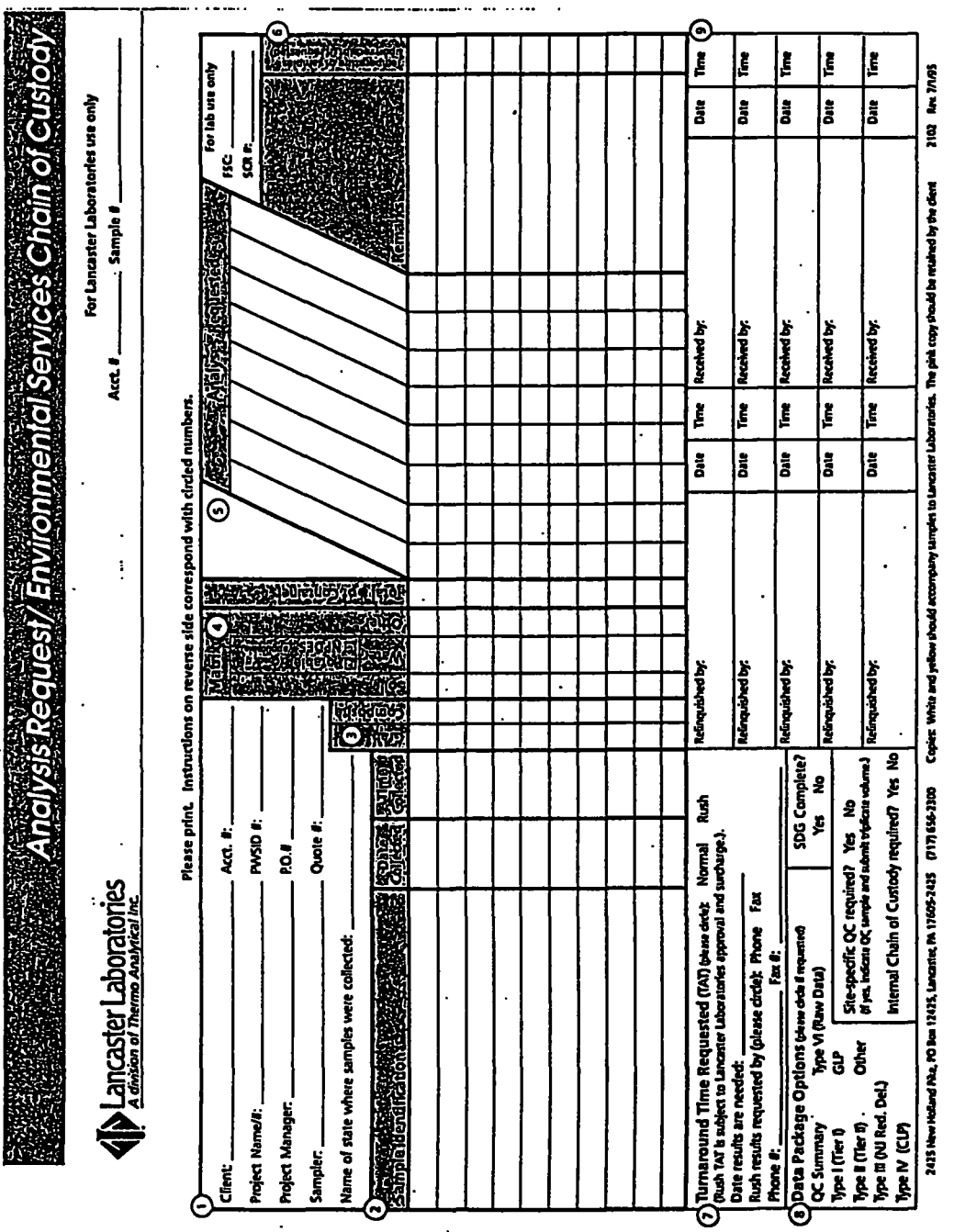
SOPQA104.DOC
081297

Prepared by: Kathy D Wetzel Date: 8/12/97

Approved by: Kathleen M. Loewen Date: 8/14/97

Approved by: J Andrew Hershey Date: 8/18/97

Figure 1



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DIRECTIONS FOR COMPLETING THIS FORM

(1) Client: Your company's name

Acct. #: Your account number with Lancaster Laboratories

Project Name/#: The way your company refers to the work involved with these samples. You may want to include project location as part of the description.

PWSID: Potable Water Source ID#

Project Manager: The person at your company responsible for overseeing the project

P.O. #: Your company's purchase order number

Sampler: The name of the person who collected the samples

Quote #: The reference number that appears on your quote (if Lancaster Laboratories gave you a number)

State where sample was collected: Please indicate where the sample was taken, e.g., Pa., N.J., etc.

(2) Sample Identification: The unique sample description you want to appear on the analytical report

Date Collected/Time Collected: When the sample was collected

(3) Grab: Check here if sample was taken at one time from a single spot.

Composite: Check here if samples were taken from more than one spot, or periodically, and combined to make one sample.

(4) Matrix: Check the type of sample you are submitting. If it is a water sample, please indicate if it is a potable water or if it is an NPDES sample.

Number of Containers: Indicate the total number of containers for each sampling point.

(5) Analyses Requested: Write the name of each analysis (or an abbreviation of it) here, and use the catalog number that appears at the beginning of each line in the *Schedule of Services*. Be sure to indicate which analyses are to be performed on which samples.

(6) Remarks: List special instructions about the sample here (e.g., hazardous elements, high levels of analyte, etc.). The space can also be used (if needed) for listing additional analyses.

(7) Turnaround time Requested: Circle Normal if you want routine TAT, which is usually within 10-15 days. If you need your results faster, call ahead to schedule Rush work.

Rush Results Requested by: Circle Fax or Phone and include the number.

(8) Data Package Options: Call our Client Services Group (717-656-2301) if you have questions about these choices.

SDG Complete? Indicate Yes if this is a complete sample delivery group or No if you will be submitting additional samples to be included in the same data package.

Note: We need to have one quality control (QC) sample for every 20 samples you send, if you are requesting site-specific QC. Please give us this sample in triplicate volume and identify it by writing "QC" in the Remarks column.

The internal chain of custody is a hand-to-hand documentation recording a sample's movement throughout the company. We routinely start a chain of custody for data-package samples unless we are told otherwise. There is a $25 per sample charge for the chain-of-custody documentation.

(9) Relinquished by/Received by: The form must be signed each time the sample changes hands. We can supply chain-of-custody seals for the outside of your packages if you require them.

Thank you for using Lancaster Laboratories.
Please call our Client Services Group (717-656-2301) if you have any questions about completing this form.

Figure 1 - Continued

Figure 2

Lancaster Laboratories

Sample Administration
Receipt Documentation Log

Client/Project: XYZ Associates / Well Monitoring — Shipping Container Sealed: (Y) / N
Date of Receipt: 6/27/97 — COC Seal Present: (Y) / N
Time of Receipt: 1350 — COC Seal Intact: (Y) / N
Source Code: 60 — Package: (Chilled) / Not Chilled
Unpacker Emp. No.: 210

Temperature of Shipping Containers	
#1	**#2**
Thermometer ID: 123	Thermometer ID:
Temp.: N/A	Temp.:
(Temp. Bottle) / Surface Temp.	Temp. Bottle / Surface Temp.
(Wet Ice) / Dry Ice / Ice Packs	Wet Ice / Dry Ice / Ice Packs
Ice Present? (Y) / N	Ice Present? Y / N
#3	**#4**
Thermometer ID:	Thermometer ID:
Temp.:	Temp.:
Temp. Bottle / Surface Temp.	Temp. Bottle / Surface Temp.
Wet Ice / Dry Ice / Ice Packs	Wet Ice / Dry Ice / Ice Packs
Ice Present? Y / N	Ice Present? Y / N

Paperwork Discrepancy/Unpacking Problems: Broken 40ml Vial, Client ID 345-01. Client Called 6/27/97 at 1610.

Sample Administration Chain of Custody			
Name	**Date**	**Time**	**Reason for Transfer**
K. Suito	6/27/97	1600	Unpacking
A. Hutchison	6/27/97	1615	(Place in Storage) or Entry
D. Neslund	6/27/97	1800	Remove from Storage / Entry
			Place in Storage or Entry
			Entry

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Figure 3

Lancaster Laboratories

Locked Storage Chain of Custody
Original Sample

Client/Project: XYZ Associates

Preservative: HCl Matrix: Water SDG: XYZ01

Sample # Range of Entry Group: 2420638-39 Bottle Type: #38 40 ml Vial

Sample Number(s) in Custody	Released By	Received By	Date of Transfer	Time of Transfer	Reason for Change of Custody	Dist., Extr., or Digest Chain Created (X)
2420638-39	D. 208 Nealund	SS Storage	11/27/95	1600	Entry & Storage	
2420638-39	SS Storage	B. 705 Weaver	11/28/95	700	Remove from SS Storage	
2420638-39	B. 705 Weaver	dept 21 Storage	11/28/95	715	VOA Storage	
2420638-39	dept 21 Storage	K. 396 Wittman	11/29/95	1315	VOA Analysis	X
2420638-39	K. Wittman 396	L. 513 Taylor	11/29/95	1700	VOA Analyst Shift Change	
2420638-39	L. 513 Taylor	dept 21 Storage	11/29/95	2100	VOA Storage	
2420638-39	dept 21 Storage	C. 266 Ayars	12/3/95	800	Transfer to SS Storage	
2420638-39	C. Ayars 266	L. 630 Kessler	12/3/95	815	Storage	

Figure 4

Lancaster Laboratories

Master List of Chain of Custodies

Client/Project: XYZ Associates

Sample # Range of Entry Group: 242032-39

SDG: XYZ01 Matrix: (Liquid) Solid Mixed Other

Original Sample Chains		
Bottle Type	**Started By**	**Date Started**
40 ml Glass Vial (#38)	D. Neslund 208	11/27/95
1000 ml Amber Glass (#45)	↓	↓
1000 ml Plastic (#09)		
1000 ml Amber Glass (#29)		

Supplemental Chains		
Bottle Type	**Started By**	**Date Started**
77	C. Ojard 266	11/27/95
21	C. Ojard 266	11/27/95

Extraction, Digestion, Distillates, Etc.		
Bottle Type	**Started By**	**Date Started**

2365 10/19/95

Figure 5

Lancaster Laboratories

Locked Storage Chain of Custody
Metals

Client/Project: XYZ Associates

Sample #: 2420632-9 SDG: XYZ01

Digest Type (circle one): Hg (Metals) GF Hydrides Trial No: 2 (If not 1, fill in)

Batch No: 95305 1849 004

Sample Number(s) in Custody	Released By	Received By	Date of Transfer	Time of Transfer	Reason for Change of Custody
2420632,4,6	S. Correa/523	J. Garrett/428	12/1/95	1631	Metals prep Shift change
2420632,4,6	J. Garrett/428	ICP Storage	12/1/95	1920	ICP Storage
2420632,4,6	ICP Storage	D. Saebett/142	12/1/95	2115	ICP Analysis
2420632,4,6	D. Saebett/142	ICP Storage	12/1/95	2135	ICP Storage.

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Figure 6

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Food, Pharmaceutical, Animal Health
Locked Storage Chain of Custody
Original Sample

Client/Project: Smith Pharmaceutical

Preservative: N/A Matrix: Tablets

Sample # Range of Entry Group: 2830419-30

Bottle Type: Amber Glass - 100 Tablets/Bottle

Sample Number(s) in Custody	Released By	Received By	Date of Transfer	Time of Transfer	Reason for Change of Custody
2830419-30	L. Carruthers 074	M. Coho 589	7/25/97	1300	Entry/ Transfer to Pharm. Storage
2830419-30	M. Coho 589	Pharm Storage	7/25/97	1315	Storage
2830419-30	Pharm Storage	E. Ortiz 572	7/27/97	800	PN analysis
2830419-30	E. Ortiz 572	Pharm Storage	7/27/97	1000	Storage
2830419-30	Pharm Storage	D. Wright 330	7/29/97	930	GC assay Analysis
2830419-30	D. Wright 330	Pharm Storage	7/29/97	1400	Storage

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8. Calibration Procedures

Procedures for initial calibration and continuing calibration verification are in place for all instruments within the laboratory. The calibrations generally involve checking instrument response to standards for each target compound to be analyzed. The source and accuracy of standards used for this purpose are integral to obtaining the best quality data. Standards used at Lancaster Laboratories are purchased from commercial supply houses either as neat compounds or as solutions with certified concentrations. The accuracy and quality of these purchased standards is verified through documentation provided by these commercial sources. Most solutions and all neat materials require subsequent dilution to an appropriate working range. All dilutions performed are documented and the resulting solution is checked by obtaining the instrument response of the new solution and comparing with the response to the solution currently in use. Any discrepancies between the responses are investigated and resolved before the new solution is used. Each standard is assigned a code which allows traceability to the original components. The standard container is marked with the code, name of solution, concentration, date prepared, expiration date, and the initials of the preparer. Shelf-life and storage conditions for standards are included in the standard operating procedures and old standards are replaced before their expiration date.

Each instrument is calibrated with a given frequency using one or more concentrations of the standard solution. As analysis proceeds, the calibration is checked for any unacceptable change in instrument response. If the calibration check verifies the initial response, the analysis proceeds. If the calibration check indicates that a significant change in instrument response has occurred, then a new calibration is initiated. If necessary, maintenance may be performed prior to the recalibration.

Calibration records are usually kept in the form of raw data with the other instrument printouts. In cases where no data system is used, calibration data is manually recorded in notebooks. Any maintenance or repair is also recorded in a notebook. The information recorded either in the notebooks or on the instrument printout includes the date, instrument ID, employee name and/or identification number, and concentration or code number of standard.

The frequency of calibration and calibration verification, number of concentrations used, and acceptance criteria for each of the instruments to be used are listed on Table 8-1. In addition to checking the instrument response to target compounds, the GC/MS units are checked to ensure that standard mass spectral abundance criteria are met. Prior to each calibration, instruments being used for semivolatile analysis are tuned using decafluorotriphenylphosphine (DFTPP). The key ions and their abundance criteria are listed in Table 8-2.

Table 8-1						
Initial Calibration			Continuing Calibration Verification			
Instrument	Frequency	Number of Standard Concentration	Acceptance Criteria	Frequency	# Std Conc	Acceptance Criteria
GC VOA BTEX	After C-cal fails	5	%RSD of <20% for individual compounds or for average of all compounds	Every 12 hours, or every 10 samples	1	%Drift ±15% for individual compounds or average of all compounds
HPLC	Each new run or after C-cal fails	5	20% RSD of RFs of initial calibration to use average RF, otherwise use curve fit Alternatively, if the average of the %RSDs of all compounds in the calibration standard is ≤20%, then the AVG RF can be used for all compounds	Every 10 samples	1	≤15% difference from initial response for quantitation C-cal – A CCV is also compliant if the average RPD is ≤15% for all compounds in the CCV standard
GC/MS* PAHs	After C-cal fails	5	RF for SPCC's ≥0.050. Max %RSD for CCC's ≤30%	Every 12 hours	1	RF for SPCC's ≥0.050. % Drift for CCC's ≤20
Autoanalyzer	Daily	6	Correlation Coefficient >0.995	Every 10 samples	1	±10% of true value
Ion Chromatograph	Daily (Every 194 injections)	6	Correlation Coefficient >0.995 ICV ±10%	Every 10 samples	1	±10% of true value
Spectrophotometer (Colorimetric)	Quarterly	6	Correlation coefficient >0.995	Daily or every 10 samples	1	±20% of EPA std.
TOC Analyzer	Daily	5	±10% at STD	Every 10 samples	1	±10% of true value
Oxygen Meter	Daily	Calibration Against Winkler Titration	NA	NA	NA	NA
pH Meter	Daily	Slope 2 Buffers	Independent calibration verification ±3%	Every 10 samples	1	±3%
Balance	Daily	4	±.5%	NA	NA	NA

Table 8-1						
Initial Calibration			Continuing Calibration Verification			
Instrument	Frequency	Number of Standard Concentration	Acceptance Criteria	Frequency	# Std Conc	Acceptance Criteria
GC/MS PAHs (625) Semivolatiles	After C-cal fails	5	RF for SSPCCs >0.050 Max %RSD for CCCs <35%	Every 24 hours	1	RF for SPCcs 0.050 %Drift for CCCs <20
ICP/Trace ICP	Each new run	1	Independent calibration verification within ±10% standards <5% RSD	Every 10 samples	1	Same as initial
CVAA	Each new run	5	Independent calibration verification within ±10% Correlation coefficient >0.995	Every 10 samples	1	±20% of true value

*All compounds with %RSD >15 must use first or second order regression fit of the six calibration points. Alternatively, if average of the %RSD of all compounds in calibration standard is ≤15%, the AVG RF can be used for all compounds.

Abbreviations

Std Conc is the number of standard concentrations used.

SPCCs are system performance check compounds.

CCCs are calibration check compounds.

RF is response factor.

%RSD is percent relative standard deviation.

%D is percent difference.

C-cal is continuing calibration.

Table 8-2	
Mass	Ion Abundance Criteria
DTFPP Key Ions and Ion Abundance Criteria:	
51	30% to 60% of mass 198
68	less than 2% of mass 69
69	mass 69 relative abundance
70	less than 2% of mass 69
127	40 to 60% of mass 198
197	less than 1% of mass 198
198	Base peak, 100% relative abundance
199	5% to 9% of mass 198
275	10% to 30% of mass 198
365	greater than 1% of mass 198
441	Present but less than mass 443
442	greater than 40% of mass 198
443	17% to 23% of mass 442

9. Analytical Procedures

The analytical procedures to be used are those described in USEPA 600/4-79-020 and in the USEPA SW-846 3rd Edition, Update III, 1996, for the preparation and analysis of water, sediment, and soil for the client specified compounds. Copies of the analytical procedures are located in the laboratory and available for use by analysts. Copies of analytical methods are available upon request.

PAHs by GC/MS - This method determines the concentration of semivolatile organic compounds that are separated into an organic solvent and are amenable to gas chromatography. The method involves solvent extraction of the sample to isolate analytes and GC/MS analysis to determine semivolatile compounds present in the sample. Method 8270C/Method 625.

Volatiles by GC - This method determines the concentration of volatile (purgeable) organic compounds. The analysis is based on purging the volatiles from the sample onto an appropriate sorbent trap and desorbing the volatiles onto a gas chromatographic column. Using an appropriate temperature program, the compounds are separated by the column and both qualitative and quantitative detection is achieved with a Photoionization and/or Electrolytic Conductivity detector. Method 5030B/8021B/5035.

PAHs by HPLC - The sample aliquot is extracted with methylene chloride. The extract is filtered (soils), dried, concentrated by evaporation and exchanged into acetonitrile. Silica gel cleanup is used if necessary. The extract is analyzed by reverse phase HPLC with both UV and Fluorescence detectors. Methods 3550B/3630C/8310.

Biochemical Oxygen Demand - A seeded sample of the waste is incubated with nutrients for five days at 200C. The reduction of dissolved oxygen (DO) concentration during the incubation yields a measure of the BOD. The DO is used by microorganisms as they breakdown carbonaceous organic material. If nitrifying bacteria are present, nitrogenous compounds can add to the BOD. Complex organic compounds may not show a BOD if they cannot be assimilated by the seed bacteria.

Methods for the Chemical Analysis of Water and Wastes, USEPA 600/4-790-020. Method 405.1.

Chemical Oxygen Demand - This method is appropriate for midlevel water samples. Chemical oxygen demand is a measure of the total amount of oxygen required for oxidation of waste to carbon and water. The sample is heated for two hours in an acidic solution with a strong oxidizing agent, potassium dichromate. The sample is analyzed colorimetrically at 600 nm.

Methods for the Chemical Analysis of Water and Wastes, USEPA 600/4-79-020. Method 410.4.

Oil and Grease - This is the gravimetric method for liquid samples. Two containers should be submitted for each sample. The sample is acidified to a low pH (<2). A 1-Liter aliquot is extracted with three 30-mL portions of freon. The extracts are passed through sodium sulfate to remove any water and are combined in a tared vessel. The freon is evaporated and the residue is weighed to a constant weight.

Methods for the Chemical Analysis of Water and Wastes, USEPA 600/4-79-020, Method 413.1.

Ammonia Nitrogen - The sample is buffered to a pH of 9.5 with borate buffer and is then distilled into a solution of boric acid. The ammonia in the distillate is titrated with standard sulfuric acid using a mixed indicator.

Methods for Chemical Analysis of Water and Wastes, USEPA 600/4-79-020. Method 350.2.

pH - The activity of hydrogen ions in the sample is measured using a glass electrode and a reference electrode.

Methods for the Chemical Analysis of Water and Wastes, USEPA 600/4-79-020, Method 150.1.

Nitrate Nitrogen - A small volume of sample is introduced into an ion chromatograph. The anions are then separated and measured by a system consisting of a guard column, separator column, suppressor, and conductivity detector. A Dionex Model 2010 Ion Chromatograph is used.

Methods for Chemical Analysis of Water and Wastes, USEPA 600/4-79-020. Method 300.0.

Kjeldahl Nitrogen - The sample is digested with sulfuric acid, potassium sulfate, and mercuric sulfate. This solution is then analyzed for the converted ammonia nitrogen using the reaction of the ammonia and sodium salicylate, sodium nitroprusside, and sodium hypochlorite in a buffered alkaline medium to form an ammonia salicylate complex. The absorbance is read at 660 nm and is compared to a standard curve. An Autoanalyzer is used.

Methods for Chemical Analysis of Water and Wastes, USEPA 600/4-79-020. Method 351.2.

Phosphorus - All forms of phosphorus are converted to orthophosphate by an acid-persulfate digestion. The orthophosphate ion reacts with ammonium molybdate in acidic solution to form an antimony-phosphomolybdate complex. On reduction with ascorbic acid, this complex turns blue. The absorbance is read at 660 nm and is compared to a standard curve. An Autoanalyzer is used.

Methods for Chemical Analysis of Water and Wastes, USEPA 600/4-79-020. Method 365.1.

Total Suspended Solids - A well-mixed sample is filtered through a tared gooch crucible. The residue on the filter is dried to a constant weight at 1030 to 1050C. The increase in weight is the Total Suspended Solids.

Methods for Chemical Analysis of Water and Wastes, USEPA 600/4-79-020. Method 160.2.

Moisture - A known sample weight is placed in a drying oven maintained at 1030 to 1050 for 12 to 24 hours. The sample is reweighed after drying and this value is divided by the original weight. The result is used to calculate analytical concentration on a dry weight basis.

Methods for Chemical Analysis of Water and Wastes, USEPA 600/4-79-020. Method 160.3.

Total Organic Carbon (TOC) - Following acidification, the sample is purged with nitrogen to remove inorganic carbon. Persulfate is injected to oxidize organic carbon to carbon dioxide which is detected by IR. An OI Model 700 TOC Analyzer is used. Method 9060.

Inductively Coupled Plasma (ICP) – This is a technique for the simultaneous determination of elements in solution after acid digestion. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio frequency inductively coupled plasma. Because the temperature of the plasma is considerably highter, it is especially useful for refractory metals. Method 6010B.

The Trace ICP is the same technique as the ICP listed above except for the orientation of the plasma (horizontal instead of vertical) and upgraded optical and sample introduction systems, resulting in instrument detection limits approximately a magnitude lower than the traditional ICP.

Total Cyanide Analysis – Distillation of the sample releases the cyanide from cyanide complexes as HCN. The liberated HCN and simple cyanides are converted to cyanogen chloride by reaction with chloramine T. This reacts with pyridine and barbituric acid reagent to give a red-colored complex. The absorbance is read at 570 nm and is compared to a standard curve. An autoanalyzer is used. Method 9012A.

PAHs by GC/MS 8270				
	Waters		Soils**	
Compound	LOQ* (µg/L)	J-Value (µg/L)	LOQ* (µg/kg)	J-Value (µg/kg)
Naphthalene	10.	1.	330.	33.
Acenaphthylene	10.	1.	330.	33.
Acenaphthene	10.	1.	330.	33.
Fluorene	10.	1.	330.	33.
Phenanthrene	10.	1.	330.	33.
Anthracene	10.	1.	330.	33.
Fluoranthene	10.	1.	330.	33.
Pyrene	10.	1.	330.	67.
Benzo(a)anthracene	10.	1.	330.	33.
Chrysene	10.	1.	330.	33.
Benzo(b)fluoranthene	10.	2.	330.	67.
Benzo(K)fluoranthene	10.	2.	330.	133.
Benzo(a)pyrene	10.	2.	330.	67.
Indeno(1,2,3-cd)pyrene	10.	2.	330.	67.
Dibenzo(a,h)anthracene	10.	2.	330.	67.
Benzo(ghi)perylene	10.	2.	330.	67.

*Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

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

The laboratory routinely reports at the limit of quantitation (LOQ) but can estimate down to the "J"-Value when requested by the client. Values reported below the LOQ are reported with a J-flag and are defined as estimated values.

Volatiles by GC				
	Waters		Soils**	
Compound	LOQ* (µg/L)	J-Value (µg/L)	LOQ* (µg/kg)	J-Value (µg/kg)
Benzene	1.	.2	20.	4.
Toluene	1.	.2	20.	4.
Ethylbenzene	1.	.2	20.	4.
m,p-Xylene***	2.	.4	40.	8.
O-Xylene***	1.	.2	20.	4.
Chloromethane	5.	.5	100.	10.
Bromomethane	5.	.5	100.	10.

*Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

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

***The laboratory will report Xylene (Total) when requested by the client.

The laboratory routinely reports at the limit of quantitation (LOQ) but can estimate down to the "J"-Value when requested by the client. Values reported below the LOQ are reported with a J-flag and are defined as estimated values.

PAHs by HPLC 8310				
Compound	Waters		Soils**	
	LOQ* (µg/L)	J-Value (µg/L)	LOQ* (µg/kg)	J-Value (µg/kg)
Naphthalene	8.	0.8	270.	27.
Acenaphthylene	8.	0.8	270.	27.
Acenaphthene	8.	0.8	270.	27.
Fluorene	0.8	0.17	27.	2.5
Phenanthrene	0.3	0.046	11.	1.
Anthracene	0.2	0.031	5.	0.5
Fluoranthene	0.2	0.02	5.	0.5
Pyrene	0.8	0.18	27.	2.5
Benzo(a)anthracene	0.08	0.018	3.	0.25
Chrysene	0.3	0.059	11.	1.
Benzo(b)fluoranthene	0.06	0.035	2.	0.2
Benzo(k)fluoranthene	0.06	0.027	2.	0.2
Benzo(a)pyrene	0.08	0.022	3.	0.25
Dibenzo(a,h)anthracene	0.2	0.47	5.	0.5
Benzo(g,h,i)perylene	0.5	0.099	16.	1.5
Indeno(1,2,3-cd)pyrene	0.3	0.064	11.	1.

PAHs by Semi VOA 625		
Compound	Waters	
	LOQ* (µg/L)	J-Value (µg/L)
Naphthalene	10.	0.4
Acenaphthylene	10.	0.7
Acenaphthene	10.	0.5
Fluorene	10.	0.6
Phenanthrene	10.	0.5
Anthracene	10.	0.6
Fluoranthene	10.	0.5
Pyrene	10	0.7
Benzo(a)anthracene	10.	0.4
Chrysene	10	0.5
Benzo(b)fluoranthene	10	0.6
Benzo(k)fluoranthene	10	0.6
Benzo(a)pyrene	10	0.7
Dibenzo(a,h)anthracene	10	0.9
Benzo(g,h,i)perylene	10	2.0
Indeno(1,2,3-cd)pyrene	10	2.0

*Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

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

The laboratory routinely reports at the limit of quantitation (LOQ) but can estimate down to the "J"-Value when requested by the client. Values reported below the LOQ are reported with a J-flag and are defined as estimated values.

Parameter	Waters		Soils**	
	LOQ* (mg/L)	J-Value (mg/L)	LOQ* (mg/kg)	J-Value (mg/kg)
TOC	1.0	.3	50.	12.
Ammonia-N	1.	.16	20.	5.
Kjeldahl-N	2.	.7	50.	38.
Phosphorus	.05	.04	Wt. dependent	4.
pH	0.01	.01	0.01	.01
Nitrate-N	0.1	.004	1.0	.09
COD	50.	5.44	50.	12.13
BOD	2.0	.56	NA	NA
TSS	9.0	3.36	NA	NA
Oil and Grease	.4	.14	50.	12.9

*Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

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

Inorganic Appendix IX Analyte List		
	Waters	
Analyte	LOQ* (mg/L)	J-Value (mg/L)
Cadmium	0.01	0.0027
Copper	0.025	0.0038
Lead[1]	0.005	0.0020
Mercury[2]	0.0002	0.000043
Nickel	0.05	0.0054
Silver	0.02	0.0036
Zinc	0.025	0.012

[1]Analysis by Trace ICP

[2]Analysis by Cold Vapor

Except for cyanide and sulfide, all other elements analyzed by ICP.

*Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

The laboratory routinely reports at the limit of quantitation (LOQ) but can estimate down to the J-value when requested by the client. Values reported below the LOQ are reported with a J-flag and are defined as estimated values.

LOQ and J-values are evaluated annually and subject to change.

10. Data Reduction, Validation and Reporting

Raw analytical data generated in the laboratories is collected on printouts from the instruments and associated data system or manually in bound notebooks. Analysts review data as it is generated to determine that the instruments are performing within specifications. This review includes calibration checks, surrogate recoveries, blank checks, retention time reproducibility, and other QC checks described in Section No. 11. If any problems are noted during the analytical run, corrective action is taken and documented.

Each analytical run is reviewed by a chemist for completeness and accuracy prior to interpretation and data reduction. The following calculations are used to reduce raw data to reportable results.

GC/MS calculation used by the data system to determine concentration in extract for semivolatiles or in the sample itself for volatiles:

$$Q = \frac{(Ax)(Is)}{(Als)(RRF)(Vi)}$$

Where:

Ax = peak area

Als = internal standard peak area

Is = amount of internal standard injected (ng)

RRF = relative response factor

Vi = volume of extract injected (μL) or volume sample purged (mL)

The extract concentration is further reduced by considering the initial sample weight or volume and the final extract volume:

$$Concentration = \frac{(Q)\,(D)\,(F)\,(1000)}{(I)}$$

Where:

Q = concentration determined by the data system (mg/L)

D = dilution factor if needed

F = final extract volume (mL)

I = initial sample weight (grams) or volume (mL)

Results are reported in µg/L for water samples and µg/kg for solid samples. Soil samples are reported on an as received and on a dry weight basis. The results are reported on Lancaster Laboratories Analysis Report Forms shown in Appendix A.

For Volatiles by GC, a calibration is performed with a minimum of five levels using either an internal standard calibration or external calibration.

A. Internal standard calibration

$$CF = \frac{(As)\,(Cis)}{(Ais)\,(Cx)} \quad or \quad CF = \frac{(Hx)\,(Cis)}{(His)\,(Cx)}$$

Where:

Ax = Peak area of the compound to be measured in that level of the initial calibration

Hx = Height area of the compound to be measured in that level of the initial calibration

Ais = Peak area of the internal standard

B. External Calibration

$$CF = \frac{Ax}{CF} \times DF \quad or \quad \frac{Hx}{CF} \times DF$$

$$Concentration = \frac{Ax}{CF} \times DF \quad or \quad \frac{Hx}{CF} \times DF$$

When all parameters are defined in A above.

Results are reported in mg/L for water samples and mg/kg for solid samples. Soil samples are reported on an as-received and on a dry-weight basis. Results are reported on Lancaster Labs Analysis Report Forms shown in Appendix A.

The results for the **PAHs by HPLC** analyses are calculated using the following equation:

$$\frac{Pk\ Ht \times FV \times DF \times AF}{ARF \times IV\ (or\ IW)} = Concentration\ (mg/L)\ or\ \mu g/kg$$

Where:

Pk Ht = Peak height found in sample

ARF = Average response factor (Pk Ht/Concentration of analyte in standard)

FV = Final volume of sample extract (mL)

DF = Dilution factor (where applicable)

IV = Initial volume of sample extracted (mL)

IW = Initial weight of the sample extracted (mg)

AF = Additional factor

If a curve is used, then $\frac{Pk\ Ht}{ARF}$ is replaced by the following in the preceding equation:

$$\frac{Pk\ Ht\ -\ y\text{-}intercept}{slope}$$

Results are reported as µg/L for water samples and µg/kg for solid samples. Soil samples are reported on an as received and on a dry weight basis. Results are reported on Lancaster Labs Analysis Report Forms shown in Appendix A.

The results for **inorganic** analyses are calculated using the following equation:

$$Concentration = (A)(D)(E)\ /\ (F)$$

Where:

A = The concentration determined by AA, ICP, or FTIR using calibration data programmed into the instrument (mg/L)

D = Dilution factor if needed

E = Final extract volume (mL)

F = Initial sample volume (mL) or weight (gm)

Results are usually reported in mg/L for water samples and in mg/kg for solid samples. Alternate units are available upon request. Soil samples are reported on an as received and on a dry weight basis. The results are reported on Lancaster Labs Analysis Report Forms shown in Appendix A.

The principle criteria used to validate data will be the acceptance criteria described in Sections No. 8 and 11 and protocols specified in laboratory SOPs. Following review, interpretation and data reduction by the analyst, data is transferred to the laboratory sample management system either by direct data upload from the analytical data system or manually. This system stores client information, sample results, and QC results. A security system is in place to control access of laboratory personnel and to provide an audit trail for information changes. The data is again reviewed by the Group Leader or another analyst whose function is to provide an independent review and verified on the sample management system. The person performing the verification step reviews all data including quality control information prior to verifying the data. Any errors identified and corrected during the review process are documented and addressed with appropriate personnel to ensure generation of quality data. If data package deliverables have been requested, the laboratory will complete the appropriate forms (see Appendix A) summarizing the quality control information, and transfer copies of all raw data (instrument printouts, spectra, chromatograms, laboratory notebooks, etc.) to the Data Packages Group. This group will combine the information from the various analytical groups and the analytical reports from the laboratory sample management system into one package in the client requested format. This package is reviewed by the Quality Assurance Department for conformance with SOPs and to ensure that all QC goals have been met. Any analytical problems are discussed in the case narrative, which is also included with the data package deliverables.

The validation of the data by the Quality Assurance Department includes spot checking raw data versus the final report, checking that all pertinent raw data is included and does refer to the samples analyzed, review of all QC results for conformance with the method, and review of the case narrative for description of any unusual occurrences during analysis. This validation is performed using techniques similar to those used by the Sample Management Office for the USEPA's Contract Laboratory Program. The validation performed by the laboratory does not address useability of the data, which usually requires some knowledge of the site. The laboratory will make every attempt to meet the requirements of this QAPP, thus reducing the need to assess useability of the data.

The laboratory sample management system is programmed to accept and track the results of quality control samples including blanks, surrogates, recoveries, duplicates, controls, and reference materials. The computer is programmed with the acceptance criteria for each type of QC sample and will display an out-of-spec message if the data is not within specifications. All data outside of specifications appears on a report to the Quality Assurance Department on the next working day. These are reviewed by the Quality Assurance Department for severity of the problems and trends in the data. The reports are then sent to the analytical groups for the purpose of documenting the corrective action taken. The sample management system also produces control charts and has searching capabilities to aid in data review. The flow of data from the time the samples enter the laboratory until the data is reported are summarized in Table 10-1.

Any data recorded manually will be collected in bound notebooks. All entries will be in ink, with no erasures or white-out being permitted. Any changes in data will be made using a single line to avoid obliteration of the original entry and will be dated and signed. Any data resulting from instrument printouts will be dated and

will contain the signature and/or identification of the analyst responsible for its generation. After copies of the data are incorporated into the data package deliverables, the originals will be stored in locked archives at the laboratory for a period of 10 years.

Project files will be created per client/project and will contain chain-of-custody records, analysis requirements, and laboratory acknowledgements which document samples received, laboratory sample number assignment, and analysis requested.

Raw data is filed per batch number assignment and laboratory sample number which correlates to the sample receipt documents. When the project is complete, all documentation is archived in a limited access area and retained for 10 years.

Table 10-1 Sample and Data Routing at Lancaster Laboratories	
Action	Personnel Involved
Sample received at Lancaster Laboratories	Sample Administration
Sample is entered onto sample management system (lab ID number assigned, analyses scheduled, chain of custody started, storage location assigned)	Sample Administration
Sample stored in assigned location (refrigerator, freezer, etc.)	Sample Support
Acknowledgement sent to client	Sample Administration
Removed from storage for analysis; necessary aliquot taken and sample returned to storage	Technical Personnel
Analysis is performed according to selected analytical method; raw data recorded, reviewed, and transferred to computer by chemist or technician*	Technical Personnel
Computer performs calculations as programmed according to methods	Data Processing
Chemist or supervisor verifies raw data	Technical Personnel
Data package deliverables are assembled	Data Package Group
Data packages are reviewed prior to mailing	Quality Assurance Dept. Laboratory Management

* Analyses requiring the chemist's interpretation may involve manual data reduction prior to entry onto the computer.

11. Internal Quality Control Checks

The particular types and frequencies of quality control checks analyzed with each sample are defined in the Chemical Analysis of Water and Wastes, USEPA 6004-79-020 and in USEPA SW-846 3rd Edition, Update III, 1996. The quality control checks routinely performed during sample analysis include surrogates, matrix spikes, duplicates, blanks, internal standards, and laboratory control samples.

Surrogates (used for organic analysis only) - Each sample, matrix spike, matrix spike duplicate, and blank are spiked with surrogate compounds prior to purging and extraction in order to monitor preparation and analysis. Surrogates are used to evaluate analytical efficiency by measuring recovery.

Matrix Spikes - A matrix (soil or water) is spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

Duplicates (matrix spike duplicate - organics and inorganic hydride generation; duplicate - inorganics) - A second aliquot of a matrix/sample is analyzed at the same time as the original sample in order to determine the precision of the method. Recovery of the original compared to the duplicate is expressed as relative percent differences (RPD).

Blanks (Method, Preparation) - Blanks are an analytical control consisting of a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples. (Metals use a digested reagent blank

with soils.) They are treated with the same reagents, internal standards, and surrogate standards and carried through the entire analytical procedure. The blank is used to define the level of laboratory background contamination.

Internal Standards (used for GC/MS analysis) - Internal standards are compounds added to every standard, blank matrix, spike, matrix spike duplicate, and sample at a known concentration, prior to analysis. Comparison of the peak areas of the internal standards are used for internal standard quantitation as well as to determine when changes in the instrument response will adversely affect quantification of target compounds.

Laboratory Control Samples - Aqueous and solid control samples of known composition are analyzed using the same sample preparation, reagents, and analytical methods employed for the sample. For inorganics, LCS recovery must fall within established control limits. For organics, an LCS is run when MS/MSD recovery falls outside established limits. The LCS recovery must fall within acceptance limits based on statistical evaluation of past lab data.

The results of all quality control samples are entered into the computer along with sample results. The computer is programmed to compare the individual values with the acceptance limits. If the results are not within the acceptance criteria, appropriate corrective action is taken where necessary. Management is kept informed by daily reports of QC outliers generated by the computerized system. Monthly reports on results of all QC analyses showing mean and standard deviation will indicate trends or method bias. Control Charts are plotted via computer and may be accessed at any time by all analysts.

The charts that follow show the types and frequency of QC performed, along with the acceptance limits and corrective action.

Table 11-1
Quality Control
GC/MS Semivolatiles

Type	Acceptance Limits (%) Waters	Soils	Frequency	Corrective Action
Surrogate:			Each sample, MS, MSD, LCS, and blank	Repeat extraction and analysis. If reanalysis confirms originals, document on report and/or case narrative
Nitrobenzene-d5	47-114	31-126		
2-Fluorobiphenyl	51-106	45-113		
Terphenyl-d14	37-119	37-130		
Phenol-d6	7-74	39-108		
2-Fluorophenol	25-88	35-108		
2,4,6-Tribromophenol	34-125	23-125		
Matrix Spikes: Spike all compounds of interest	See Table 11-2 for acceptance limits		Each group (≤20) of samples per matrix/level	Run LCS for compounds outside recovery window
Laboratory Control Sample: Spike all compounds of interest	Same as for spikes		Each group (≤20) When MS/MSD falls outside established limits.	Re-extract and re-analyze LCS and associated samples for compounds outside acceptance limits
Matrix Spike Duplicates (RPD): Same as for matrix spikes	≤30%		Each group (≤20) of samples per matrix/level	Evaluated by analyst in relationship to other QC results
Blanks:	≤LOQ for all compounds		Once per case or group (≤20) of samples, each matrix, level, instrument	Re-extract and re-analyze blank and associated samples
Internal Standards:	-50 to +100 of internal standard area of 12 hour STD		Each sample, MS, MSD, LCS, and blank	Re-analyze samples. If re-analysis confirms original, document on report and/or case narrative
1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	RT change ≤30 sec.			

Accuracy is subject to change over time.

Table 11-2

PAHs by GC/MS Matrix Spike/
Matrix Spike Duplicate Sample Recovery

Compound Name	WATER		SOIL	
	LCS/LCSD (%)	MS/MSD (%)	LCS/LCSD (%)	MS/MSD (%)
Naphthalene	58 – 99	50 – 106	60 – 97	41 – 115
Acenaphthylene	64 – 100	61 – 103	62 – 101	42 – 119
Acenaphthene	61 – 100	60 – 101	61 – 100	47 – 114
Fluorene	61 – 108	59 – 110	59 – 109	59 – 121
Phenanthrene	68 – 102	64 – 105	62 – 107	54 – 120
Anthracene	66 – 101	62 – 103	62 – 105	42 – 119
Fluoranthene	66 – 106	61 – 109	58 – 110	26 – 137
Pyrene	58 – 112	55 – 114	52 – 115	52 – 115
Benzo(a)anthracene	69 – 101	64 – 103	63 – 106	33 – 135
Chrysene	67 – 101	63 – 104	60 – 107	9 – 153
Benzo(b)fluoranthene	64 – 101	54 – 108	59 – 105	24 – 148
Benzo(k)fluoranthene	67 – 105	59 – 112	63 – 108	41 – 126
Benzo(a)pyrene	65 – 101	60 – 102	61 – 107	21 – 139
Indeno(1,2,3-cd)pyrene	59 – 111	55 – 114	55 – 111	28 – 127
Dibenz(a,h)anthracene	66 – 117	57 – 124	60 – 117	11 – 152
Benzo(g,h,i)perylene	52 – 113	12 – 133	55 – 115	49 – 121

Acceptance limits are based on statistical evaluation of compiled laboratory data and are subject to change.

Table 11-3

Quality Control

PAHs by HPLC (8310)

Type	Acceptance Limits (%) WATERS	SOILS	Frequency	Corrective Action
Surrogate:				
Nitrobenzene or	60 – 120	50 – 120	Added to each sample, MS/MSD, LCS, blank, LCS/LCSD during the extraction phase	Surrogate must be in spec unless matrix-related problems are evident. If matrix-related problems are evident, report results and comment in case narrative.
Triphenylene	60 – 120	50 – 120		
Matrix Spike:				
Spike all compounds of interest	See attached Table 11-4		Each group (≤20) of samples per matrix/level	Run LCS for compounds outside acceptance window
Laboratory Control Sample:				
Spike all compounds of interest	See attached Table 11-4		Each group (≤20) When MS/MSD falls outside established limits.	Re-extract and re-analyze LCS and associated samples for compounds outside acceptance limits
Matrix Spike Duplicates (RPD):				
Spike all compounds of interest	≤30%	≤50%	Each group (≤20) of samples per matrix/level	Evaluated by analyst in relationship to other QC results

Table 11-3

Quality Control

PAHs by HPLC (8310)

Type	Acceptance Limits (%) WATERS	SOILS	Frequency	Corrective Action
Blanks:	≤LOQ for all compounds		Once per case or extraction group (≤20) of samples, each matrix, level, instrument	Inject a hexane or solvent blank first to be sure the analytical system is clean then reinject the blank itself. If the reinjected blank is acceptable, any samples extracted with this blank should be reinjected if they, too, contain the analyte which was contaminating the blank. If the reinjected blank is unacceptable, any affected samples must be re-extracted.

Acceptance limits are based on statistical evaluation of compiled laboratory data and are subject to change.

Table 11-4

Quality Control

PAHs by HPLC Spike Acceptance Limits

Compound Name	Matrix Spike and Laboratory Control Sample Limits	
	Waters (%)	Soils (%)
Napthalene	37 – 120	31 – 162
Acenaphthylene	41 – 135	39 – 166
Acenaphthene	38 – 135	38 – 170
Fluorene	41 – 140	40 – 175
Phenanthrene	48 – 152	47 – 176
Anthracene	42 – 143	38 – 164
Fluoranthene	48 – 155	48 – 167
Pyrene	51 – 146	42 – 162
Benzo(a)anthracene	52 – 146	51 – 148
Chrysene	56 – 145	52 – 148
Benzo(b)fluoranthene	59 – 141	55 – 142
Benzo(k)fluoranthene	60 – 137	56 – 139
Benzo(a)pyrene	42 – 158	33 – 156
Dibenzo(a,h)anthracene	49 – 142	47 – 139
Benzo(g,h,i)perylene	46 – 148	40 – 150
Indeno(1,2,3-CD)pyrene	64 – 134	50 – 146

Acceptance limits are based on statistical evaluation of compiled laboratory data and are subject to change.

Table 11-5

Petroleum Analysis
Acceptance Criteria

Type	Acceptance Limits (%) WATERS	SOILS	Frequency	Corrective Action
Surrogates: ααα-Trifluorotoluene	70 – 130	20 – 130	Each sample, MS, MSD, LCS, and blank	Reanalyze sample if outside limits; if reanalysis confirms original, document a report and/or case narrative
Matrix Spike: Spike all compounds of interest	See Table 11-6		Each group (≤20) of samples per matrix/level	Run LCS for compound outside of acceptance limits
Laboratory Control Sample: Spike all compounds of interest	See Table 11-6		Each group (≤20) of samples per matrix/level	Reanalyze LCS and associated samples for compounds outside acceptance limits that are also outside MS/MSD acceptance limits
Matrix Spike Duplicates (RPD):	≤30%		Each group (≤20) of samples per matrix/level	Evaluated by an analysis in relationship to other QC results
Blanks:	≤LOQ for all compounds		At least one per 20 samples	Reanalyze blank and associated samples if blank is outside limits
Internal Standards: 1-Chloro-3-fluorobenzene	-50% to +100% if internal standard area		Each sample, MS, MSD, LCS, and blank analyzed on the PID	Reanalyze samples; if reanalysis confirms original, document on report or case narrative

Accuracy is subject to change over time.

Table 11-6

Petroleum Analysis

Compound Name	MS% Water	MS% Soil	Max. % RPD Water	Max. % RPD Soil	LCS % Water	LCS % Soil
Benzene	78 – 138	70 – 130	30	30	78 – 138	70 – 130
Toluene	78 – 118	70 – 130	30	30	78 – 118	70 – 130
Ethylbenzene	77 – 119	70 – 130	30	30	77 – 119	70 – 130
Total Xylenes	76 – 116	70 – 130	30	30	76 – 116	70 – 130

Acceptance limits are based on statistical evaluation of laboratory data and are subject to change.

Table 11-7

Quality Control Acceptance Criteria

Parameter	Blank	Spike Recovery (%)	Duplicate RPD (%)	Lab Control Recovery (%)
TOC	<LOQ	75 – 125	≤20	80 – 120
Moisture	NA	NA	≤20	89.07 - 90.33 % by wt.
Ammonia-N	<LOQ	75 – 125	≤20	80 – 120
Kjeldahl-N	<LOQ	75 – 125	≤20	80 – 120
Phosphorus	<LOQ	75 – 125	≤20	80 – 120
pH	NA	NA	≤20	6.75 - 6.99 pH units
Nitrate-N	<LOQ	75 – 125	≤20	80 – 120
COD	<LOQ	90.7 - 115.7	≤20	469.8 – 526.6 mg/L
BOD	<LOQ	77.1 - 127.7	≤20	174.4 - 261.6 mg/L
TSS	<LOQ	NA	≤20	121.6 - 166.6 mg/L
Oil and Grease	<LOQ	24.5 - 166.1	≤20	34.7 - 59.9 mg/L
Cyanide (reactivity)	<LOQ	N/A	≤20	80 – 120

Corrective Action: If either the LCS or Blank are outside the criteria, the QC and associated samples will be reprepped and re-analyzed.

Maximum batch size is 20 field samples.

Table 11-8

Quality Control
Inorganics

Type	Acceptance Limits (%) WATERS SOILS	Frequency	Corrective Action
Matrix Spikes:	80% to 120% except where sample conc. exceeds spike conc. by ≥4×	Each group of samples of similar matrix/level (≤20) each method	Analyze post-digestion spike sample
Matrix Spike Duplicate RPD):	Same as above ±20% RPD	Each group of samples of similar matrix/level (20) each method	Analyze post-digestion spike sample if not already run for MS, flag the data
Duplicates (RPD):	±20% RPD for sample values ≥5× LOQ	Each group of samples of similar matrix-level (≤20) each method	Flag the data
Blanks: Initial Calibration (ICB) Continuing Calibration (CCB) Preparation Blank	 ≤LOQ ≤LOQ >LOQ then lowest conc. in sample must be 20× blk. conc.	Each wavelength immediately after calibration verification at 10% frequency or every 2 hours (beginning at end of run min.) Each SDG or batch (≤20 samples) Exception: As/Se by Hydride Generation ≤10 samples	Correct problem, recalibrate, and rerun Redigest and reanalyze blank and associated samples if sample result <20× blank result
Serial Dilutions (ICP &GFAA only):	Within ±10% of the original determination	Each group of (≤20) of similar matrix/level	Flag the data
Interference Check Sample (ICP only):	±20% of the true value for the analytes	Each wavelength after Initial Calibration Verification at beginning and end of the run or min. of 2× per 8 hour	Recalibrate the instrument
Laboratory Control Sample:	Aqueous 80% to 120% (except Ag and Sb) Solids commercial certified standard advisory range See Table 11-11	Each SDG or batch (≤20 samples), each method	Redigest and reanalyze LCS and associated samples
Post Digestion Spike:	85% to 115%	When matrix spikes are outside 80% to 120% range (not performed on Hg or GFAA analyses)	Flag the data
Analytical Spike	85% to 115%	One per 20 field samples	See Table 11-10

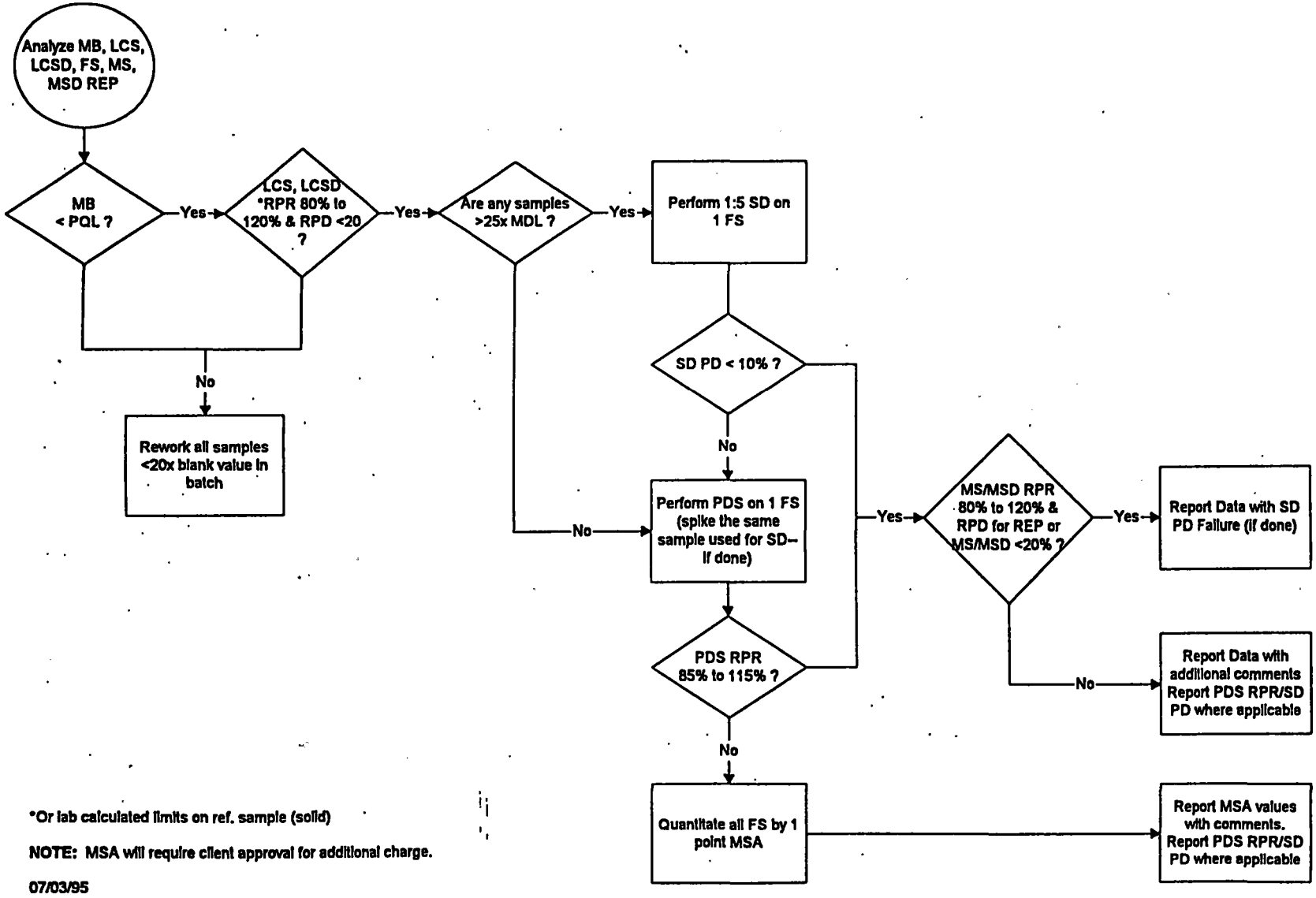
Table 11-9

Quality Control
GC/MS Semivolatiles PAHs (625)

Type	Acceptance Limits (%) Waters	Frequency	Corrective Action
Surrogate: Nitrobenzene-d5 2-Fluorobiphenyl Terphenyl-d14 Phenol-d6 2-Fluorophenol 2,4,6-Tribromophenol	 47 – 114 51 – 106 37 – 119 7 – 74 25 - 88 34 – 125	Each sample, MS, MSD, LCS, and blank	Repeat extraction and analysis. If reanalysis confirms originals, document on report and/or case narrative
Matrix Spikes: Spike all compounds of interest	See Table 11-2 for acceptance limits	Each group (≤20) of samples per matrix/level	Run LCS for compounds outside recovery window
Laboratory Control Sample: Spike all compounds of interest	Same as for spikes	Each group (≤20) when MS/MSD falls outside established limits.	Re-extract and re-analyze LCS and associated samples for compounds outside acceptance limits
Matrix Spike Duplicates (RPD):	≤30%	Each group (≤20) of samples per matrix/level	Evaluated by analyst in relationship to other QC results
Blanks:	≤LOQ for all compounds	Once per case or group (≤20) of samples, each matrix, level, instrument	Re-extract and re-analyze blank and associated samples
Internal Standards: 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	-50 to +100 of internal standard area of 24 hour STD RT change ≤30 sec.	Each sample, MS, MSD, LCS, and blank	Re-analyze samples. If re-analysis confirms original, document on report and/or case narrative

Table 11-10

SW846 Method 7000A GFAA
Batch QC Decision Tree



*Or lab calculated limits on ref. sample (solid)

NOTE: MSA will require client approval for additional charge.

07/03/95

Table 11-11

ENVIRONMENTAL
RESOURCE ASSOCIATES
ARVADA, COLORADO 1-800-372-0122

Certification

PriorityPollutnT™/CLP Inorganic Soils Quality Control Standards

Catalog Nº PPS-46 Lot Nº 229

Parameter	Certified Value	Performance Acceptance Limits™
TRACE METALS PriorityPollutnT™ (Catalog No 540)	mg/Kg	mg/Kg
aluminum	4590	2280 - 7590
antimony	39.8	8.37 - 119
arsenic	75.4	37.1 - 112
barium	106	74.3 - 139
beryllium	51.0	11.7 - 90.3
boron	94.1	26.9 - 161
cadmium	45.4	11.9 - 79.0
calcium	1290	875 - 1750
chromium	71.0	38.0 - 100
cobalt	49.6	29.8 - 70.5
copper	112	63.9 - 162
iron	9160	5560 - 13000
lead	53.5	28.1 - 75.9
magnesium	1160	691 - 1670
manganese	154	107 - 208
mercury	1.50	0.389 - 2.35
molybdenum	47.4	29.2 - 70.2
nickel	39.4	21.5 - 57.5
potassium	1420	880 - 1870
selenium	72.3	37.8 - 108
silver	116	58.2 - 170
sodium	198	111 - 287
strontium	109	46.3 - 173
thallium	40.0	20.0 - 60.0
tin	102	35.9 - 168
titanium	230	60.0 - 400
vanadium	65.9	32.0 - 88.9
zinc	134	72.2 - 199
CYANIDE PriorityPollutnT™ (Catalog No 541)	mg/Kg	mg/Kg
total cyanide	323	123 - 559

The ***Trace Metals Certified Values*** are equal to the mean recoveries for each parameter as determined in an interlaboratory round robin study. The standard was digested using Method 3050, SW-846 and the digest analyzed by ICP and atomic absorption spectroscopy.

The ***Cyanide Certified Value*** is equal to the mean recovery as determined in an interlaboratory round robin study. The standard was distilled and analyzed following the procedure outlined in Method 9010, SW-846.

The ***Performance Acceptance Limits (PALs™)*** are listed as guidelines for acceptable analytical results given the limitations of the USEPA methodologies commonly used to determine these parameters and closely approximate the 95% confidence interval. The PALs™ are based on data generated by your peer laboratories in ERA's InterLaB™ program using the same samples you are analyzing and data from USEPA methods, WP, WS and CLP interlaboratory studies. If your result falls outside of the PALs™, ERA recommends that you investigate potential sources of error in your preparation and/or analytical procedures. For further technical assistance, call ERA at 1-800-372-0122.

For users of internal standards, ERA has determined that scandium is present in this soil at 1.66 mg/Kg and that yttrium is present at 9.43 mg/Kg.

*Each lot of standards will have different certified values and the advisory range will be adjusted accordingly.

12. Performance and System Audits

System audits are conducted on each department at Lancaster Laboratories by members of the Quality Assurance Department. The audits include checks on methodology, reagent preparation, equipment calibration and maintenance, quality control results, and training of personnel. The results of the audits and corrective action, where necessary, are communicated to laboratory personnel and management by means of a written report. Audits by outside organizations including clients, regulatory personnel, and the USEPA are permitted by arrangement with the Quality Assurance Department.

The Quality Assurance Department reviews summaries of the quality control data entered onto the computerized sample management system by analysts. Control charts and statistics are reviewed for trends which may indicate problems with the analytical data. In this way, small problems are identified before they have any significant impact on laboratory results.

Performance audits consist of both intralaboratory and interlaboratory check samples. QC samples from commercial suppliers are analyzed quarterly to assess laboratory accuracy including a double blind program. The Laboratory also participates in a number of interlaboratory performance evaluation studies which involve analysis of samples with concentrations of analytes that are known to the sponsoring organization, but unknown to the laboratory. Inorganics, pesticide/herbicides, trihalomethanes, volatile organic compounds, semivolatile organic compounds, and traditional wet chemistry analyses are analyzed by Lancaster Laboratories for studies conducted by the USEPA and the New York Department of Health. Lancaster Laboratories has participated

in the USEPA Contract Laboratory Program which provides laboratory analysis in support of the Superfund program. Part of maintaining this contract includes analysis of quarterly blind samples. Representative results from some of these studies are attached to this section

Report: PE005
Performance Evaluation Report Page: 1
USEPA Water Supply Study WS041 Date: 30SEP98

Participant ID: PA00009 Lancaster Labs Type: OTHER Requesting Office: NH

Sample Number	Reported Value	True Value*	Acceptance Limits	Performance Evaluation
TRACE METALS IN MICROGRAMS PER LITER:				
001-ARSENIC				
001	67.6	65.6	58.2- 72.9	Accept.
003-CADMIUM				
001	17.7	18.2	14.6- 21.8	Accept.
004-CHROMIUM				
001	55.2	55.5	47.2- 63.8	Accept.
005-LEAD				
001		Not Evaluated in this Study		
006-MERCURY				
001	5.50	5.82	4.07- 7.57	Accept.
007-SELENIUM				
001	46.8	46.3	37- 55.6	Accept.
091-COPPER				
001	692.	702	632- 772	Accept.
140-ANTIMONY				
001	31.8	31.4	22- 40.8	Accept.
141-BERYLLIUM				
001	2.44	2.58	2.19- 2.97	Accept.
142-NICKEL				
001	348.	352	299- 405	Accept.
143-THALLIUM				
001	3.59	3.50	2.45- 4.55	Accept.
226-BORON				
002	819.	790	736- 874	Accept.
236-MANGANESE				
002	182.	183	167- 196	Accept.
237-MOLYBDENUM				
002	78.7	76.7	66.2- 86.4	Accept.
239-ZINC				
002	380.	402	359- 441	Accept.
NITRATE/NITRITE/FLUORIDE IN MILLIGRAMS PER LITER:				
009-NITRATE AS N				
001	12.4	15.0	13.5- 16.5	Not Accept.
010-FLUORIDE				
001	5.61	6.20	5.58- 6.82	Accept.
092-NITRITE AS N				
001	1.80	1.70	1.45- 1.96	Accept.
261-ORTHOPHOSPHATE AS P				
001	1.26	1.30	1.19- 1.39	Accept.
INSECTICIDES IN MICROGRAMS PER LITER:				
011-ENDRIN				
001	0.784	0.789	0.552- 1.03	Accept.
012-LINDANE				
001	2.69	2.50	1.38- 3.63	Accept.

Report: PE005
Performance Evaluation Report Page: 2
USEPA Water Supply Study WS041 Date: 30SEP98

Participant ID: PA00009 Type: OTHER Requesting Office: NH

Sample Number	Reported Value	True Value*	Acceptance Limits	Performance Evaluation
013-METHOXYCHLOR				
001	26.4	26.8	14.7- 38.9	Accept.
014-TOXAPHENE				
002	7.29	6.90	3.8- 10	Accept.
093-ALACHLOR				
004	12.7	12.9	7.1- 18.7	Accept.
094-ATRAZINE				
004	14.3	14.5	7.98- 21	Accept.
095-HEPTACHLOR				
001	0.710	0.830	0.457- 1.2	Accept.
096-HEPTACHLOR EPOXIDE				
001	0.620	0.630	0.347-0.914	Accept.
097-CHLORDANE (TOTAL)				
003	2.57	2.90	1.6- 4.21	Accept.
112-HEXACHLOROCYCLOPENTADIENE				
001	1.08	1.93	0.0861- 2.58	Accept.
113-SIMAZINE				
004	13.1	11.8	3.29- 17.9	Accept.
172-HEXACHLOROBENZENE				
001	1.00	1.03	0.426- 1.23	Accept.
244-TRIFLURALIN				
001	3.23	3.82	1.55- 5.4	Accept.
256-ALDRIN				
001	1.01	1.23	0.505- 1.56	Accept.
258-DIELDRIN				
001	0.928	0.920	0.622- 1.14	Accept.
259-PROPACHLOR				
001	5.38	5.02	3.14- 6.8	Accept.
CARBAMATES IN MICROGRAMS PER LITER:				
098-ALDICARB				
001	35.7	35.3	26.6- 45.1	Accept.
099-ALDICARB SULFONE				
001	14.8	15.3	10.4- 17.5	Accept.
100-ALDICARB SULFOXIDE				
001	23.0	26.0	16.6- 30	Accept.
101-CARBOFURAN				
001	43.4	43.7	24- 63.4	Accept.
114-OXAMYL (VYDATE)				
001	31.8	33.8	23.6- 40.8	Accept.
245-METHOMYL				
001	217.	238	187- 270	Accept.
HERBICIDES IN MICROGRAMS PER LITER:				
015-2,4-D				
001	49.7	73.1	36.6- 110	Accept.
016-2,4,5-TP (SILVEX)				
001	21.9	24.1	12.1- 36.2	Accept.

Report: PE005
Page: 3
Date: 30SEP98

Performance Evaluation Report
USEPA Water Supply Study WS041

Participant ID: PA00009 Type: OTHER Requesting Office: NH

Sample Number	Reported Value	True Value*	Acceptance Limits	Performance Evaluation
102-PENTACHLOROPHENOL				
001	24.9	34.6	17.3- 51.9	Accept.
115-DALAPON				
001	143.	183	D.L. - 258	Accept.
116-DINOSEB				
001	13.9	27.6	0.568- 41.9	Accept.
117-PICLORAM				
001	47.0	62.1	D.L. - 86.9	Accept.
247-DICAMBA				
001	94.4	123	32.1- 167	Accept.
262-ACIFLUORFEN				
001	71.4	72.1	25.4- 101	Accept.
POLYCHLORINATED BIPHENYLS IN MICROGRAMS PER LITER:				
118-DECACHLOROBIPHENYL				
001	1.98	1.80	D.L. - 3.6	Accept.
PAH'S IN MICROGRAMS PER LITER:				
122-BENZO(A)PYRENE				
001	1.28	2.37	0.502- 2.87	Accept.
ADIPATE/PHTHALATES IN MICROGRAMS PER LITER:				
134-DI (2-ETHYLHEXYL)ADIPATE				
001	4.56	4.20	1.6- 7.56	Accept.
136-DI (2-ETHYLHEXYL)PHTHAL.				
001	16.0	15.3	6.95- 24.7	Accept.
TRIHALOMETHANES IN MICROGRAMS PER LITER:				
017-CHLOROFORM				
001	13.2	14.4	11.5- 17.3	Accept.
018-BROMOFORM				
001	14.5	16.6	13.3- 19.9	Accept.
019-BROMODICHLOROMETHANE				
001	10.7	12.3	9.84- 14.8	Accept.
020-CHLORODIBROMOMETHANE				
001	16.3	19.4	15.5- 23.3	Accept.
021-TOTAL TRIHALOMETHANE				
001	54.7	62.7	50.2- 75.2	Accept.
VOLATILE ORGANIC COMPOUNDS IN MICROGRAMS PER LITER:				
032-VINYL CHLORIDE				
001	24.1	22.3	13.4- 31.2	Accept.
034-1,1-DICHLOROETHYLENE				
001	6.60	5.25	3.15- 7.35	Accept.
035-1,2-DICHLOROETHANE				
001	15.4	13.7	11- 16.4	Accept.
036-1,1,1-TRICHLOROETHANE				
001	13.7	12.6	10.1- 15.1	Accept.

Report: PE005
Performance Evaluation Report — Page: 4
USEPA Water Supply Study WS041 — Date: 30SEP98

Participant ID: PA00009 Type: OTHER Requesting Office: NH

Sample Number	Reported Value	True Value*	Acceptance Limits	Performance Evaluation
037-CARBON TETRACHLORIDE				
001	15.7	14.2	11.4- 17	Accept.
038-TRICHLOROETHYLENE				
001	7.33	6.87	4.12- 9.62	Accept.
039-BENZENE				
001	19.6	18.7	15- 22.4	Accept.
040-TETRACHLOROETHYLENE				
001	10.5	11.5	9.2- 13.8	Accept.
041-1,4-DICHLOROBENZENE				
001	15.8	15.8	12.6- 19	Accept.
042-T 1,2 DICHLOROETHYLENE				
001	17.1	18.5	14.8- 22.2	Accept.
043-C 1,2 DICHLOROETHYLENE				
001	22.8	25.3	20.2- 30.4	Accept.
044-1,2 DICHLOROPROPANE				
001	15.8	15.4	12.3- 18.5	Accept.
045-1,2DIBROMO3CHLOROPROPANE				
003	0.301	0.451	0.271-0.631	Accept.
046-ETHYLENE DIBROMIDE (EDB)				
003	0.267	0.344	0.206-0.482	Accept.
047-TOLUENE				
001	19.9	18.7	15- 22.4	Accept.
048-ETHYLBENZENE				
001	15.1	14.7	11.8- 17.6	Accept.
049-CHLOROBENZENE				
001	19.3	18.6	14.9- 22.3	Accept.
053-STYRENE				
001	13.1	12.4	9.92- 14.9	Accept.
054-1,2 DICHLOROBENZENE				
001	12.7	11.3	9.04- 13.6	Accept.
055-DICHLOROMETHANE				
001	15.6	15.9	12.7- 19.1	Accept.
060-2,2-DICHLOROPROPANE				
002	13.2	12.7	9.73- 14.4	Accept.
061-1,1,2-TRICHLOROETHANE				
001	12.6	13.3	10.6- 16	Accept.
063-1,1,1,2TETRACHLOROETHANE				
002	14.9	15.2	12- 17.5	Accept.
064-1,2,3-TRICHLOROPROPANE				
002	15.5	14.8	10.2- 18	Accept.
076-1,2,4-TRICHLOROBENZENE				
001	14.6	14.2	11.4- 17	Accept.
077-1,2,3-TRICHLOROBENZENE				
002	20.6	18.4	12.7- 21.5	Accept.
081-HEXACHLOROBUTADIENE				
002	13.5	11.6	8.53- 14.5	Accept.
090-TOTAL XYLENES				
001	34.8	30.8	24.6- 37	Accept.

Report: PE005
Performance Evaluation Report — Page: 5
USEPA Water Supply Study WS041 — Date: 30SEP98

Participant ID: PA00009 Type: OTHER Requesting Office: NH

Sample Number	Reported Value	True Value*	Acceptance Limits	Performance Evaluation
152-C 1,3 DICHLOROPROPENE				
002	13.7	15.2	12.1- 17.4	Accept.
153-T 1,3 DICHLOROPROPENE				
002	13.0	13.7	10- 15.6	Accept.
MISCELLANEOUS ANALYTES:				
022-RESIDUAL FREE CHLORINE(MILLIGRAMS PER LITER)				
001	1.79	1.90	1.55- 2.32	Accept.
023-TURBIDITY(NTU'S)				
001	2.89	2.60	2.37- 3.31	Accept.
024-TOTAL FILTERABLE RESIDUE(MILLIGRAMS PER LITER)				
001	407.	474	287- 826	Accept.
025-CALCIUM HARDNESS(MG. CACO3/L)				
001	261.	248	229- 266	Accept.
026-PH-UNITS				
001	9.06	9.13	8.88- 9.28	Accept.
027-ALKALINITY(MG. CACO3/L)				
001	51.6	50.6	48- 56.7	Accept.
029-SODIUM(MILLIGRAMS PER LITER)				
001	23.7	23.3	21.6- 26.3	Accept.
145-SULFATE(MILLIGRAMS PER LITER)				
001	49.6	49.0	44.1- 54.2	Accept.
146-TOTAL CYANIDE(MILLIGRAMS PER LITER)				
001	0.312	0.326	0.245-0.408	Accept.
263-TOC				
001	1.70	1.60	1.21- 2.05	Accept.
264-LOW-LEVEL TURBIDITY(BY BENCH-TOP, PORTABLE, OR IR INSTR.)				
002		Not Evaluated in this Study		
003		Not Evaluated in this Study		
004		Not Evaluated in this Study		

********** END OF DATA FOR PA00009 **********
NOTE: FOR LIMITS AND TRUE VALUES, ASSUME THREE SIGNIFICANT DIGITS.
********** END OF REPORT FOR PA00009 **********

* Based on gravimetric calculations, or a reference value when necessary.

Report: FE005
Performance Evaluation Report — Page: 1
USEPA Water Pollution Study WP040 — Date: 19NOV98

Participant ID: PA0CCC9 Lancaster Labs Type: OTHER Requesting Office: NR

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
TRACE METALS IN MICROGRAMS/LITER					
001-ALUMINUM					
01	3040.	3105	2740- 3440	2830- 3350	Accept.
002-ARSENIC					
01	148.	160	128- 190	136- 183	Accept.
003-BERYLLIUM					
02	36.9	36.9	33.1- 42.2	34.2- 41	Accept.
004-CADMIUM					
01	162.	170	147- 193	152- 187	Accept.
005-COBALT					
01	483.	503	449- 561	464- 547	Accept.
006-CHROMIUM					
01	634.	650	579- 726	598- 708	Accept.
007-COPPER					
01	697.	700	654- 781	670- 765	Accept.
008-IRON					
01	790.	834	744- 936	768- 912	Accept.
009-MERCURY					
01	1.21	1.15	0.813- 1.47	0.895- 1.39	Accept.
010-MANGANESE					
01	230.	240	216- 259	222- 254	Accept.
011-NICKEL					
01	2490.	2501	2340- 2860	2400- 2790	Accept.
012-LEAD					
01	68.4	70.6	61.7- 83.3	64.4- 80.6	Accept.
013-SELENIUM					
01	231.	260	189- 297	202- 284	Accept.
014-VANADIUM					
01	4240.	4202	3880- 4640	3980- 4550	Accept.
015-ZINC					
01	609.	631	563- 709	581- 690	Accept.
016-ANTIMONY					
02	500.	499	381- 590	407- 564	Accept.
017-SILVER					
02	821.	851	736- 930	760- 906	Accept.
018-THALLIUM					
02	830.	841	747- 970	775- 942	Accept.
074-MOLYBDENUM					
02	19.4	18.2	14- 22	15- 21	Accept.
075-STRONTIUM					
02	302.	301	263- 342	273- 332	Accept.
076-TITANIUM					
02	161.	160	142- 177	147- 173	Accept.
MINERALS IN MILLIGRAMS/LITER (EXCEPT AS NOTED)					
020-SPEC. COND.(UMHOS/CM AT 25 C)					
01	490.	525	471- 544	480- 535	Accept.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
NATIONAL EXPOSURE RESEARCH LABORATORY
CINCINNATI, OH 45268

Date: November 30, 1998

OFFICE OF
RESEARCH AND DEVELOPMENT

SUBJECT: Results from Water Pollution Performance Evaluation Study 40 (WP040)

FROM: Paul W. Britton, Statistician
National Water Quality Assurance Programs Branch
Ecological Exposure Research Division

TO: Quality Assurance Coordinators/Officers
Project Officers
Interested Laboratories

THRU: Raymond J. Wesselman, Chief
National Water Quality Assurance Programs Branch
Ecological Exposure Research Division

WP040 has been completed by NERL-Cincinnati in its continuing evaluation of the performance of USEPA, state and other selected laboratories for 80 water pollution analytes (76 possible reported values). As you are all aware, this is the last WP study to be conducted directly by the USEPA.

Addressees that nominated a group of laboratories directly to NERL-Cincinnati will find:

1) a single copy of the performance evaluation report for each laboratory they nominated and related study information;

2) a 3.5" diskette containing the study results as personal computer (PC) files that have been compressed using PKZIP, and directions regarding use of the diskette. Three "PK" commands have also been provided for your use on these files;

3) a printed list of the participating laboratories within their jurisdiction, including their assigned codes and identification of which specific addressee must distribute the report, etc., to EACH laboratory.

The diskette constitutes their file copy of the study results and the printed copy is for distribution to each of their participating laboratories (also see paragraph at the top of page 2 regarding which coordinator has the primary responsibility for distributing the report to each specific lab). As a study coordinator, it is also their responsibility to provide study results to any interested state or regional official within their jurisdiction that needs the information and is not listed as an addressee above. Study coordinators may distribute copies of any reports, files or other study information, as they consider necessary.

US ENVIRONMENTAL PROTECTION AGENCY
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-CINCINNATI

TERMS USED FOR WATER POLLUTION (WP) LABORATORY PERFORMANCE EVALUATION STUDIES

TRUE VALUES:	A THEORETICALLY CALCULATED VALUE BASED UPON CAREFUL WEIGHT AND VOLUME MEASUREMENT OF CONSTITUENTS. PLEASE NOTE THAT, FOR SOME ANALYTES, THIS VALUE IS QUITE DIFFERENT FROM THE RESULT THAT CAN BE EXPECTED WHEN THE STUDY SAMPLE IS ANALYZED. WHEN UNAVOIDABLE, THIS MAY BE A REFERENCE VALUE THAT REPRESENTS ACHIEVABLE RESULTS.
ACCEPTANCE LIMITS:	A 99% PREDICTION INTERVAL CALCULATED FROM AVAILABLE PERFORMANCE EVALUATION DATA OF EPA AND STATE LABORATORIES. BY DEFINITION THE ANALYTIC RESULTS FROM A LABORATORY PRODUCING VALID DATA SHOULD FALL WITHIN ACCEPTANCE LIMITS 99 TIMES OUT OF 100.
WARNING LIMITS:	A 95% PREDICTION INTERVAL PRODUCED IN THE SAME WAY AS THE ACCEPTANCE LIMITS. DATA FALLING OUTSIDE THESE LIMITS BUT INSIDE THE ACCEPTANCE LIMITS SHOULD BE REVIEWED FOR POSSIBLE PROBLEMS, BUT SUCH DATA SHOULD NOT NECESSARILY BE CONSIDERED UNACCEPTABLE.

MEANING OF THE PERFORMANCE EVALUATION COMMENTS:

ACCEPT.:	THE REPORTED VALUE WAS WITHIN ALL LIMITS.
CK. FOR ERR.:	THE REPORTED VALUE WAS WITHIN THE ACCEPTANCE LIMITS, I.E., TECHNICALLY ACCEPTABLE, BUT SINCE THE VALUE WAS OUTSIDE OF THE WARNING LIMITS A MARGINAL PROBLEM MAY EXIST.
NOT ACCEPT.:	THE REPORTED VALUE WAS BEYOND THE ACCEPTANCE LIMITS.
UNUSABLE:	THE VALUE WAS REPORTED AS A "LESS THAN" OR "GREATER THAN" VALUE AND COULD NOT BE QUANTITATIVELY JUDGED.

ANY PERFORMANCE EVALUATION OTHER THAN ACCEPTABLE REQUIRES A REVIEW BY THE LABORATORY TO IDENTIFY POSSIBLE DEFICIENCIES OR ERRORS THAT NEED CORRECTING.

Report: EECCS
Page: 2
Date: 19NOV98

Performance Evaluation Report
USEPA Water Pollution Study WP040

Participant ID: PA0CCCS Type: CIREB Requesting Office: BB

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
021-TDS AT 180 C					
01	277.	274	230- 337	243- 323	Accept.
022-TOTAL HARDNESS(AS CACO3)					
01	99.2	105	93.3- 119	96.5- 116	Accept.
023-CALCIUM					
01	28.3	29.0	25.1- 33.2	26.1- 32.2	Accept.
024-MAGNESIUM					
01	7.50	8.00	7.06- 8.86	7.29- 8.64	Accept.
025-SODIUM					
01	40.0	39.0	35.7- 44.1	36.7- 43	Accept.
026-POTASSIUM					
01	24.2	25.0	21.4- 28.8	22.4- 27.9	Accept.
027-TOTAL ALKALINITY(AS CACO3)					
01	42.6	42.4	35.5- 48.1	37.1- 46.5	Accept.
028-CHLORIDE					
01	74.3	74.9	68.1- 83.2	70- 81.3	Accept.
029-FLUORIDE					
01	0.826	0.860	0.738- 1.05	0.777- 1.01	Accept.
030-SULFATE					
01	67.8	69.0	58.3- 78.4	60.8- 75.9	Accept.
NUTRIENTS IN MILLIGRAMS/LITER					
031-AMMONIA-NITROGEN					
01	4.65	4.80	3.89- 5.84	4.13- 5.6	Accept.
032-NITRATE-NITROGEN					
01	11.8	12.0	10.1- 13.9	10.6- 13.4	Accept.
033-ORTHOPHOSPHATE					
01	0.578	0.580	0.496-0.669	0.516-0.648	Accept.
034-KJELDAHL-NITROGEN					
02	4.37	5.40	4.02- 6.79	4.35- 6.45	Accept.
035-TOTAL PHOSPHORUS					
02	3.49	4.00	3.3- 4.68	3.47- 4.52	Accept.
DEMANDS IN MILLIGRAMS/LITER					
036-COD					
01	64.0	60.7	42.5- 73.5	46.5- 69.5	Accept.
037-TOC					
01	24.8	24.0	20.7- 28.1	21.6- 27.2	Accept.
038-5-DAY BOD					
01	42.2	37.6	17.7- 57.2	22.6- 52.2	Accept.
102-CARBONACEOUS BOD					
01	39.0	31.9	14.5- 53.4	19.6- 48.3	Accept.
PCB'S IN MICROGRAMS/LITER					
042-PCB-AROCLOR 1232					
01	3.53	4.73	1.1- 7.18	1.88- 6.41	Accept.
046-PCB-AROCLOR 1260					
02	2.52	3.24	1.29- 4.6	1.71- 4.18	Accept.

Report: EECC5
Page: 3
Date: 15PCT95

Performance Evaluation Report
USEPA Water Pollution Study WP04C

Participant ID: PACCCC9 Type: OTHER Requesting Office: NH

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
PCB'S IN OIL IN MILLIGRAMS/MICROGRAM					
099-PCB IN OIL- 1016/1242					
02	34.7	38.7	3.96- 57.2	10.8- 50.3	Accept.
100-PCB IN OIL- 1254					
01	22.2	24.0	D.L. - 40.2	3.97- 34.9	Accept.
PESTICIDES IN MICROGRAMS/LITER					
047-ALDRIN					
01	0.544	0.940	0.164- 1.45	0.326- 1.29	Accept.
048-DIELDRIN					
01	2.35	2.78	1.55- 3.96	1.86- 3.66	Accept.
049-DDD					
01	5.33	5.25	3.21- 7.12	3.7- 6.62	Accept.
050-DDE					
01	3.20	3.64	1.74- 5.1	2.16- 4.68	Accept.
051-DDT					
01	6.79	8.42	2.61- 12.7	3.89- 11.5	Accept.
052-HEPTACHLOR					
01	1.60	2.45	0.837- 3.41	1.16- 3.08	Accept.
053-CHLORDANE					
02	3.11	4.81	2.08- 6.8	2.68- 6.2	Accept.
078-HEPTACHLOR EPOXIDE					
01	1.37	1.98	1.13- 2.42	1.29- 2.26	Accept.
VOLATILE HALOCARBONS IN MICROGRAMS/LITER					
054-1,2 DICHLOROETHANE					
01	15.8	14.6	9.85- 18.5	10.9- 17.4	Accept.
055-CHLOROFORM					
01	19.0	18.4	13.7- 21.7	14.7- 20.7	Accept.
056-1,1,1 TRICHLOROETHANE					
01	33.8	32.8	22.2- 41.1	24.6- 38.7	Accept.
057-TRICHLOROETHENE					
01	23.2	23.4	15.6- 29.8	17.4- 28	Accept.
058-CARBONTETRACHLORIDE					
01	27.0	26.3	16.3- 32.9	18.3- 30.8	Accept.
059-TETRACHLOROETHENE					
01	32.3	32.5	21.6- 39.5	23.8- 37.2	Accept.
060-BROMODICHLOROMETHANE					
01	17.7	16.5	10.9- 19.8	12- 18.7	Accept.
061-DIBROMOCHLOROMETHANE					
01	30.9	32.7	21.4- 40.6	23.8- 38.2	Accept.
062-BROMOFORM					
01	14.6	14.7	8.12- 18.7	9.44- 17.3	Accept.
063-METHYLENE CHLORIDE					
01	45.4	44.1	29.7- 58.8	33.3- 55.1	Accept.
064-CHLOROBENZENE					
01	24.6	24.7	17.6- 29.3	19- 27.8	Accept.

Report: RECCS
Page: 4
Date: 19NOV98

Performance Evaluation Report
USEPA Water Pollution Study WP040

Participant ID: PA00CCS Type: CTREE Requesting Office: NE

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
VOLATILE AROMATICS IN MICROGRAMS/LITER					
065-BENZENE					
01	29.1	25.7	21.9- 33.7	23.4- 32.2	Accept.
066-ETHYLBENZENE					
01	42.3	42.6	29.9- 54	32.9- 50.9	Accept.
067-TOLUENE					
01	33.9	32.3	23.9- 39.8	25.9- 37.8	Accept.
094-1,2-DICHLOROBENZENE					
01	33.4	36.3	27.1- 45.1	29.4- 42.8	Accept.
095-1,4-DICHLOROBENZENE					
01	41.1	41.6	30.4- 52.3	33.1- 49.6	Accept.
096-1,3-DICHLOROBENZENE					
01	30.1	33.7	23.7- 40.4	25.8- 38.3	Accept.
MISCELLANEOUS ANALYTES					
019-PH-UNITS					
01	8.54	8.60	8.31- 8.92	8.38- 8.84	Accept.
071-TOTAL CYANIDE(IN MG/L)					
01	0.122	0.140	0.089-0.184	0.101-0.172	Accept.
072-NON-FILTERABLE RESIDUE(IN MG/L)					
01	54.2	64.0	12.4- 80.7	20.9- 72.1	Accept.
073-OIL AND GREASE (FREON EXTRACTION)(IN MG/L)					
01	14.3	19.1	5.9- 27.5	8.78- 24.6	Accept.
097-TOTAL PHENOLICS(IN MG/L)					
01	.0405	0.0668	0.0261-0.108	0.0367-0.097	Accept.
098-TOTAL RESIDUAL CHLORINE(IN MG/L)					
01	1.06	0.930	0.811- 1.32	0.878- 1.25	Accept.
104-OIL AND GREASE (HEXANE EXTRACTION)(IN MG/L)					
01	18.1	19.1	5.65- 28.7	8.82- 25.6	Accept.

********** END OF DATA FOR PA00009 **********
NOTE: FOR LIMITS AND TRUE VALUES, ASSUME THREE SIGNIFICANT DIGITS.
********** END OF REPORT FOR PACCCC9 **********

* Based on gravimetric calculations, or a reference value when necessary.

13. Preventive Maintenance

In order to ensure timely production of data, Lancaster Laboratories schedules routine preventive maintenance of instruments based on manufacturer's recommendations. Maintenance of the laboratory instruments is the responsibility of the technical group using the equipment in conjunction with our in-house equipment maintenance group. A schedule of routinely performed instrument maintenance tasks is attached as Table 13-1. All preventive maintenance, as well as maintenance performed as corrective action, is recorded in instrument logs.

Critical spare parts are kept in supply at the laboratory by the equipment maintenance group. Most items not kept in stock at the laboratory are available through overnight delivery from the manufacturer. In addition, the Laboratory maintains multiple numbers of most of the critical instruments used in our laboratory operations. A recent equipment inventory may be found in the *Qualifications Manual.* Because we are a large laboratory with redundant capacity, the problems of instrument downtime are minimized.

Table 13-1

Preventive Maintenance Schedule

Instrument	Preventive Maintenance	Frequency
GC/MS	Change septum	Weekly or AN*
	Check fans	Monthly
	Check cool flow	Monthly
	Clean source	Bimonthly or AN
	Change oil in vacuum pump	Semiannually
	Change oil in turbo pump	Semiannually
GC Volatiles	Check propanol level	Semiweekly or AN
	Check all flows	Prior to calibration or AN
	Conductivity Det. Maintenance	AN
	Clean cell	AN
	Change reaction tube	AN
	Change Teflon line	AN
	Change resin	AN
	Replace trap	AN
	Column Maintenance	AN
	Change PID Lamp	AN
	Precalibration instrument settings Check	Prior to each calibration
HPLC	Pump lubrication	Annually
	Check pump seals	Annually
	Check valves cleaned or Rebuilt	AN
	Detector Maintenance	AN
	Bulb replacement and Adjustment	
	Flow cell cleaning	
	Routine column maintenance	AN
	Replace Teflon lines	AN
	Autosampler septa Replacement	AN
	In-line filter sonication/ Cleaning	AN
	System Pasivation	AN
	PCRS pump lubrication	AN

Table 13-1

Preventive Maintenance Schedule

Instrument	Preventive Maintenance	Frequency
Autoanalyzer	Clean sample probe	AN
	Clean proportioning pump	Weekly
	Inspect pump tubing, replace if worn	AN
	Clean wash receptacles,	Monthly
	Inspect condition of distillation head	Monthly
Spectrometer	Check absorbance	Monthly
	Check wavelength	Quarterly
Ion Chromatograph	Check guard column filters	Bimonthly
	Check bed supports	Bimonthly
	Check void space	Bimonthly
	Clean columns	Bimonthly
	Check anal. pump for leaks	Bimonthly
	Check DX-100 interior for leaks and spills	Bimonthly
	Oil sample pump and check Seals	Every 2 months
	Check air lines/tubing for crimping and/or Discoloration	Every 2 months
	Clean check valve	Every 3 months
	Check conductivity cell	AN

Table 13-1

Preventive Maintenance Schedule

Instrument	Preventive Maintenance	Frequency
Total Organic Carbon Analyzer	Check IR zero	AN
	Check for leaks	AN
	Check acid pump calibration	Bimonthly
	Check persulfate pump Calibration	Bimonthly
	Inspect 6-port rotary Valve	AN
	Inspect sample pump head	AN
	Wash molecular sieve	AN
	Check sample loop Calibration	Monthly
	Clean gas permeation tube	AN
	Inspect digestion vessel o-rings	AN
	Check activated carbon Scrubber	AN
	Dust back and clean circuit boards	AN
	Check IR cell	AN
Oxygen Meter	Check membrane	AN
pH Meter	Check level of buffer solution	Weekly
Cold Vapor AA	Change drying tube	Daily
	Replace pump tubing	AN: Min. weekly
	Lubricate pump head	Weekly
	Lubricate autosampler	Weekly
	Inspect optical cell and windows	Monthly
	Clean	AN
ICP	Clean torch	AN
	Clean nebulizer & spray chamber	AN
	Replace pump winding	Check daily
	Lubricate autosampler	Check daily
	Check mirror	Daily
	Checking tubing to torch	Daily
	Check fan filters, clean if needed	Weekly
	Check cool flow, clean if needed	Weekly
	Check water filter, replace if needed	Quarterly

*AN means as needed. Any of these items may be performed more frequently if response during operation indicates this is necessary.

14. Specific Routine Procedures Used to Assess Data Precision, Accuracy and Completeness

Precision - Precision refers to the reproducibility of a method when it is repeated on a second aliquot of the same sample. The degree of agreement is expressed as the Relative Percent Difference (RPD). The RPD will be calculated according to the following equation:

$$RPD = \frac{D_2 - D_1}{(D_1 + D_2) / 2} \times 100$$

Where:

D_1 = First sample value

D_2 = Second sample value (Duplicate)

Duplicates will be run on at least 5% of the samples. Acceptance criteria shall be based on statistical evaluation of past lab data. (See Section No. 11.) All Quality Control sample results are entered into the computer and compared with acceptance limits. In addition, there is a monthly review of values on the computer QC system. Data obtained from quality control samples is entered onto our computer system which charts the data, and calculates a mean and standard deviation on a monthly basis. The Quality Assurance Department then reviews this data for trends which may indicate analytical problems. The control charts are graphical methods for monitoring precision and bias over time.

Accuracy - Accuracy refers to the agreement between the amount of a compound measured by the test method and the amount actually present. Accuracy is usually expressed as a percent Recovery (R). Recoveries will be calculated according to the following equations:

$$\textit{Surrogate Recovery} = \frac{Qd}{Qa} \times 100$$

Where:

Qd = quantity determined by analysis

Qa = quantity added to sample

$$\textit{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked Sample Results

SR = Sample Results

SA = Spike added

$$\textit{Laboratory Control Sample Recovery} = \frac{LCS\ Found}{LCS\ True} \times 100$$

Surrogate standards are added to each sample analyzed for organics. Spikes and Laboratory Control Samples will be run on at least 5% of the samples (each batch or SDG, ≤20 samples). Refer to Section 11 for acceptance criteria for accuracy. The computer is programmed to compare the individual values with the acceptance limits and inform the analyst if the results meet specification. If the

results are not within the acceptance criteria, corrective action suitable to the situation will be taken. This may include, but is not limited to, checking calculations and instrument performance, reanalysis of the associated samples, examining other QC analyzed with the same batch of samples, and qualifying results with documentation of any QC problems in the Case Narrative.

Commercial quality control materials are run at least quarterly to ensure accuracy of the analytical procedure. Repetitive analysis of a reference material will also yield precision data. Accuracy information determined from reference materials is valuable because variables specific to sample matrix are eliminated.

The QC program is capable of charting data for surrogates, spikes, control materials and reference materials. The Quality Assurance Department reviews these charts for any indication of possible problems (i.e., shift in the mean and standard deviation).

Completeness - Completeness is the percentage of valid data acquired from a measurement system compared to the amount of valid measurements that were planned to be collected. The objective is analysis of all samples submitted intact, and to ensure that sufficient sample weight/volume is available should the initial analysis not meet acceptance criteria. The laboratory's Sample Management System will assign a unique identification number to the sample which tracks and controls movement of samples from the time of receipt until disposal. All data generated will be recorded referencing the corresponding sample identification number. The completeness of an analysis can be documented by including in the data deliverables sufficient information to allow the data user to assess the quality of the results. This information will include, but is not limited to, summaries of QC

data and sample results, chromatograms, spectra, and instrument tune and calibration data. Additional information will be stored in the laboratory's archives, both hard copy and magnetic tape.

$$Completeness = \frac{Number\ of\ valid\ measurements}{Total\ measurements\ needed} \times 100$$

Method Detection Limit (MDL) - It is important to ascertain the limit of quantitation (LOQ) that can be achieved by a given method, particularly when the method is commonly used to determine trace levels of analyte. The Environmental Protection Agency has set forth one method for determining MDLs from which LOQs can be extrapolated.

MDL is defined as follows for all measurements:

$$MDL = t_{(n-1,1-\alpha=0.99)} \times s$$

Where:

MDL = method detection limit

s = standard deviation of the replicate analyses

$t_{(n-1,1-\alpha=0.99)}$ = students' t-value for a one-sided 99% confidence level and a standard deviation estimate with n-1 degrees of freedom

Definitions:

Method Detection Limit (MDL): The method detection limit is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. It is determined from analysis of a sample in a given matrix containing the analyte.

Limit of Quantitation (LOQ): The limit of quantitation is defined as the level above which quantitative results may be obtained with a specified degree of confidence. The EPA recommends setting quantitation limits at a value of five-to-ten times the MDL.

A list of MDLs and LOQs determined for each sample matrix type will be kept on file in the QA department. MDLs will be verified on an annual basis.

15. Corrective Action

Whenever any of the data generated falls outside of the established acceptance criteria outlined for instrument tune and calibration (Section 8) and Internal QC (Section 11), the cause of this irregularity must be investigated, corrected, and documented. The documentation will be used to prevent a recurrence of the problem and to inform management of the situation.

If the results are not within acceptance criteria, the appropriate corrective action will be initiated. This may include, but is not limited to, checking calculation and instrument performance, reanalysis of the associated samples, examining other QC analyzed with the same batch of samples, and qualifying results with a comment stating the observed deviation.

A Standard Operating Procedure is in place which outlines the procedures to be followed when quality control data for an analysis falls outside of previously established acceptance limits. All QC data must be entered onto the computerized QC system promptly after its generation and daily "out-of-spec" data is reported via this system. Any data outside the acceptance criteria will be reviewed by the Quality Assurance Department. Where appropriate, the Quality Assurance Department will place outliers in one of three categories:

A. Marginal Outlier

   Data that are outside the 95% confidence interval but within the 99% confidence interval. This category may also be used for QC samples subject to matrix interferences or sample inhomogeneity.

B. Outlier

   Data outside the 99% confidence interval and/or observable trends such as a shift in mean and standard deviation.

C. Extreme Outlier

Such data would indicate the system is out of control and no results should be reported to clients; an example would be more than one reference or control falling outside the 99% confidence interval.

The daily out-of-spec reports are then distributed to Group Leaders or their QC Coordinator who will check all supporting data and document their findings and any corrective action taken. Documentation of QC Data will be filed in the departmental QC notebook. In the case of Outliers or Extreme Outliers the Quality Assurance Department may issue a formal request for investigation and corrective action (see sample form that follows). The Quality Assurance Department is responsible for initiating the corrective actions, insuring that the actions are taken in a timely manner, and that the desired results are produced. The QA Department will circulate all completed Investigation & Corrective Action forms to the appropriate manager.

The Quality Assurance Department is also responsible for conducting periodic audits which ensure compliance with laboratory SOPs and assist in identifying and correcting any deficiencies. These audits may entail observation as procedures are carried out or a review of records to demonstrate traceability and compliance with all documented record keeping procedures. The QA Department will then issue a written report which summarizes the audit. The technical centers must respond in writing to the audit report within 30 days of report receipt. The response will address the corrective action that needs to be taken along with an expected completion date. Audit results and the corresponding response are communicated to laboratory personnel and management. Follow-up audits verify that proper corrective action has been taken for the identified discrepancy.

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601-5994

No. _____

Investigation and Corrective Action Report (ICAR)

Part I - Description of the Problem (Attach additional page, if needed, in addition to supporting documentation.)

1. Date of issue:
2. LL sample number(s) involved:
3. Nature of the problem (describe in detail):

Initiated by: _____

Part II - The Investigation (Attach additional page, if needed, in addition to supporting documentation.)

1. Steps taken to investigate the problem:
2. Explanation of probable cause(s):
3. Steps taken to prevent future occurrence:
4. Must investigation be complete before reporting further data to clients? Yes No
5. In addition to the samples listed above, would any additional data already reported to clients be affected by this problem? Yes No If yes, please explain

Investigator(s): _____ Date: _____

Investigator(s): _____ Date: _____

Supervisor(s): _____ Date: _____

Supervisor(s): _____ Date: _____

Quality Assurance: _____ Date: _____

Return to QA by: _____

16. Quality Assurance Reports to Management

Reports of quality status from the Quality Assurance Department to management are made frequently and in various forms. All results from internal or external performance evaluation samples are circulated to management. A report of each audit performed is prepared and copied to management. Monthly summaries of data obtained from analysis of quality control check samples are generated via the computerized sample management system. These summaries include mean and standard deviation to aid in assessment of data accuracy and precision. Forms summarizing problems which require investigation and corrective action are completed by Group Leaders and circulated to management. Through these channels, laboratory management is kept apprised of QA/QC activities.

Any problems or unusual observations that occur during the analysis of samples for a specific project will be listed on the laboratory report and/or in the case narrative delivered with the data package. The items often discussed in this manner include samples with surrogate recovery outside of the acceptance criteria and samples with matrix problems requiring dilution and causing increased detection limits. Where applicable, any corrective action attempted or performed to address the problem will also be presented.

The laboratory will contact the client for direction regarding major problems such as samples listed on the chain of custody but missing from the shipping container, samples which arrive broken or are accidentally broken in the laboratory, and samples with severe matrix problems. The client will be contacted if it is necessary to change any item in the original project plan.

Appendix A

Example Reporting Forms

Data Package Content

- Title Page
- Sample Reference
- Table of Contents
- Chain of Custody
- Laboratory Chronicle
- Methodology/Reference Summary
- Laboratory Analysis Reports
- Per Parameter:
  - Case Narrative
  - Quality Control Summary
    - Tune[1]
    - Surrogate Recovery
    - Method Blank
    - Matrix Spike/Matrix Spike Duplicate
    - Duplicate[2]
    - Standard Addition[2]
    - Serial Dilution[2]
    - Laboratory Control Sample Recovery (if applicable)
    - Interference Check[2]
    - Internal Standard[1]
  - Sample Data
    - Sample Result Summary and LOQs
    - Sample Chromatograms
    - Quantitation Reports
    - Mass Spectra[1]
    - Library Searches[1] (if applicable)
    - Confirmatory Chromatogram[3]
    - Confirmatory Quantitation Report[3]

Standards Data Package

- Initial Calibration Summary Forms
- Initial Calibration Data
- Continuing Calibration Summary Forms
- Continuing Calibration Data
- Chromatograms and Quantitation Reports of Standards
- Calibration Data for Confirmation Columns[3]
- Calibration Curve (When quantitating against init. calib.)
- ICAP Interference Table[2]

Raw QC Data

- BFB/DFTPP Spectra and Mass Listing[1]
- Method Blank Chromatograms, Quantitation Reports,
  Mass Spectra[1] (GC/MS)
- Matrix Spike/Matrix Spike Duplicate Chromatograms and Quant.
- Duplicate Data Printouts[2]
- Standard Addition Data[2]
- Serial Dilution Data[2]
- Laboratory Control Sample (if applicable)
- Copy of Instrument Run Log

Extraction/Digestion Logs

Gel Permeation Chromatography (GPC), if applicable

- All Peaks Identified
- % Resolution Calculations

[1] GC/MS only
[2] Inorganics only
[3] GC only (if applicable)

* Amount of documentation is dependent upon client request.

Lancaster Laboratories
Where quality is a science.

LLI Sample No. WW 2300873
Collected:

Submitted: 4/25/95 Reported: 6/14/95
Discard: 6/22/95

Volatile Halocarbons - 1
EPA WP 034

Account No: 00649
Lancaster Laboratories, Inc.
2425 New Holland Pike
Lancaster, PA 17601-5994

P.O.
Rel.

CAT NO.	ANALYSIS NAME	AS RECEIVED RESULTS	LIMIT OF QUANTITATION	UNITS
Purgeable Halocarbons				
0711	Chloromethane	< 5.	5.	ug/l
0712	Bromomethane	< 5.	5.	ug/l
1590	Dichlorodifluoromethane	< 2.	2.	ug/l
0714	Vinyl chloride	< 1.	1.	ug/l
0715	Chloroethane	< 1.	1.	ug/l
0716	Methylene chloride	54.	1.	ug/l
1589	Trichlorofluoromethane	< 1.	1.	ug/l
0717	1,1-Dichloroethene	< 1.	1.	ug/l
0718	1,1-Dichloroethane	< 1.	1.	ug/l
0719	1,2-Dichloroethene (cis/trans)	< 1.	1.	ug/l
0720	Chloroform	57.	1.	ug/l
0721	1,2-Dichloroethane	64.	1.	ug/l
0722	1,1,1-Trichloroethane	50.	1.	ug/l
0723	Carbon tetrachloride	66.	1.	ug/l
0724	Dichlorobromomethane	62.	1.	ug/l
0725	1,2-Dichloropropane	< 1.	1.	ug/l
0726	trans-1,3-Dichloropropene	< 1.	1.	ug/l
0727	Trichloroethene	54.	1.	ug/l
0728	Dibromochloromethane	49.	1.	ug/l
0729	1,1,2-Trichloroethane	< 1.	1.	ug/l
0730	cis-1,3-Dichloropropene	< 1.	1.	ug/l
0713	2-Chloroethylvinyl ether	< 10.	10.	ug/l
0731	Bromoform	50.	2.	ug/l
0732	1,1,2,2-Tetrachloroethane	< 2.	2.	ug/l
0733	Tetrachloroethene	55.	1.	ug/l
0705	Chlorobenzene	54.	1.	ug/l

Under the analytical conditions of EPA methods 601 and 8010B, the cis and trans isomers of 1,2-dichloroethene coelute and cannot be distinguished from one another. The result reported above represents the total for both isomers.

Questions? Contact your Client Services Representative
Kimberly A. Zeeman at (717) 656-2300

Respectfully Submitted
Judy A. Colello, B.S.
Group Leader

MEMBER
Lancaster Laboratories, Inc.
2425 New Holland Pike
PO Box 12425
Lancaster, PA 17605-2425
717-656-2300 Fax 717-656-2681

See reverse side for explanation of symbols and abbreviations.

Lancaster Laboratories
Where quality is a science.

LLI Sample No. WW 2300869
Collected:

Submitted: 4/25/95 Reported: 6/14/95
Discard: 6/22/95

Pesticides - 1
EPA WP 034

Account No: 00649
Lancaster Laboratories, Inc.
2425 New Holland Pike
Lancaster, PA 17601-5994

P.O.
Rel.

CAT NO.	ANALYSIS NAME	AS RECEIVED RESULTS	LIMIT OF QUANTITATION	UNITS
PPL Pesticides in Water				
1600	Alpha BHC	< 0.1	0.1	ug/l
1601	Beta BHC	< 0.1	0.1	ug/l
1602	Gamma BHC - Lindane	< 0.1	0.1	ug/l
1603	Delta BHC	< 0.1	0.1	ug/l
1604	Heptachlor	2.0	0.1	ug/l
1605	Aldrin	1.5	0.1	ug/l
1606	Heptachlor Epoxide	1.9	0.1	ug/l
1607	DDE	2.6	0.1	ug/l
1608	DDD	3.9	0.1	ug/l
1609	DDT	2.9	0.1	ug/l
1610	Dieldrin	4.7	0.1	ug/l
1611	Endrin	< 0.1	0.1	ug/l
1860	Methoxychlor	< 0.5	0.5	ug/l
1612	Chlordane	< 3.	3.	ug/l
1613	Toxaphene	< 40.	40.	ug/l
1616	Endosulfan I	< 0.1	0.1	ug/l
1615	Endosulfan II	< 0.1	0.1	ug/l
1617	Endosulfan Sulfate	< 0.3	0.3	ug/l
1618	Endrin Aldehyde	< 1.	1.	ug/l
1619	PCB-1016	< 10.	10.	ug/l
1620	PCB-1221	< 10.	10.	ug/l
1621	PCB-1232	< 10.	10.	ug/l
1622	PCB-1242	< 10.	10.	ug/l
1623	PCB-1248	< 10.	10.	ug/l
1624	PCB-1254	< 10.	10.	ug/l
1626	PCB-1260	< 10.	10.	ug/l

Questions? Contact your Client Services Representative
Kimberly A. Zeeman at (717) 656-2300

Respectfully Submitted
Jenifer E. Hess, B.S.
Group Leader Pesticides/PCBs

MEMBER ACIL
Lancaster Laboratories, Inc
2425 New Holland Pike
PO Box 12425
Lancaster, PA 17605-2425
717-656-2301 FAX 717-656-2681

See reverse side for explanation of symbols and abbreviations.

Lancaster Laboratories

Where quality is a science.

LLI Sample No. WW 2300851
Collected:

Submitted: 4/25/95 Reported: 6/14/95
Discard: 6/22/95

Trace Metals - 1
EPA WP 034

Account No: 00649
Lancaster Laboratories, Inc.
2425 New Holland Pike
Lancaster, PA 17601-5994

P.O.
Rel.

CAT NO.	ANALYSIS NAME	AS RECEIVED RESULTS	LIMIT OF QUANTITATION	UNITS
1743	Aluminum	0.96	0.20	mg/l
1747	Beryllium	0.012	0.010	mg/l
1749	Cadmium	0.013	0.010	mg/l
1751	Chromium	0.095	0.030	mg/l
1752	Cobalt	0.129	0.050	mg/l
1753	Copper	0.049	0.025	mg/l
1754	Iron	0.65	0.10	mg/l
1755	Lead	0.19	0.10	mg/l
1758	Manganese	0.292	0.010	mg/l
1761	Nickel	0.083	0.050	mg/l
1771	Vanadium	5.21	0.015	mg/l
1772	Zinc	0.446	0.020	mg/l
0243	Aluminum	1.02	0.20	mg/l
0245	Arsenic	0.118	0.0020	mg/l
0247	Beryllium	0.013	0.010	mg/l
0249	Cadmium	0.014	0.010	mg/l
0251	Chromium	0.090	0.030	mg/l
0253	Copper	0.050	0.025	mg/l
0254	Iron	0.59	0.10	mg/l
0255	Lead	0.191	0.050	mg/l
0258	Manganese	0.267	0.010	mg/l
0259	Mercury	0.00143	0.00020	mg/l
0261	Nickel	0.081	0.050	mg/l
0264	Selenium	0.194	0.0020	mg/l
0272	Zinc	0.454	0.025	mg/l

1 COPY TO Susan Shorter

Questions? Contact your Client Services Representative
Kimberly A. Zeeman at (717) 656-2300
03:31:03 D 0001 36 0 0 463245
044 15.00 00040800 ASR000

Respectfully Submitted
Ramona V. Layman, Group Leader
ICP Metals/Leachates

MEMBER ACIL
Lancaster Laboratories Inc
2425 New Holland Pike
PO Box 17425
Lancaster PA 17605-7425
717-656-2301 Fax 717-656-2681

See reverse side for explanation of symbols and abbreviations.

Lancaster Laboratories
Where quality is a science.

LLI Sample No. WW 2300863
Collected:

Submitted: 4/25/95 Reported: 6/14/95
Discard: 6/22/95

Demand - 1
EPA WP 034

Account No: 00649
Lancaster Laboratories, Inc.
2425 New Holland Pike
Lancaster, PA 17601-5994

P.O.
Rel.

CAT NO.	ANALYSIS NAME	AS RECEIVED RESULTS	LIMIT OF QUANTITATION	UNITS
0273	Total Organic Carbon	19.	1.	mg/l
	The Total Organic Carbon (TOC) result reported above was determined by measuring total carbon by a persulfate digestion/infrared detection method on an acidified sample which has been purged of inorganic carbon using nitrogen. It represents "non-purgeable TOC".			
0235	Biochemical Oxygen Demand	29.	2.	mg/l
1364	Carbonaceous BOD	31.	2.	mg/l
1553	Chemical Oxygen Demand	45.	7.	mg/l

1 COPY TO Susan Shorter

Questions? Contact your Client Services Representative
Kimberly A. Zeeman at (717) 656-2300
03:32:57 D 0001 36 0 0 463245
044 0.00 00012600 ASR000

Respectfully Submitted
Ramona V. Layman, Group Leader
ICP Metals/Leachates

MEMBER ACIL
Lancaster Laboratories, Inc
2425 New Holland Pike
PO Box 12425
Lancaster, PA 17605-2425
717-656-2301 Fax: 717-656-2681

5B

SEMIVOLATILE ORGANIC INSTRUMENT PERFORMANCE CHECK
DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____. SAS No.: _____. SDG No.: _____.

Lab File ID: >Z730Z DFTPP Injection Date: 07/05/95

Instrument ID: HP02550 DFTPP Injection Time: 15:40

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
51	30.0 - 80.0% of mass 198	58.0
68	Less than 2.0% of mass 69	0.0 (0.0)1
69	Mass 69 relative abundance	66.2
70	Less than 2.0% of mass 69	0.0 (0.0)1
127	25.0 - 75.0% of mass 198	44.0
197	Less than 1.0% of mass 198	0.0
198	Base Peak, 100% relative abundance	100.
199	5.0 to 9.0% of mass 198	6.6
275	10.0 - 30.0% of mass 198	19.9
365	Greater than 0.75% of mass 198	1.87
441	Present, but less than mass 443	8.8
442	40.0 - 110.0% of mass 198	59.9
443	15.0 - 24.0% of mass 442	11.4 (19.0)2

1-Value is % mass 69 2-Value is % mass 442

THIS TUNE APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01	SSTD50	CLP1815	>Z7301	07/05/95	16:08
02	0303BMSD	2337099	>C7301	07/05/95	18:14
03	0400B	2337092	>C7302	07/05/95	19:07
04	0303BMS	2337098	>C7304	07/05/95	21:44
05	0405B	2337094	>C7305	07/05/95	22:45
06					
07					
08					
09					
10					
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13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

2C

WATER SEMIVOLATILE SURROGATE RECOVERY

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____. SAS No.: _____. SDG No.: _____.

	EPA SAMPLE NO.	S1 (NBZ)#	S2 (FBP)#	S3 (TPH)#	S4 (PHL)#	S5 (2FP)#	S6 (TBP)#	OTHER	TOT OUT
01	SBLKWA1714	75	78	77	38	54	89		0
02	171WALCS	86	85	88	43	61	108		0
03	171WALCSD	91	85	92	42	60	103		0
04	171WAUS	88	86	73	43	62	103		0
05	171WAMS	93	88	92	44	64	106		0
06	171WAMSD	85	83	94	41	58	106		0
07	SEDFB	81	83	80	37	56	81		0
08	SDFB2	79	81	76	37	58	78		0
09	NVBRM	59	80	70	39	59	90		0
10	2HP6-	80	85	74	38	59	77		0
11									
12									
13									
14									
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29									
30									

	QC LIMITS
S1 (NBZ) = Nitrobenzene-d5	(35-114)
S2 (FBP) = 2-Fluorobiphenyl	(43-116)
S3 (TPH) = Terphenyl-d14	(33-141)
S4 (PHL) = Phenol-d6	(10-94)
S5 (2FP) = 2-Fluorophenol	(21-100)
S6 (TBP) = 2,4,6-Tribromophenol	(10-123)

Column to be used to flag recovery values
* Values outside of contract required QC limits
D Surrogates diluted out

SOIL SEMIVOLATILE SURROGATE RECOVERY

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____. SAS No.: _____. SDG No.: _____.

Level: (low/med) LOW

	EPA SAMPLE NO.	S1 (NBZ) #	S2 (FBP) #	S3 (TPH) #	S4 (PHL) #	S5 (2FP) #	S6 (TBP) #	OTHER	TOT OUT
01	SBLKLA1561	89	73	75	42	28	92		0
02	156LALCS	83	92	124	77	77	76		0
03	EDGE1	53	52	75	56	57	55		0
04	EDGE1MS	79	83	108	77	77	74		0
05	EDGE1MSD	62	67	97	60	63	61		0
06									
07									
08									
09									
10									
11									
12									
13									
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28									
29									
30									

	QC LIMITS
S1 (NBZ) = Nitrobenzene-d5	(23-120)
S2 (FBP) = 2-Fluorobiphenyl	(30-115)
S3 (TPH) = Terphenyl-d14	(18-137)
S4 (PHL) = Phenol-d6	(24-113)
S5 (2FP) = 2-Fluorophenol	(25-121)
S6 (TBP) = 2,4,6-Tribromophenol	(19-122)

Column to be used to flag recovery values
* Values outside of contract required QC limits
D Surrogates diluted out

4B

SEMIVOLATILE METHOD BLANK SUMMARY

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____. SAS No.: _____. SDG No.: _____.

Lab File ID: >D3750 Lab Sample ID:SBLKWA171

Date Extracted: 06/20/95 Extraction:(SepF/Cont/Sonc) SEPF

Date Analyzed: 06/21/95 Time Analyzed: 13:00

Matrix: (soil/water) WATER Level:(low/med) LOW

Instrument ID: HP03301

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED
01	171WALCS	171WALCS	>D3751	06/21/95
02	171WALCSD	171WALCSD	>D3752	06/21/95
03	171WAUS	171WAUS	>D3753	06/21/95
04	171WAMS	171WAMS	>D3754	06/21/95
05	171WAMSD	171WAMSD	>D3755	06/21/95
06	SEDFB	2329395	>D3801	06/22/95
07	SDFB2	2330033	>D3802	06/22/95
08	NVBRM	2325395	>D3803	06/22/95
09	2HP6-	2330176	>D3804	06/22/95

COMMENTS: _____

1B

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

SBLKWA1714

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____. SAS No.: _____. SDG No.: _____.

Matrix: (soil/water) WATER Lab Sample ID: SBLKWA171

Sample wt/vol: 1000 (g/mL) ML Lab File ID: >D3750

Level: (low/med) LOW Date Received:

% Moisture: not dec. _____. dec. _____. Date Extracted: 06/20/95

Extraction: (SepF/Cont/Sonc) SEPF Date Analyzed: 06/21/95

GPC Cleanup: (Y/N) N pH: _____. Dilution Factor: 1.0

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) UG/L	Q
110-86-1	Pyridine	10	U
62-75-9	N-Nitrosodimethylamine	10	U
108-95-2	Phenol	10	U
111-44-4	bis(2-Chloroethyl)ether	10	U
95-57-8	2-Chlorophenol	10	U
541-73-1	1,3-Dichlorobenzene	10	U
106-46-7	1,4-Dichlorobenzene	10	U
95-50-1	1,2-Dichlorobenzene	10	U
95-48-7	2-Methylphenol	10	U
108-60-1	bis(2-Chloroisopropyl)ether	10	U
65794-96-9	3 or 4-Methylphenol	10	U
621-64-7	N-Nitroso-di-n-propylamine	10	U
67-72-1	Hexachloroethane	10	U
98-95-3	Nitrobenzene	10	U
78-59-1	Isophorone	10	U
88-75-5	2-Nitrophenol	10	U
105-67-9	2,4-Dimethylphenol	10	U
111-91-1	bis(2-Chloroethoxy)methane	10	U
120-83-2	2,4-Dichlorophenol	10	U
120-82-1	1,2,4-Trichlorobenzene	10	U
91-20-3	Naphthalene	10	U
87-68-3	Hexachlorobutadiene	10	U
59-50-7	4-Chloro-3-methylphenol	10	U
77-47-4	Hexachlorocyclopentadiene	10	U
88-06-2	2,4,6-Trichlorophenol	10	U
95-95-4	2,4,5-Trichlorophenol	10	U
91-58-7	2-Chloronaphthalene	10	U
131-11-3	Dimethylphthalate	10	U
606-20-2	2,6-Dinitrotoluene	10	U
208-96-8	Acenaphthylene	10	U
83-32-9	Acenaphthene	10	U
51-28-5	2,4-Dinitrophenol	25	U
100-02-7	4-Nitrophenol	25	U

1C

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO

SBLKWA1714

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____. SAS No.: _____. SDG No.: _____.

Matrix: (soil/water) WATER Lab Sample ID: SBLKWA171

Sample wt/vol: 1000 (g/mL) ML Lab File ID: >D3750

Level: (low/med) LOW Date Received:

% Moisture: not dec. _____. dec. _____. Date Extracted: 06/20/95

Extraction: (SepF/Cont/Sonc) SEPF Date Analyzed: 06/21/95

GPC Cleanup: (Y/N) N pH: _____. Dilution Factor: 1.0

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) UG/L	Q
121-14-2	2,4-Dinitrotoluene	10	U
84-66-2	Diethylphthalate	10	U
7005-72-3	4-Chlorophenyl-phenylether	10	U
86-73-7	Fluorene	10	U
534-52-1	4,6-Dinitro-2-methylphenol	25	U
86-30-6	N-Nitrosodiphenylamine (1)	10	U
122-66-7	1,2-Diphenylhydrazine	10	U
101-55-3	4-Bromophenyl-phenylether	10	U
118-74-1	Hexachlorobenzene	10	U
87-86-5	Pentachlorophenol	25	U
85-01-8	Phenanthrene	10	U
120-12-7	Anthracene	10	U
84-74-2	Di-n-butylphthalate	10	U
206-44-0	Fluoranthene	10	U
92-87-5	Benzidine	50	U
129-00-0	Pyrene	10	U
85-68-7	Butylbenzylphthalate	10	U
91-94-1	3,3'-Dichlorobenzidine	20	U
56-55-3	Benzo(a)anthracene	10	U
117-81-7	bis(2-Ethylhexyl)phthalate	10	U
218-01-9	Chrysene	10	U
117-84-0	Di-n-octylphthalate	10	U
205-99-2	Benzo(b)fluoranthene	10	U
207-08-9	Benzo(k)fluoranthene	10	U
50-32-8	Benzo(a)pyrene	10	U
193-39-5	Indeno(1,2,3-cd)pyrene	10	U
53-70-3	Dibenz(a,h)anthracene	10	U
191-24-2	Benzo(g,h,i)perylene	10	U

(1) - Cannot be separated from Diphenylamine

WATER SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLE RECOVERY

Lab Name: LANCASTER LABS Lab Code: LANCAS Instrument: HP03301

SW846 METHOD 8270 SPIKE LEVEL: 100 UG/ML AMT USED: 1000.

SAMPLE SPIKE LEVEL: 100.UG/L % MOISTURE 0. DILUTION: 1

US SAMPLE: 171WAUS 171WAUS MS SAMPLE: 171WAMS 171WAMS MSD SAMPLE: 171WAMSD 171WAMSD

COMPOUND NAME	US CONC UG/L	MS CONC UG/L	MSD CONC UG/L	MS REC %	MSD REC %	RPD %	RANGE LOWER-UPPER	IN SPEC
N-Nitrosodimethylamine	0.00	71.19	63.55	71	64	11.00	35.0-100.8	YES
Phenol	0.00	50.36	47.69	50	48	5.00	5.0-112.0	YES
bis(2-Chloroethyl)ether	0.00	91.95	85.37	92	85	7.00	12.0-158.0	YES
2-Chlorophenol	0.00	93.27	87.54	93	88	6.00	23.0-134.0	YES
1,3-Dichlorobenzene	0.00	87.92	79.61	88	80	10.00	1.0-172.0	YES
1,4-Dichlorobenzene	0.00	89.59	81.76	90	82	9.00	20.0-124.0	YES
1,2-Dichlorobenzene	0.00	92.62	84.48	93	84	9.00	32.0-129.0	YES
bis(2-Chloroisopropyl)ether	0.00	98.40	92.17	98	92	7.00	36.0-166.0	YES
N-Nitroso-di-n-propylamine	0.00	110.79	104.31	111	104	6.00	1.0-230.0	YES
Hexachloroethane	0.00	80.42	73.16	80	73	9.00	40.0-113.0	YES
Nitrobenzene	0.00	100.44	93.63	100	94	7.00	35.0-180.0	YES
Isophorone	0.00	91.13	86.68	91	87	5.00	21.0-196.0	YES
2-Nitrophenol	0.00	97.51	94.45	98	94	3.00	29.0-182.0	YES
2,4-Dimethylphenol	0.00	84.53	77.29	84	77	9.00	32.0-119.0	YES
bis(2-Chloroethoxy)methane	0.00	89.50	84.21	89	84	6.00	33.0-184.0	YES
2,4-Dichlorophenol	0.00	95.88	91.26	96	91	5.00	39.0-135.0	YES
1,2,4-Trichlorobenzene	0.00	89.02	82.33	89	82	8.00	44.0-142.0	YES
Naphthalene	0.00	90.10	83.34	90	83	8.00	21.0-133.0	YES
Hexachlorobutadiene	0.00	82.27	73.61	82	74	11.00	24.0-116.0	YES
4-Chloro-3-methylphenol	0.00	97.77	95.61	98	96	2.00	22.0-147.0	YES
Hexachlorocyclopentadiene	0.00	138.52	88.83	69	44	44.00	1.0-100.0	YES
2,4,6-Trichlorophenol	0.00	97.75	92.93	98	93	5.00	37.0-144.0	YES
2-Chloronaphthalene	0.00	89.52	85.35	90	85	5.00	60.0-118.0	YES
Dimethylphthalate	0.00	90.86	87.84	91	88	3.00	1.0-112.0	YES
2,6-Dinitrotoluene	0.00	86.36	84.61	86	85	2.00	50.0-158.0	YES
Acenaphthylene	0.00	90.55	85.28	90	85	6.00	33.0-145.0	YES
Acenaphthene	0.00	89.05	85.24	89	85	4.00	47.0-145.0	YES
2,4-Dinitrophenol	0.00	94.45	92.15	94	92	2.00	1.0-191.0	YES
4-Nitrophenol	0.00	47.71	46.64	48	47	2.00	1.0-132.0	YES
2,4-Dinitrotoluene	0.00	103.67	102.08	104	102	2.00	39.0-139.0	YES
1-Naphthylamine	0.00	41.80	36.84	42	37	13.00	1.0-100.0	YES
2-Naphthylamine	0.00	55.40	44.65	55	45	22.00	1.0-100.0	YES
Diethylphthalate	0.00	95.85	93.03	96	93	3.00	1.0-114.0	YES
4-Chlorophenyl-phenylether	0.00	92.22	88.60	92	88	4.00	25.0-158.0	YES
Fluorene	0.00	90.96	87.93	91	88	3.00	59.0-121.0	YES
4,6-Dinitro-2-methylphenol	0.00	88.46	86.50	88	86	2.00	1.0-181.0	YES
N-Nitrosodiphenylamine	0.00	86.53	83.21	86	83	4.00	37.8-147.0	YES
1,2-Diphenylhydrazine	0.00	86.10	82.37	86	82	4.00	25.7-124.9	YES
4-Bromophenyl-phenylether	0.00	92.83	88.97	93	89	4.00	53.0-127.0	YES

WATER SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLE RECOVERY

Lab Name: LANCASTER LABS Lab Code: LANCAS Instrument: HP03301

SW846 METHOD 8270 SPIKE LEVEL: 100 UG/ML AMT USED: 1000.

SAMPLE SPIKE LEVEL: 100.UG/L % MOISTURE 0. DILUTION: 1

US SAMPLE: 171WAUS 171WAUS MS SAMPLE: 171WAMS 171WAMS MSD SAMPLE: 171WAMSD 171WAMSD

COMPOUND NAME	US CONC UG/L	MS CONC UG/L	MSD CONC UG/L	MS REC %	MSD REC %	RPD %	RANGE LOWER-UPPER	IN SPEC
Hexachlorobenzene	0.00	93.90	90.89	94	91	3.00	1.0-152.0	YES
Pentachlorophenol	0.00	71.04	78.11	71	78	-9.00	14.0-176.0	YES
Phenanthrene	0.00	89.59	85.11	90	85	5.00	54.0-120.0	YES
Anthracene	0.00	88.13	84.06	88	84	5.00	27.0-133.0	YES
Di-n-butylphthalate	0.00	97.28	90.78	97	91	7.00	1.0-118.0	YES
Fluoranthene	0.00	97.82	92.76	98	93	5.00	26.0-137.0	YES
Benzidine	0.00	409.22	309.00	82	62	28.00	1.0-155.0	YES
Pyrene	0.00	86.40	89.91	86	90	-4.00	52.0-115.0	YES
Butylbenzylphthalate	0.00	94.76	93.22	95	93	2.00	1.0-152.0	YES
3,3'-Dichlorobenzidine	0.00	95.40	87.15	95	87	9.00	1.0-262.0	YES
Benzo(a)anthracene	0.00	89.52	86.79	90	87	3.00	33.0-143.0	YES
bis(2-Ethylhexyl)phthalate	0.00	94.36	92.14	94	92	2.00	8.0-158.0	YES
Chrysene	0.00	91.49	90.70	91	91	1.00	17.0-168.0	YES
Di-n-octylphthalate	0.00	90.04	93.87	90	94	-4.00	4.0-146.0	YES
Benzo(b)fluoranthene	0.00	88.77	89.34	89	89	-1.00	24.0-159.0	YES
Benzo(k)fluoranthene	0.00	90.41	89.11	90	89	1.00	11.0-163.0	YES
Benzo(a)pyrene	0.00	89.68	86.37	90	86	4.00	17.0-163.0	YES
Indeno(1,2,3-cd)pyrene	0.00	88.00	81.41	88	81	8.00	1.0-171.0	YES
Dibenz(a,h)anthracene	0.00	86.73	81.84	87	82	6.00	1.0-227.0	YES
Benzo(g,h,i)perylene	0.00	86.02	79.43	86	79	8.00	1.0-219.0	YES

COMMENTS:

SOIL SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLE RECOVERY

Lab Name: LANCASTER LABS Lab Code: LANCAS Instrument: HP01597

SW846 METHOD 8270 SPIKE LEVEL: 100 UG/ML AMT USED: 30.0

SAMPLE SPIKE LEVEL: 4115.UG/KG % MOISTURE 19. DILUTION: 1

US SAMPLE: EDGE1 2321686 MS SAMPLE: EDGE1MS 2321686 MSD SAMPLE: EDGE1MSD 2321686

COMPOUND NAME	US CONC UG/KG	MS CONC UG/KG	MSD CONC UG/KG	MS REC %	MSD REC %	RPD %	RANGE LOWER-UPPER	IN SPEC
Pyridine	0.00	671.29	621.86	16	15	7.65	28.1-100.0	NO
N-Nitrosodimethylamine	0.00	2989.88	2010.07	73	49	39.19	35.0-100.8	YES
Phenol	0.00	3029.55	2407.92	74	58	22.86	5.0-112.0	YES
bis(2-Chloroethyl)ether	0.00	3308.06	2544.26	80	62	26.10	12.0-158.0	YES
2-Chlorophenol	0.00	3372.29	2674.63	82	65	23.08	23.0-134.0	YES
1,3-Dichlorobenzene	0.00	3320.26	2291.33	81	56	36.67	1.0-172.0	YES
1,4-Dichlorobenzene	0.00	3315.43	2345.48	80	57	34.27	20.0-124.0	YES
1,2-Dichlorobenzene	0.00	3458.49	2539.93	84	62	30.63	32.0-129.0	YES
2-Methylphenol	0.00	3903.10	2783.85	95	68	33.48	45.9-122.5	YES
bis(2-Chloroisopropyl)ether	0.00	3010.85	2306.81	73	56	26.48	36.0-166.0	YES
3 or 4-Methylphenol	0.00	4177.06	3214.33	102	78	26.05	53.6-175.2	YES
N-Nitroso-di-n-propylamine	0.00	3475.73	2766.08	84	67	22.74	1.0-230.0	YES
Hexachloroethane	0.00	3386.81	2233.34	82	54	41.05	40.0-113.0	YES
Nitrobenzene	0.00	3170.46	2525.16	77	61	22.66	35.0-180.0	YES
Isophorone	0.00	3161.95	2549.84	77	62	21.43	21.0-196.0	YES
2-Nitrophenol	0.00	3868.27	3047.33	94	74	23.74	29.0-182.0	YES
2,4-Dimethylphenol	0.00	3131.66	2495.26	76	61	22.62	32.0-119.0	YES
bis(2-Chloroethoxy)methane	0.00	3175.26	2528.70	77	61	22.67	33.0-184.0	YES
2,4-Dichlorophenol	0.00	3615.82	2946.73	88	72	20.39	39.0-135.0	YES
1,2,4-Trichlorobenzene	0.00	3363.48	2557.20	82	62	27.24	44.0-142.0	YES
Naphthalene	0.00	3518.13	2746.84	85	67	24.62	21.0-133.0	YES
Hexachlorobutadiene	0.00	3352.09	2423.31	81	59	32.16	24.0-116.0	YES
4-Chloro-3-methylphenol	0.00	2859.49	2823.15	69	69	1.28	22.0-147.0	YES
Hexachlorocyclopentadiene	0.00	1308.72	4078.26	16	50	-102.82	1.0-100.0	YES
2,4,6-Trichlorophenol	0.00	3497.81	2662.91	85	65	27.10	37.0-144.0	YES
2,4,5-Trichlorophenol	0.00	4067.08	3293.27	99	80	21.03	39.2-151.4	YES
2-Chloronaphthalene	0.00	3541.46	2846.72	86	69	21.75	60.0-118.0	YES
Dimethylphthalate	0.00	3537.29	2776.59	86	67	24.10	1.0-112.0	YES
2,6-Dinitrotoluene	0.00	3694.19	2922.38	90	71	23.33	50.0-158.0	YES
Acenaphthylene	0.00	3246.05	2821.60	79	68	13.99	33.0-145.0	YES
Acenaphthene	0.00	3345.51	2787.25	81	68	18.21	47.0-145.0	YES
2,4-Dinitrophenol	0.00	4014.86	2664.18	98	65	40.45	1.0-191.0	YES
4-Nitrophenol	0.00	2736.61	1867.67	66	45	37.74	1.0-132.0	YES
2,4-Dinitrotoluene	0.00	3681.75	2802.69	89	68	27.11	39.0-139.0	YES
Diethylphthalate	0.00	3470.08	2805.90	84	68	21.17	1.0-114.0	YES
4-Chlorophenyl-phenylether	0.00	3022.14	2606.55	73	63	14.77	25.0-158.0	YES
Fluorene	0.00	3295.26	2811.13	80	68	15.86	59.0-121.0	YES
4,6-Dinitro-2-methylphenol	0.00	3962.85	2795.01	96	68	34.56	1.0-181.0	YES
N-Nitrosodiphenylamine	0.00	4161.00	3326.51	101	81	22.29	37.8-147.0	YES

SOIL SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLE RECOVERY

Lab Name: LANCASTER LABS Lab Code: LANCAS Instrument: HP01597

SW846 METHOD 8270 SPIKE LEVEL: 100 UG/ML AMT USED: 30.0

SAMPLE SPIKE LEVEL: 4115.UG/KG % MOISTURE 19. DILUTION: 1

US SAMPLE: EDGE1 2321686 MS SAMPLE: EDGE1MS 2321686 MSD SAMPLE: EDGE1MSD 2321686

COMPOUND NAME	US CONC UG/KG	MS CONC UG/KG	MSD CONC UG/KG	MS REC %	MSD REC %	RPD %	RANGE LOWER-UPPER	IN SPEC
1,2-Diphenylhydrazine	0.00	3588.44	2999.77	87	73	17.87	25.7-124.9	YES
4-Bromophenyl-phenylether	0.00	3643.05	3083.36	88	75	16.64	53.0-127.0	YES
Hexachlorobenzene	0.00	3314.66	2835.07	80	69	15.60	1.0-152.0	YES
Pentachlorophenol	0.00	2968.18	2349.17	72	57	23.28	14.0-176.0	YES
Phenanthrene	0.00	3451.12	2899.17	84	70	17.38	54.0-120.0	YES
Anthracene	0.00	3317.50	2810.66	81	68	16.54	27.0-133.0	YES
Di-n-butylphthalate	0.00	3165.96	2613.52	77	64	19.12	1.0-118.0	YES
Fluoranthene	0.00	2842.48	2332.06	69	57	19.73	26.0-137.0	YES
Benzidine	0.00	0.00	1036.46	0	5	-200.00	1.0-155.0	NO
Pyrene	0.00	4496.35	4039.28	109	98	10.71	52.0-115.0	YES
Butylbenzylphthalate	0.00	4068.23	3511.16	99	85	14.70	1.0-152.0	YES
3,3'-Dichlorobenzidine	0.00	2929.76	2837.67	71	69	3.19	1.0-262.0	YES
Benzo(a)anthracene	0.00	3686.02	3113.91	90	76	16.83	33.0-143.0	YES
bis(2-Ethylhexyl)phthalate	0.00	3793.89	3307.93	92	80	13.69	8.0-158.0	YES
Chrysene	0.00	3689.95	3129.35	90	76	16.44	17.0-168.0	YES
Di-n-octylphthalate	0.00	3235.43	2875.64	79	70	11.77	4.0-146.0	YES
Benzo(b)fluoranthene	0.00	3272.29	2761.26	80	67	16.94	24.0-159.0	YES
Benzo(k)fluoranthene	0.00	3055.39	2634.77	74	64	14.78	11.0-163.0	YES
Benzo(a)pyrene	0.00	3336.59	2815.38	81	68	16.94	17.0-163.0	YES
Indeno(1,2,3-cd)pyrene	0.00	3411.71	2627.22	83	64	25.98	1.0-171.0	YES
Dibenz(a,h)anthracene	0.00	3268.85	2542.07	79	62	25.01	1.0-227.0	YES
Benzo(g,h,i)perylene	0.00	3358.45	2559.68	82	62	26.99	1.0-219.0	YES

COMMENTS:

WATER SEMIVOLATILE QUALITY CONTROL REFERENCE SAMPLE RECOVERY

LAB NAME: LANCASTER LABS LAB CODE: LANCAS INSTRUMENT: HP03301

SW846 METHOD 8270 SPIKE LEVEL: 100 UG/L

LCS SAMPLE NO: 171WALCS 171WALCS

COMPOUND NAME	EXTRACT CONC UG/L	QCREF REC %	RANGE LOWER-UPPER	IN SPEC
N-Nitrosodimethylamine	68.92	69	35.0- 100.8	YES
Phenol	48.72	49	5.0- 112.0	YES
bis(2-Chloroethyl)ether	94.39	94	12.0- 158.0	YES
2-Chlorophenol	92.50	92	23.0- 134.0	YES
1,3-Dichlorobenzene	85.86	86	1.0- 172.0	YES
1,4-Dichlorobenzene	88.26	88	20.0- 124.0	YES
1,2-Dichlorobenzene	91.46	91	32.0- 129.0	YES
bis(2-Chloroisopropyl)ether	101.78	102	36.0- 166.0	YES
N-Nitroso-di-n-propylamine	110.79	111	1.0- 230.0	YES
Hexachloroethane	74.98	75	40.0- 113.0	YES
Nitrobenzene	99.22	99	*35.0- 180.0*	*YES*
Isophorone	92.80	93	21.0- 196.0	YES
2-Nitrophenol	91.79	92	29.0- 182.0	YES
2,4-Dimethylphenol	80.92	81	32.0- 119.0	YES
bis(2-Chloroethoxy)methane	90.28	90	33.0- 184.0	YES
2,4-Dichlorophenol	93.31	93	39.0- 135.0	YES
1,2,4-Trichlorobenzene	84.99	85	44.0- 142.0	YES
Naphthalene	88.58	88	21.0- 133.0	YES
Hexachlorobutadiene	71.61	72	24.0- 116.0	YES
4-Chloro-3-methylphenol	96.46	96	22.0- 147.0	YES
Hexachlorocyclopentadiene	115.53	58	1.0- 100.0	YES
2,4,6-Trichlorophenol	93.96	94	37.0- 144.0	YES
2-Chloronaphthalene	88.09	88	60.0- 118.0	YES
Dimethylphthalate	86.59	86	1.0- 112.0	YES
2,6-Dinitrotoluene	87.41	87	50.0- 158.0	YES
Acenaphthylene	87.90	88	33.0- 145.0	YES
Acenaphthene	87.91	88	47.0- 145.0	YES
2,4-Dinitrophenol	99.86	100	1.0- 191.0	YES
4-Nitrophenol	47.64	48	1.0- 132.0	YES
2,4-Dinitrotoluene	104.96	105	39.0- 139.0	YES
1-Naphthylamine	40.76	41	1.0- 100.0	YES
2-Naphthylamine	52.41	52	1.0- 100.0	YES
Diethylphthalate	96.64	97	1.0- 114.0	YES
4-Chlorophenyl-phenylether	91.82	92	25.0- 158.0	YES
Fluorene	91.73	92	59.0- 121.0	YES
4,6-Dinitro-2-methylphenol	88.46	88	1.0- 181.0	YES
N-Nitrosodiphenylamine	82.97	83	37.8- 147.0	YES
1,2-Diphenylhydrazine	87.54	88	25.7- 124.9	YES
4-Bromophenyl-phenylether	92.08	92	53.0- 127.0	YES

WATER SEMIVOLATILE QUALITY CONTROL REFERENCE SAMPLE RECOVERY

LAB NAME: LANCASTER LABS. LAB CODE: LANCAS INSTRUMENT: HP03301

SW846 METHOD 8270 SPIKE LEVEL: 100 UG/L

LCS SAMPLE NO: 171WALCS 171WALCS

COMPOUND NAME	EXTRACT CONC UG/L	QCREF REC %	RANGE LOWER-UPPER	IN SPEC
Hexachlorobenzene	94.46	94	1.0- 152.0	YES
Pentachlorophenol	79.44	79	14.0- 176.0	YES
Phenanthrene	88.96	89	54.0- 120.0	YES
Anthracene	89.44	89	27.0- 133.0	YES
Di-n-butylphthalate	98.70	99	1.0- 118.0	YES
Fluoranthene	101.20	101	26.0- 137.0	YES
Benzidine	319.00	64	1.0- 155.0	YES
Pyrene	85.98	86	52.0- 115.0	YES
Butylbenzylphthalate	94.12	94	1.0- 152.0	YES
3,3'-Dichlorobenzidine	91.48	91	1.0- 262.0	YES
Benzo(a)anthracene	91.01	91	33.0- 143.0	YES
bis(2-Ethylhexyl)phthalate	95.20	95	8.0- 158.0	YES
Chrysene	92.76	93	17.0- 168.0	YES
Di-n-octylphthalate	95.22	95	4.0- 146.0	YES
Benzo(b)fluoranthene	92.11	92	24.0- 159.0	YES
Benzo(k)fluoranthene	92.30	92	11.0- 163.0	YES
Benzo(a)pyrene	86.47	86	17.0- 163.0	YES
Indeno(1,2,3-cd)pyrene	87.25	87	1.0- 171.0	YES
Dibenz(a,h)anthracene	87.06	87	1.0- 227.0	YES
Benzo(g,h,i)perylene	84.92	85	1.0- 219.0	YES

COMMENTS:

SOIL SEMIVOLATILE QUALITY CONTROL REFERENCE SAMPLE RECOVERY

LAB NAME: LANCASTER LABS LAB CODE: LANCAS INSTRUMENT: HP01597

SW846 METHOD 8270 SPIKE LEVEL: 100 UG/L

LCS SAMPLE NO: 156LALCS 156LALCS

COMPOUND NAME	EXTRACT CONC UG/L	QCREF REC %	RANGE LOWER-UPPER	IN SPEC
Pyridine	69.01	69	28.1- 100.0	YES
N-Nitrosodimethylamine	73.47	73	35.0- 100.8	YES
Phenol	75.17	75	5.0- 112.0	YES
bis(2-Chloroethyl)ether	82.26	82	12.0- 158.0	YES
2-Chlorophenol	84.78	85	23.0- 134.0	YES
1,3-Dichlorobenzene	84.04	84	1.0- 172.0	YES
1,4-Dichlorobenzene	83.77	84	20.0- 124.0	YES
1,2-Dichlorobenzene	87.98	88	32.0- 129.0	YES
2-Methylphenol	98.26	98	45.9- 122.5	YES
bis(2-Chloroisopropyl)ether	76.59	76	36.0- 166.0	YES
3 or 4-Methylphenol	102.03	102	53.6- 175.2	YES
N-Nitroso-di-n-propylamine	88.75	89	1.0- 230.0	YES
Hexachloroethane	87.03	87	40.0- 113.0	YES
Nitrobenzene	83.22	83	35.0- 180.0	YES
Isophorone	83.07	83	21.0- 196.0	YES
2-Nitrophenol	100.40	100	29.0- 182.0	YES
2,4-Dimethylphenol	82.11	82	32.0- 119.0	YES
bis(2-Chloroethoxy)methane	83.62	84	33.0- 184.0	YES
2,4-Dichlorophenol	94.66	95	39.0- 135.0	YES
1,2,4-Trichlorobenzene	89.03	89	44.0- 142.0	YES
Naphthalene	92.92	93	21.0- 133.0	YES
Hexachlorobutadiene	89.67	90	24.0- 116.0	YES
4-Chloro-3-methylphenol	84.62	85	22.0- 147.0	YES
Hexachlorocyclopentadiene	186.69	93	1.0- 100.0	YES
2,4,6-Trichlorophenol	90.31	90	37.0- 144.0	YES
2,4,5-Trichlorophenol	108.60	109	39.2- 151.4	YES
2-Chloronaphthalene	96.14	96	60.0- 118.0	YES
Dimethylphthalate	86.41	86	1.0- 112.0	YES
2,6-Dinitrotoluene	88.17	88	50.0- 158.0	YES
Acenaphthylene	92.80	93	33.0- 145.0	YES
Acenaphthene	91.01	91	47.0- 145.0	YES
2,4-Dinitrophenol	85.38	85	1.0- 191.0	YES
4-Nitrophenol	57.29	57	1.0- 132.0	YES
2,4-Dinitrotoluene	87.22	87	39.0- 139.0	YES
Diethylphthalate	85.45	85	1.0- 114.0	YES
4-Chlorophenyl-phenylether	80.72	81	25.0- 158.0	YES
Fluorene	86.70	87	59.0- 121.0	YES
4,6-Dinitro-2-methylphenol	91.84	92	1.0- 181.0	YES
N-Nitrosodiphenylamine	109.44	109	37.8- 147.0	YES

SOIL SEMIVOLATILE QUALITY CONTROL REFERENCE SAMPLE RECOVERY

LAB NAME: LANCASTER LABS LAB CODE: LANCAS INSTRUMENT: HP01597

SW846 METHOD 8270 SPIKE LEVEL: 100 UG/L

LCS SAMPLE NO: 156LALCS 156LALCS

COMPOUND NAME	EXTRACT CONC. UG/L	QCREF REC %	RANGE LOWER-UPPER	IN SPEC
1,2-Diphenylhydrazine	98.89	99	25.7- 124.9	YES
4-Bromophenyl-phenylether	100.31	100	53.0- 127.0	YES
Hexachlorobenzene	93.42	93	1.0- 152.0	YES
Pentachlorophenol	77.36	77	14.0- 176.0	YES
Phenanthrene	93.67	94	54.0- 120.0	YES
Anthracene	91.20	91	27.0- 133.0	YES
Di-n-butylphthalate	87.68	88	1.0- 118.0	YES
Fluoranthene	79.82	80	26.0- 137.0	YES
Benzidine	413.82	83	1.0- 155.0	YES
Pyrene	127.18	127	52.0- 115.0	NO
Butylbenzylphthalate	113.89	114	1.0- 152.0	YES
3,3'-Dichlorobenzidine	89.64	90	1.0- 262.0	YES
Benzo(a)anthracene	100.31	100	33.0- 143.0	YES
bis(2-Ethylhexyl)phthalate	110.28	110	8.0- 158.0	YES
Chrysene	102.15	102	17.0- 168.0	YES
Di-n-octylphthalate	98.97	99	4.0- 146.0	YES
Benzo(b)fluoranthene	96.06	96	24.0- 159.0	YES
Benzo(k)fluoranthene	85.00	85	11.0- 163.0	YES
Benzo(a)pyrene	93.18	93	17.0- 163.0	YES
Indeno(1,2,3-cd)pyrene	88.65	89	1.0- 171.0	YES
Dibenz(a,h)anthracene	86.02	86	1.0- 227.0	YES
Benzo(g,h,i)perylene	84.58	84	1.0- 219.0	YES

COMMENTS:

8B

SEMIVOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____. SAS No.: _____. SDG No.: _____.

Lab File ID (Standard): >Z7301 Date Analyzed: 07/05/95

Instrument ID: HP02550 Time Analyzed: 16:08

	EPA SAMPLE NO.	IS1(DCB) AREA #	RT	IS2(NPT) AREA #	RT	IS3(ANT) AREA #	RT
	12 HOUR STD	41975	11.65	146677	15.00	76661	19.81
	UPPER LIMIT	83950	12.15	293354	15.50	153322	20.31
	LOWER LIMIT	20988	11.15	73339	14.50	38331	19.31
01	0303BMSD	43212	11.66	147781	15.01	77687	19.81
02	0400B	44648	11.67	148989	15.01	77461	19.81
03	0303BMS	41859	11.67	147450	15.01	79313	19.81
04	0405B	40183	11.66	137070	15.01	73874	19.81
05							
06							
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18							
19							
20							
21							
22							

IS1 (DCB) = 1,4-Dichlorobenzene-d4
IS2 (NPT) = Naphthalene-d8
IS3 (ANT) = Acenaphthene-d10

AREA UPPER LIMIT = +100% of internal standard area
AREA LOWER LIMIT = -50% of internal standard area
RT UPPER LIMIT = +0.50 minutes of internal standard RT
RT LOWER LIMIT = -0.50 minutes of internal standard RT

Column used to flag internal standard area values with an asterisk.
* Values outside of QC limits.

8C

SEMIVOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____. SAS No.: _____. SDG No.: _____.

Lab File ID (Standard): >Z7301 Date Analyzed: 07/05/95

Instrument ID: HP02550 Time Analyzed: 16:08

		IS4(PHN) AREA	#	RT	IS5(CRY) AREA	#	RT	IS6(PRY) AREA	#	RT
	12 HOUR STD	113834		23.91	104503		30.73	48838		35.61
	UPPER LIMIT	227668		24.41	209006		31.23	97676		36.11
	LOWER LIMIT	56917		23.41	52252		30.23	24419		35.11
	EPA SAMPLE NO.									
01	0303BMSD	119527		23.93	68256		30.71	28576		35.62
02	0400B	120999		23.92	72241		30.71	27091		35.61
03	0303BMS	122200		23.93	71578		30.72	26418		35.62
04	0405B	109187		23.93	56771		30.72	25260		35.63
05										
06										
07										
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09										
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17										
18										
19										
20										
21										
22										

IS4 (PHN) = Phenanthrene-d10
IS5 (CRY) = Chrysene-d12
IS6 (PRY) = Perylene-d12
AREA UPPER LIMIT = +100% of internal standard area
AREA LOWER LIMIT = - 50% of internal standard area
RT UPPER LIMIT = +0.50 minutes of internal standard RT
RT LOWER LIMIT = -0.50 minutes of internal standard RT

Column used to flag internal standard area values with an asteris)
* Values outside of QC limits.

SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____.SAS No.: _____. SDG No.: _____.

Instrument ID: HP03189 Calibration Date(s): 06/27/95 06/28/95

Min RRF for SPCC(#) = 0.050 Max %RSD for CCC(*) = 30.0%

LAB FILE ID: RRF5 = >V6255 RRF50 = >V6253
RRF80 = >V6254 RRF120= >V6252 RRF160= >V6251

COMPOUND	RRF5	RRF50	RRF80	RRF120	RRF160	RRF	% RSD	CAL. METHOD
Pyridine	2.209	2.342	2.408	2.388	2.237	2.317	3.9	AVG
N-Nitrosodimethylamine	1.364	1.415	1.410	1.407	1.337	1.387	2.5	AVG
2-Picoline	1.926	2.008	2.036	2.130	2.058	2.032	3.7	AVG
Phenol *	2.829	2.495	2.447	2.372	2.184	2.465	9.5	AVG *
Aniline	3.094	2.723	2.510	2.507	2.377	2.642	10.7	AVG
bis(2-Chloroethyl)ether	1.993	1.711	1.645	1.508	1.364	1.644	14.4	AVG
2-Chlorophenol	2.142	2.017	1.983	1.899	1.722	1.953	8.0	AVG
1,3-Dichlorobenzene	2.598	2.319	2.291	2.140	1.982	2.266	10.1	AVG
1,4-Dichlorobenzene *	2.597	2.332	2.315	2.141	1.987	2.274	10.1	AVG *
Benzyl alcohol	1.026	1.024	1.034	1.011	.964	1.012	2.7	AVG
1,2-Dichlorobenzene	2.410	2.017	1.933	1.749	1.546	1.931	16.7	2NDDEG
2-Methylphenol	1.710	1.633	1.661	1.621	1.546	1.634	3.7	AVG
2,2'-oxybis(1-Chloropropane)	4.554	4.381	4.436	4.318	3.919	4.322	5.6	AVG
bis(2-Chloroisopropyl)ether	4.554	4.381	4.436	4.318	3.919	4.322	5.6	AVG
4-Methylphenol	2.047	1.659	1.515	1.329	1.176	1.545	21.7	2NDDEG
3 or 4-Methylphenol	2.047	1.659	1.515	1.329	1.176	1.545	21.7	2NDDEG
Acetophenone	6.825	5.536	5.590	5.364	4.908	5.645	12.6	AVG
N-Nitroso-di-n-propylamine #	1.652	1.502	1.466	1.243	.922	1.357	20.9	2NDDEG #
o-Toluidine	4.277	3.462	3.154	3.247	2.841	3.396	15.9	2NDDEG
Hexachloroethane	.982	.991	1.008	.971	.878	.966	5.3	AVG
Nitrobenzene	.629	.672	.677	.677	.637	.658	3.6	AVG
Isophorone	1.110	1.151	1.177	1.205	1.177	1.164	3.1	AVG
2-Nitrophenol *	.251	.336	.335	.347	.327	.319	12.2	AVG *
2,4-Dimethylphenol	.594	.599	.603	.455	.581	.566	11.1	AVG
Benzoic acid	.313	.367	.418	.454	.455	.401	15.2	2NDDEG
bis(2-Chloroethoxy)methane	.703	.669	.671	.685	.657	.677	2.6	AVG
2,4-Dichlorophenol *	.488	.501	.494	.489	.466	.488	2.7	AVG *
1,2,4-Trichlorobenzene	.582	.575	.561	.553	.509	.556	5.2	AVG
Naphthalene	1.631	1.540	1.540	1.457	1.320	1.498	7.8	AVG
4-Chloroaniline	.703	.692	.704	.688	.644	.686	3.6	AVG
Hexachlorobutadiene *	.378	.376	.377	.380	.328	.368	6.1	AVG *
4-Chloro-3-methylphenol *	.454	.517	.536	.516	.470	.499	7.0	AVG *
2-Methylnaphthalene	1.056	.971	.979	.916	.842	.953	8.3	AVG
Hexachlorocyclopentadiene #	.473	.717	.808	.896	.877	.754	22.8	2NDDEG #
2,4,6-Trichlorophenol *	.646	.710	.764	.805	.814	.748	9.4	AVG *
2,4,5-Trichlorophenol	.688	.768	.806	.832	.800	.779	7.2	AVG
2-Chloronaphthalene	2.025	1.878	1.968	1.986	1.928	1.957	2.9	AVG
2-Nitroaniline	.587	.810	.886	.918	.917	.824	16.9	2NDDEG

SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____.SAS No.: _____. SDG No.: _____.

Instrument ID: HP03189 Calibration Date(s): 06/27/95 06/28/95

Min RRF for SPCC(#) = 0.050 Max %RSD for CCC(*) = 30.0%

LAB FILE ID: RRF5 = >V6255 RRF50 = >V6253
RRF80 = >V6254 RRF120= >V6252 RRF160= >V6251

COMPOUND	RRF5	RRF50	RRF80	RRF120	RRF160	RRF	% RSD	CAL. METHOD
Dimethylphthalate	2.261	2.254	2.330	2.273	2.270	2.278	1.3	AVG
2,6-Dinitrotoluene	.349	.519	.539	.518	.501	.485	15.9	2NDDEG
Acenaphthylene	3.176	3.027	3.096	3.021	2.837	3.032	4.1	AVG
3-Nitroaniline	.430	.579	.588	.575	.580	.550	12.2	AVG
Acenaphthene *	2.043	1.911	1.934	1.941	1.836	1.933	3.8	AVG *
2,4-Dinitrophenol #	.200	.255	.319	.342	.350	.293	21.9	2NDDEG #
4-Nitrophenol #	.247	.330	.356	.329	.305	.313	13.1	AVG #
Dibenzofuran	2.965	2.634	2.573	2.472	2.302	2.589	9.5	AVG
2,4-Dinitrotoluene	.639	.727	.764	.724	.728	.717	6.5	AVG
1-Naphthylamine	2.389	2.081	2.245	2.219	2.082	2.203	5.8	AVG
2-Naphthylamine	1.992	1.750	1.926	1.933	1.867	1.893	4.8	AVG
Diethylphthalate	2.257	2.214	2.285	2.202	2.121	2.216	2.8	AVG
4-Chlorophenyl-phenylether	1.116	1.055	1.095	1.046	1.015	1.065	3.8	AVG
Fluorene	2.127	2.066	2.034	1.971	1.854	2.011	5.2	AVG
4-Nitroaniline	.340	.523	.559	.551	.558	.506	18.6	1STDEG
4,6-Dinitro-2-methylphenol	.164	.228	.243	.230	.214	.216	14.3	AVG
N-Nitrosodiphenylamine (1) *	.913	.878	.901	.884	.826	.880	3.8	AVG *
1,2-Diphenylhydrazine	1.615	1.564	1.623	1.628	1.530	1.592	2.7	AVG
4-Bromophenyl-phenylether	.427	.431	.461	.448	.405	.434	4.9	AVG
Hexachlorobenzene	.622	.598	.606	.596	.536	.592	5.5	AVG
Pentachlorophenol *	.285	.301	.333	.340	.321	.316	7.1	2NDDEG *
Phenanthrene	2.084	1.809	1.839	1.727	1.550	1.802	10.7	AVG
Anthracene	1.974	1.888	1.843	1.768	1.630	1.821	7.1	AVG
Carbazole	1.611	1.704	1.696	1.598	1.453	1.612	6.3	AVG
Di-n-butylphthalate	1.789	2.216	2.170	2.113	1.865	2.031	9.4	AVG
Fluoranthene *	1.773	1.992	1.869	1.749	1.467	1.770	11.0	AVG *
Benzidine	1.272	1.031	1.100	1.092	1.058	1.111	8.5	AVG
Pyrene	2.420	2.464	3.044	3.047	2.917	2.779	11.2	AVG
Butylbenzylphthalate	.725	1.077	1.132	1.102	1.085	1.024	16.5	1STDEG
3,3'-Dichlorobenzidine	.527	.638	.732	.774	.820	.698	16.7	1STDEG
Benzo(a)anthracene	1.751	1.797	1.845	1.953	1.956	1.861	4.9	AVG
bis(2-Ethylhexyl)phthalate	1.073	1.442	1.470	1.452	1.445	1.376	12.4	AVG
Chrysene	1.782	1.738	1.798	1.880	1.888	1.817	3.6	AVG
Di-n-octylphthalate *	2.921	3.449	3.004	3.320	3.296	3.198	7.0	2NDDEG *
7,12-Dimethylbenz[a]anthrace	1.302	1.273	1.261	1.359	1.288	1.297	3.0	AVG
Benzo(b)fluoranthene	2.863	2.440	2.659	2.620	2.739	2.664	5.9	AVG
Benzo(k)fluoranthene	2.657	2.473	2.469	2.609	2.304	2.502	5.5	AVG

(1) Cannot be separated from Diphenylamine

6C Cont.

SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____.SAS No.: _____. SDG No.: _____.

Instrument ID: HP03189 Calibration Date(s): 06/27/95 06/28/95

Min RRF for SPCC(#) = 0.050 Max %RSD for CCC(*) = 30.0%

LAB FILE ID: RRF5 = >V6255 RRF50 = >V6253
RRF80 = >V6254 RRF120= >V6252 RRF160= >V6251

COMPOUND	RRF5	RRF50	RRF80	RRF120	RRF160	RRF	%RSD	CAL. METHOD
Benzo(a)pyrene *	2.221	2.245	2.371	2.380	2.215	2.286	3.6	AVG *
Indeno(1,2,3-cd)pyrene	1.379	1.900	1.747	1.782	1.630	1.687	11.7	AVG
Dibenz(a,h)anthracene	1.399	1.881	1.686	1.749	1.631	1.669	10.6	AVG
Benzo(g,h,i)perylene	1.290	1.854	1.643	1.669	1.530	1.597	13.0	AVG
2-Fluorophenol	1.820	1.850	1.874	1.832	1.734	1.822	2.9	AVG
Phenol-d5	2.400	2.227	2.123	2.048	1.895	2.139	8.9	AVG
Phenol-d6	2.400	2.227	2.123	2.048	1.895	2.139	8.9	AVG
Nitrobenzene-d5	.572	.634	.655	.672	.638	.634	6.0	AVG
2-Fluorobiphenyl	2.316	2.138	2.205	2.181	2.162	2.200	3.2	AVG
2,4,6-Tribromophenol	.435	.518	.586	.553	.524	.523	10.8	AVG
Terphenyl-d14	1.380	1.658	1.996	1.897	1.808	1.748	13.7	AVG

4,6-Dinitro-2-methylphenol and 4-Nitrophenol are at 10 ng/uL in the 5 standard.
2,4-Dinitrophenol and 2 or 4-Chloronitrobenzene levels are 40 and 100 ng/uL respectively in the 5 standard.
Benzoic acid and Pentachlorophenol are at 20 ng/uL in the 5 standard.
Benzidine levels in the 5,50,80,120,160 standards are 95,200,320,480 and 640 ng/uL respectively.

Initial Calibration Data
HSL Compounds

Case No: Instrument ID: HP03189

Contractor: LANCASTER LABS Calibration Date: 06/28/95

Contract No:

Minimum $\overline{RF}$ for SPCC is 0.05 Maximum % RSD for CCC is 30.0%

Laboratory ID:	>V6255	>V6253	>V6254	>V6252	>V6251						
Compound	RF 5.00	RF 50.00	RF 80.00	RF 120.00	RF 160.00	$\overline{RRT}$	$\overline{RF}$	% RSD	CORR1	CORR2	CCC SPCC
Pyridine	1.38088	1.46399	1.50487	1.49281	1.39794	.259	1.44810	3.862	.998279	.999317	
2-Picoline	1.20375	1.25487	1.27265	1.33115	1.28612	.463	1.26971	3.657	.999470	.999473	
N-Nitrosodimethylamine	.85223	.88437	.88145	.87966	.83543	.256	.86663	2.505	.999003	.999694	
3-Chloropropionitrile	-	-	-	-	-	-	-	-	-	-	
Methyl methanesulfonate	-	-	-	-	-	-	-	-	-	-	
Phenol	1.76798	1.55918	1.52924	1.48261	1.36499	.912	1.54080	9.537	.996933	.999390	*
Aniline	1.93389	1.70183	1.56847	1.56675	1.48548	.907	1.65128	10.659	.998645	.999650	
bis(2-Chloroethyl)ether	1.24564	1.06949	1.02811	.94230	.85281	.934	1.02767	14.361	.992803	.999484	
2-Chlorophenol	1.33872	1.26047	1.23940	1.18673	1.07652	.939	1.22037	7.966	.995277	.999092	
1,3-Dichlorobenzene	1.62398	1.44936	1.43212	1.33748	1.23856	.982	1.41630	10.127	.995980	.999667	
1,4-Dichlorobenzene	1.62289	1.45768	1.44714	1.33786	1.24186	1.005	1.42148	10.059	.995689	.999672	*
Benzyl alcohol	.64116	.63989	.64618	.63211	.60274	1.049	.63242	2.742	.998922	.999794	
1,2-Dichlorobenzene	1.50654	1.26080	1.20831	1.09342	.96645	1.047	1.20710	16.743	.989268	.998784	
2-Methylphenol	1.06906	1.02068	1.03824	1.01341	.96625	1.089	1.02153	3.682	.998915	.999754	
2,2'-oxybis(1-Chloropropane)	2.84608	2.73835	2.77272	2.69897	2.44925	1.093	2.70107	5.583	.995947	.998684	
bis(2-Chloroisopropyl)ether	2.84608	2.73835	2.77272	2.69897	2.44925	1.093	2.70107	5.583	.995947	.998684	
4-Methylphenol	1.27931	1.03681	.94716	.83044	.73515	1.135	.96577	21.670	.985430	.999485	
3 or 4-Methylphenol	1.27931	1.03681	.94716	.83044	.73515	1.135	.96577	21.670	.985430	.999485	
N-Methylaniline	-	-	-	-	-	-	-	-	-	-	
Acetophenone	4.26555	3.45993	3.49401	3.35231	3.06730	1.127	3.52782	12.620	.996392	.999046	
N-Nitroso-di-n-propylamine	1.03265	.93870	.91620	.77714	.57599	1.132	.84813	20.925	.940946	.961451	**
o-Toluidine	2.67333	2.16375	1.97112	2.02910	1.77576	1.136	2.12261	15.924	.993679	.996437	
Hexachloroethane	.61351	.61966	.62997	.60694	.54893	1.147	.60380	5.271	.995270	.998633	
2-Fluorophenol	1.13750	1.15613	1.17156	1.14477	1.08383	.636	1.13876	2.923	.998591	.999696	
Phenol-d5	1.50024	1.39209	1.32710	1.28028	1.18458	.908	1.33686	8.869	.996816	.999674	
Phenol-d6	1.50024	1.39209	1.32710	1.28028	1.18458	.908	1.33686	8.869	.996816	.999674	
Nitrobenzene	.39301	.41991	.42313	.42316	.39824	.849	.41149	3.562	.998634	.999481	
N,N-Dimethylaniline	-	-	-	-	-	-	-	-	-	-	

RF - Response Factor (Subscript is amount in ng/ul)

$\overline{RRT}$ - Average Relative Retention Time (RT Std/RT Istd)

$\overline{RF}$ - Average Response Factor

%RSD - Percent Relative Standard Deviation

CORRn - Coefficient of Correlation (nth degree)

CCC - Calibration Check Compounds (*) SPCC - System Performance Check Compounds (**)

RE 6/28/95

Initial Calibration Data
NSL Compounds

Case No: Instrument ID: HP03189

Contractor: LANCASTER LABS Calibration Date: 06/28/95

Contract No:

Minimum $\overline{RF}$ for SPCC is 0.05 Maximum % RSD for CCC is 30.0%

Laboratory ID: >V6255 >V6253 >V6254 >V6252 >V6251

Compound	RF 5.00	RF 50.00	RF 80.00	RF 120.00	RF 160.00	$\overline{RRT}$	$\overline{RF}$	% RSD	CORR1	CORR2	CCC	SPCC	
Isophorone	.69355	.71911	.73588	.75335	.73587	.904	.72755	3.098	.999757	.999765			
2-Nitrophenol	.15673	.20999	.20927	.21659	.20464	.918	.19944	12.161	.998905	.999289	*		
2,4-Dimethylphenol	.37144	.37436	.37659	.28416	.36312	.937	.35394	11.115	.979572	.982405			
Benzoic acid	.19591	.22950	.26096	.28403	.28418	.973	.25092	15.160	.999208	.999308			(Conc=20.
bis(2-Chloroethoxy)methane	.43929	.41809	.41920	.42831	.41085	.959	.42315	2.588	.999465	.999608			
1-Methyl-2-nitrobenzene	-	-	-	-	-	-	-	-	-	-			
2,4-Dichlorophenol	.30512	.31282	.30862	.30573	.29118	.973	.30469	2.672	.999009	.999810	*		
1,2,4-Trichlorobenzene	.36356	.35911	.35050	.34562	.31790	.990	.34734	5.153	.997118	.999248			
1,3-Dimethyl-2-nitrobenzene	-	-	-	-	-	-	-	-	-	-			
Naphthalene	1.01932	.96255	.96279	.91047	.82497	1.005	.93602	7.804	.994744	.998998			
1-Methyl-3-nitrobenzene	-	-	-	-	-	-	-	-	-	-			
4-Chloroaniline	.43967	.43258	.44031	.43007	.40257	1.023	.42904	3.601	.998054	.999471			
Hexachlorobutadiene	.23605	.23527	.23559	.23738	.20494	1.038	.22985	6.067	.992078	.995408	*		
1-Methyl-4-nitrobenzene	-	-	-	-	-	-	-	-	-	-			
2 or 4-Choloronitrobenzene	-	-	-	-	-	-	-	-	-	-			
2-Tertbutylphenol	-	-	-	-	-	-	-	-	-	-			
1,4-Dimethyl-2-nitrobenzene	-	-	-	-	-	-	-	-	-	-			
4-Chloro-3-methylphenol	.28401	.32316	.33489	.32269	.29348	1.140	.31165	6.972	.995592	.998662	*		
3 or 4-Tertbutylphenol	-	-	-	-	-	-	-	-	-	-			
2-Methylnaphthalene	.65989	.60717	.61176	.57262	.52622	1.164	.59553	8.341	.995542	.999353			
Nitrobenzene-d5	.35737	.39655	.40945	.41992	.39875	.845	.39641	5.986	.999063	.999292			
Hexachlorocyclopentadiene	.29573	.44824	.50519	.56009	.54839	.858	.47153	22.824	.997953	.998449		**	
2,4,6-Trichlorophenol	.40358	.44376	.47732	.50322	.50866	.879	.46731	9.399	.999136	.999692	*		
2,4,5-Trichlorophenol	.42992	.48022	.50371	.52018	.49977	.884	.48676	7.152	.999308	.999354			
2-Chloronaphthalene	1.26531	1.17375	1.23027	1.24134	1.20491	.910	1.22312	2.871	.999578	.999626			
1,2-Dichloro-4-nitrobenzene	-	-	-	-	-	-	-	-	-	-			
1,2-Dichloro-3-nitrobenzene	-	-	-	-	-	-	-	-	-	-			
2,6-Ditertbutylphenol	-	-	-	-	-	-	-	-	-	-			

RF - Response Factor (Subscript is amount in ng/ul)

$\overline{RRT}$ - Average Relative Retention Time (RT Std/RT Istd)

$\overline{RF}$ - Average Response Factor

%RSD - Percent Relative Standard Deviation

CORRn - Coefficient of Correlation (nth degree)

CCC - Calibration Check Compounds (*) SPCC - System Performance Check Compounds (**)

Initial Calibration Data
HSL Compounds

Case No: Instrument ID: HP03189

Contractor: LANCASTER LABS Calibration Date: 06/28/95

Contract No:

Minimum $\overline{RF}$ for SPCC is 0.05 Maximum % RSD for CCC is 30.0%

Laboratory ID: >V6255 >V6253 >V6254 >V6252 >V6251

Compound	RF 5.00	$RF_{50.00}$	RF 80.00	RF 120.00	RF 160.00	$\overline{RRT}$	$\overline{RF}$	% RSD	CORR1	CORR2	CCC	SPCC	
2-Nitroaniline	.36685	.50597	.55398	.57376	.57317	.930	.51475	16.932	.999486	.999623			
1,4-Naphthoquinone	-	-	-	-	-	-	-	-	-	-			
Dimethylphthalate	1.41281	1.40877	1.45645	1.42087	1.41882	.966	1.42355	1.335	.999865	.999901			
3,4-Dichloro-nitrobenzene	-	-	-	-	-	-	-	-	-	-			
Acenaphthylene	1.98511	1.89204	1.93526	1.88824	1.77331	.976	1.89479	4.139	.998229	.999494			
2,4-Ditertbutylphenol	-	-	-	-	-	-	-	-	-	-			
2,6-Dinitrotoluene	.21818	.32428	.33713	.32363	.31307	.974	.30326	15.933	.998817	.999823			
3-Nitroaniline	.26895	.36198	.36733	.35939	.36245	.999	.34402	12.226	.999889	.999920			
3,4-Dichloroaniline	-	-	-	-	-	-	-	-	-	-			
Acenaphthene	1.27673	1.19417	1.20874	1.21317	1.14745	1.006	1.20805	3.840	.998950	.999486	*		
BHT	-	-	-	-	-	-	-	-	-	-			
2,4-Dinitrophenol	.12489	.15954	.19965	.21350	.21893	1.017	.18330	21.865	.999458	.999958		**	(Conc:
4-Nitrophenol	.15438	.20623	.22222	.20557	.19043	1.033	.19577	13.140	.994540	.998997		**	(Conc=10.
3,5-Ditertbutylphenol	-	-	-	-	-	-	-	-	-	-			
Dibenzofuran	1.85327	1.64651	1.60817	1.54503	1.43890	1.035	1.61838	9.455	.997369	.999712			
2,4-Dinitrotoluene	.39948	.45413	.47774	.45246	.45529	1.039	.44782	6.461	.999474	.999619			
1-Naphthylamine	1.49330	1.30071	1.40330	1.38668	1.30136	1.049	1.37707	5.838	.998181	.998845			
2-Naphthylamine	1.24483	1.09370	1.20360	1.20807	1.16657	1.062	1.18335	4.840	.999073	.999124			
Diethylphthalate	1.41047	1.38369	1.42813	1.37648	1.32566	1.085	1.38489	2.821	.999016	.999784			
4-Chlorophenyl-phenylether	.69726	.65907	.68413	.65391	.63413	1.096	.66570	3.767	.999086	.999766			
Fluorene	1.32926	1.29133	1.27131	1.23203	1.15895	1.091	1.25658	5.164	.998082	.999787			
4-Nitroaniline	.21247	.32706	.34919	.34412	.34904	1.100	.31638	18.582	.999836	.999840			
2-Fluorobiphenyl	1.44779	1.33640	1.37809	1.36288	1.35114	.895	1.37526	3.151	.999903	.999920			
2,4,6-Tribromophenol	.27188	.32388	.36653	.34569	.32748	1.130	.32709	10.766	.997152	.998619			
4,6-Dinitro-2-methylphenol	.10236	.14261	.15181	.14347	.13361	.889	.13477	14.267	.995876	.999298			(Conc=10.
N-Nitrosodiphenylamine	.57071	.54875	.56285	.55248	.51625	.898	.55021	3.791	.998071	.999336	*		
1,2-Diphenylhydrazine	1.00912	.97774	1.01429	1.01739	.95595	.902	.99490	2.704	.998583	.999198			
1-Nitronaphthalene	-	-	-	-	-	-	-	-	-	-			

RF - Response Factor (Subscript is amount in ng/ul)

$\overline{RRT}$ - Average Relative Retention Time (RT Std/RT Istd)

$\overline{RF}$ - Average Response Factor

%RSD - Percent Relative Standard Deviation

CORRn - Coefficient of Correlation (nth degree)

CCC - Calibration Check Compounds (*) SPCC - System Performance Check Compounds (**)

Initial Calibration Data
HSL Compounds

Case No: Instrument ID: HP03189

Contractor: LANCASTER LABS Calibration Date: 06/28/95

Contract No:

Minimum $\overline{RF}$ for SPCC is 0.05 Maximum % RSD for CCC is 30.0%

Laboratory ID:	>V6255	>V6253	>V6254	>V6252	>V6251							
Compound	RF 5.00	RF 50.00	RF 80.00	RF 120.00	RF 160.00	$\overline{RRT}$	$\overline{RF}$	% RSD	CORR1	CORR2	CCC	SPCC
4-Methyl-3-nitrobenzoic acid	-	-	-	-	-	-	-	-	-	-		
4-Bromophenyl-phenylether	.26716	.26914	.28797	.28002	.25326	.946	.27151	4.875	.995471	.997912		
Hexachlorobenzene	.38890	.37360	.37881	.37219	.33499	.949	.36970	5.540	.995545	.998231		
Pentachlorophenol	.17835	.18836	.20794	.21264	.20060	.977	.19758	7.150	.998091	.998821	*	(Conc=20.
Phenanthrene	1.30231	1.13074	1.14906	1.07919	.96863	1.003	1.12599	10.749	.993780	.998476		
Anthracene	1.23366	1.17976	1.15214	1.10502	1.01880	1.010	1.13787	7.139	.996521	.999554		
Carbazole	1.00677	1.06470	1.06005	.99880	.90799	1.035	1.00766	6.279	.994617	.999200		
Di-n-butylphthalate	1.11785	1.38509	1.35616	1.32072	1.16592	1.091	1.26915	9.424	.992858	.997819		
Diphenyl sulfone	-	-	-	-	-	-	-	-	-	-		
Fluoranthene	1.10814	1.24518	1.16830	1.09288	.91712	1.161	1.10632	10.987	.982971	.994920	*	
Benzidine	.79530	.64462	.68744	.68233	.66132	.878	.69420	8.506	.998910	.998932		(Conc=95.
Pyrene	1.51267	1.54018	1.90263	1.90440	1.82332	.881	1.73664	11.224	.997316	.997391		
Butylbenzylphthalate	.45305	.67294	.70737	.68846	.67829	.954	.64002	16.459	.999558	.999854		
3,3'-Dichlorobenzidine	.32924	.39869	.45773	.48361	.51241	1.001	.43634	16.749	.997334	.999560		
Benzo(a)anthracene	1.09443	1.12340	1.15327	1.22088	1.22224	.999	1.16284	4.945	.999403	.999756		
Chrysene	1.11392	1.08629	1.12381	1.17478	1.17991	1.003	1.13574	3.560	.999513	.999833		
bis(2-Ethylhexyl)phthalate	.67037	.90094	.91858	.90758	.90337	1.014	.86017	12.360	.999907	.999982		
Terphenyl-d14	.86274	1.03648	1.24725	1.18543	1.12976	.902	1.09233	13.725	.996937	.997799		
Di-n-octylphthalate	1.82533	2.15541	1.87757	2.07480	2.05971	.952	1.99856	7.023	.998195	.998346	*	
7,12-Dimethylbenz[a]anthracene	.81365	.79566	.78786	.84968	.80496	.973	.81036	2.964	.998711	.998713		
Benzo(b)fluoranthene	1.78963	1.52473	1.66213	1.63769	1.71180	.972	1.66520	5.865	.998972	.999625		
Benzo(k)fluoranthene	1.66053	1.54548	1.54306	1.63048	1.44016	.975	1.56394	5.524	.995044	.996068		
Benzo(a)pyrene	1.38819	1.40317	1.48194	1.48731	1.38459	.996	1.42904	3.587	.998038	.998808	*	
Indeno(1,2,3-cd)pyrene	.86170	1.18761	1.09197	1.11352	1.01853	1.087	1.05466	11.716	.996458	.998741		
Dibenz(a,h)anthracene	.87427	1.17539	1.05383	1.09305	1.01953	1.091	1.04321	10.628	.997517	.998805		
Benzo(g,h,i)perylene	.80602	1.15883	1.02677	1.04306	.95595	1.113	.99813	13.000	.995775	.998692		

RF - Response Factor (Subscript is amount in ng/ul)

$\overline{RRT}$ - Average Relative Retention Time (RT Std/RT Istd)

$\overline{RF}$ - Average Response Factor

%RSD - Percent Relative Standard Deviation

CORRn - Coefficient of Correlation (nth degree)

CCC - Calibration Check Compounds (*) SPCC - System Performance Check Compounds (**)

SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____. SAS No.: _____. SDG No.: _____.

Instrument ID: HP03189 Calibration Date: 06/28/95 Time: 13:03

Lab File ID: >V6303 Init. Calib. Date(s): 06/27/95 06/28/95

Min RRF50 for SPCC(#) = 0.050 Max %Drift for CCC(*) = 20

COMPOUND		RRF	RRF80	ACTUAL CONC	TRUE CONC	% DRIFT
Pyridine		2.317	1.488	82.20	80.0	-2.8
N-Nitrosodimethylamine		1.387	.878	81.02	80.0	-1.3
2-Picoline		2.032	1.292	81.41	80.0	-1.8
Phenol	*	2.465	1.514	78.60	80.0	1.8*
Aniline		2.642	1.580	76.53	80.0	4.3
bis(2-Chloroethyl)ether		1.644	.969	75.43	80.0	5.7
2-Chlorophenol		1.953	1.238	81.18	80.0	-1.5
1,3-Dichlorobenzene		2.266	1.408	79.53	80.0	.6
1,4-Dichlorobenzene	*	2.274	1.404	78.99	80.0	1.3*
Benzyl alcohol		1.012	.635	80.30	80.0	-.4
1,2-Dichlorobenzene		1.931	1.153	76.10	80.0	4.9
2-Methylphenol		1.634	1.039	81.38	80.0	-1.7
2,2'-oxybis(1-Chloropropane)		4.322	2.682	79.45	80.0	.7
bis(2-Chloroisopropyl)ether		4.322	2.682	79.45	80.0	.7
4-Methylphenol		1.545	.885	73.51	80.0	8.1
3 or 4-Methylphenol		1.545	.885	73.51	80.0	8.1
Acetophenone		5.645	3.435	77.89	80.0	2.6
N-Nitroso-di-n-propylamine	#	1.357	.872	83.41	80.0	-4.3#
o-Toluidine		3.396	1.872	71.74	80.0	10.3
Hexachloroethane		.966	.606	80.33	80.0	-.4
Nitrobenzene		.658	.427	83.11	80.0	-3.9
Isophorone		1.164	.729	80.17	80.0	-.2
2-Nitrophenol	*	.319	.221	88.47	80.0	-10.6*
2,4-Dimethylphenol		.566	.380	86.00	80.0	-7.5
Benzoic acid		.401	.269	81.38	80.0	-1.7
bis(2-Chloroethoxy)methane		.677	.423	79.94	80.0	.1
2,4-Dichlorophenol	*	.488	.311	81.53	80.0	-1.9*
1,2,4-Trichlorobenzene		.556	.357	82.19	80.0	-2.7
Naphthalene		1.498	.938	80.19	80.0	-.2
4-Chloroaniline		.686	.439	81.83	80.0	-2.3
Hexachlorobutadiene	*	.368	.237	82.48	80.0	-3.1*
4-Chloro-3-methylphenol	*	.499	.330	84.71	80.0	-5.9*
2-Methylnaphthalene		.953	.610	81.98	80.0	-2.5
Hexachlorocyclopentadiene	#	.754	.534	83.13	80.0	-3.9#
2,4,6-Trichlorophenol	*	.748	.475	81.27	80.0	-1.6*
2,4,5-Trichlorophenol		.779	.535	87.97	80.0	-10.0
2-Chloronaphthalene		1.957	1.206	78.91	80.0	1.4
2-Nitroaniline		.824	.564	81.85	80.0	-2.3

SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____. SAS No.: _____. SDG No.: _____.

Instrument ID: HP03189 Calibration Date: 06/28/95 Time: 13:03

Lab File ID: >V6303 Init. Calib. Date(s): 06/27/95 06/28/95

Min RRF50 for SPCC(#) = 0.050 Max %Drift for CCC(*) = 20.0

COMPOUND	RRF	RRF80	ACTUAL CONC	TRUE CONC	% DRIFT
Dimethylphthalate	2.278	1.441	81.01	80.0	-1.3
1,3-Dinitrobenzene	0.000	0.000	0.00	80.0	100.0
2,6-Dinitrotoluene	.485	.341	82.33	80.0	-2.9
Acenaphthylene	3.032	1.924	81.21	80.0	-1.5
3-Nitroaniline	.550	.360	83.65	80.0	-4.6
Acenaphthene *	1.933	1.213	80.30	80.0	-.4*
2,4-Dinitrophenol #	.293	.193	78.49	80.0	1.9#
4-Nitrophenol #	.313	.204	83.41	80.0	-4.3#
Dibenzofuran	2.589	1.637	80.93	80.0	-1.2
2,4-Dinitrotoluene	.717	.455	81.37	80.0	-1.7
1-Naphthylamine	2.203	1.373	79.79	80.0	.3
2-Naphthylamine	1.893	1.201	81.17	80.0	-1.5
Diethylphthalate	2.216	1.348	77.87	80.0	2.7
4-Chlorophenyl-phenylether	1.065	.672	80.73	80.0	-.9
Fluorene	2.011	1.256	79.99	80.0	.0
4-Nitroaniline	.506	.319	74.55	80.0	6.8
4,6-Dinitro-2-methylphenol	.216	.143	85.12	80.0	-6.4
N-Nitrosodiphenylamine (1) *	.880	.575	83.66	80.0	-4.6*
1,2-Diphenylhydrazine	1.592	1.032	82.99	80.0	-3.7
4-Bromophenyl-phenylether	.434	.293	86.30	80.0	-7.9
Hexachlorobenzene	.592	.380	82.25	80.0	-2.8
Pentachlorophenol *	.316	.201	77.16	80.0	3.6*
Phenanthrene	1.802	1.116	79.31	80.0	.9
Anthracene	1.821	1.136	79.86	80.0	.2
Carbazole	1.612	.925	73.44	80.0	8.2
Di-n-butylphthalate	2.031	1.136	71.63	80.0	10.5
Fluoranthene *	1.770	.972	70.30	80.0	12.1*
Benzidine	1.111	.615	283.70	320.0	11.3
Pyrene	2.779	1.520	70.02	80.0	12.5
Butylbenzylphthalate	1.024	.617	72.24	80.0	9.7
3,3'-Dichlorobenzidine	.698	.476	79.95	80.0	.1
Benzo(a)anthracene	1.861	1.148	78.96	80.0	1.3
bis(2-Ethylhexyl)phthalate	1.376	.822	76.45	80.0	4.4
Chrysene	1.817	1.134	79.85	80.0	.2
Di-n-octylphthalate *	3.198	2.097	83.38	80.0	-4.2*
7,12-Dimethylbenz[a]anthrace	1.297	.882	87.11	80.0	-8.9
Benzo(b)fluoranthene	2.664	1.754	84.28	80.0	-5.3
Benzo(k)fluoranthene	2.502	1.558	79.71	80.0	.4

(1) Cannot be separated from Diphenylamine

7C cont

SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name: LANCASTER LABS Contract: _____.

Lab Code: LANCAS Case No.: _____. SAS No.: _____. SDG No.: _____.

Instrument ID: HP03189 Calibration Date: 06/28/95 Time: 13:03

Lab File ID: >V6303 Init. Calib. Date(s): 06/27/95 06/28/95

Min RRF50 for SPCC(#) = 0.050 Max %Drift for CCC(*) = 20

COMPOUND		RRF	RRF80	ACTUAL CONC	TRUE CONC	% DRIFT
Benzo(a)pyrene	*	2.286	1.529	85.57	80.0	-7.0*
Indeno(1,2,3-cd)pyrene		1.687	1.088	82.51	80.0	-3.1
Dibenz(a,h)anthracene		1.669	1.074	82.38	80.0	-3.0
Benzo(g,h,i)perylene		1.597	1.022	81.89	80.0	-2.4
2-Fluorophenol		1.822	1.151	80.89	80.0	-1.1
Phenol-d5		2.139	1.296	77.58	80.0	3.0
Phenol-d6		2.139	1.296	77.58	80.0	3.0
Nitrobenzene-d5		.634	.421	84.95	80.0	-6.2
2-Fluorobiphenyl		2.200	1.367	79.51	80.0	.6
2,4,6-Tribromophenol		.523	.341	83.36	80.0	-4.2
Terphenyl-d14		1.748	.973	71.24	80.0	11.0

(1) Cannot be separated from Diphenylamine

Benzidine level in the 50 standard is 200 ng/uL.

Surrogate Recovery
Volatiles by GC - Water

LLI Sample No.	Sample Designation	Dilution Factor	S1 (MeBrCl)	S2 (1Cl3FBn)	S3 (1Cl3FBn)	S4 (1,2,3-TCP)	S5 (ProBn)	Other	TOT OUT	Comment

	QC Limits LOW	HIGH
S1 (MeBrCl) = Bromochloromethane (Hall Det)	75	125
S2 (1Cl3FBn) = 1-Chloro-3-fluorobenzene (Hall Det)	75	125
S3 (1Cl3FBn) = 1-Chloro-3-fluorobenzene (PID Det)	75	125
S4 (1,2,3-TCP) = 1,2,3-Trichloropropane (Hall Det)	75	125
S5 (ProBn) = n-Propylbenzene (PID Det)	75	125

* Values outside QC limits

D Surrogates diluted out

Nominal concentration of the surrogate spike used is 30 ug/l.

Quality Control Summary

Method Blank
Primary Run
Volatiles by GC

*** BLANK INFORMATION ***
Matrix.....: Water
Batch Number.....: 95108/A15
Injection Number.....: 186
Analysis Date.....: 04/20/95
Concentration Units.....: ug/l

Instrument.....: 05586
Column ID.....: 75m x 0.45mm ID J&W Scientific DB-VRX

Sample Information				Blank Contamination Information			
LLI Sample #	Sample Designation	Analysis Date	Analysis Time	CAS Number	Compound	Blank Result	LOQ
				74-87-3	Chloromethane	ND	5
				74-83-9	Bromomethane	ND	5
				75-01-4	Vinyl chloride	ND	1
				75-00-3	Chloroethane	ND	1
				75-09-2	Methylene chloride	ND	1
				75-69-4	Trichlorofluormethane	ND	1
				75-35-4	1,1-Dichloroethene	ND	1
				75-34-3	1,1-Dichloroethane	ND	1
				540-59-0	1,2-Dichloroethene (c/t)	ND	1
				67-66-3	Chloroform	ND	1
				76-13-1	Trichlorotrifluoroethane	ND	1
				107-06-2	1,2-Dichloroethane	ND	1
				71-55-6	1,1,1-Trichloroethane	ND	1
				56-23-5	Carbon tetrachloride	ND	1
				75-27-4	Bromodichloromethane	ND	1
				78-87-5	1,2-Dichloropropane	ND	1
				10061-02-6	trans-1,3-Dichloropropene	ND	1
				79-01-6	Trichloroethene	ND	1
				124-48-1	Dibromochloromethane	ND	1
				79-00-5	1,1,2-Trichloroethane	ND	1
				10061-01-5	cis-1,3-Dichloropropene	ND	1
				75-25-2	Bromoform	ND	2
				79-34-5	1,1,2,2-Tetrachloroethane	ND	2
				127-18-4	Tetrachloroethene	ND	1
				108-90-7	Chlorobenzene	ND	1
				71-43-2	Benzene	ND	1
				108-88-3	Toluene	ND	1

ABBREVIATION KEY

LOQ = Limit of Quantitation
ND = None Detected
* = above detection limit

Quality Control Summary

Matrix Spike/Matrix Spike Duplicate
Primary Run
Volatiles by GC

Unspiked Sample Number : 2120251 Inj. : 529
Spiked Sample Number : 2120252 Inj. : 530
Spiked Dup Sample Number : 2120253 Inj. : 531

Batch Number : 94124/A12 Date : 05/05/94
Matrix : Water

Instrument.....: 03819
Column.....: 1% SP-1000 on Carbopack B

This MS/MSD applies to the following samples	Compound	Spike Added (ug/l)	Sample Conc (ug/l)	MS Conc (ug/l)	MSD Conc (ug/l)	MS % REC	MSD % REC	QC Limits REC	RPD	QC Limits RPD
	Chloromethane	20.0	ND					25 -168		20
	Bromomethane	20.0	ND					46 -136		20
	Vinyl chloride	20.0	ND					48 -163		20
	Chloroethane	20.0	ND					46 -137		20
	Methylene chloride	19.3	ND					78 -128		15
	1,1-Dichloroethene	18.6	ND					74 -137		15
	1,1-Dichloroethane		ND					91 -130		15
	1,2-Dichloroethene(cis/trans)		ND					92 -126		15
	Chloroform		ND					91 -127		15
	1,2-Dichloroethane		ND					80 -130		15
	1,1,1-Trichloroethane		ND					87 -138		15
	Carbon Tetrachloride		ND					91 -134		15
	Bromodichloromethane		ND					87 -123		15
	1,2-Dichloropropane		ND					87 -128		15
	Trichloroethene		ND					91 -131		15
	Dibromochloromethane		ND					88 -131		15
	Bromoform		ND					74 -119		15
	Tetrachloroethene		ND					91 -129		15
	Chlorobenzene		ND					90 -125		15
	Benzene		ND					93 -124		15
	Toluene		ND					92 -120		15
	Ethylbenzene		ND					94 -119		15

ABBREVIATION KEY
MS = Matrix Spike
MSD = Matrix Spike Duplicate
ND = None Detected
RPD = Relative Percent Difference

Quality Control Summary

Initial Calibration
Primary Run
Volatiles by GC

Calibration Batch.....: 95003/A15

Sample Batch Number.....: 95026/A15

Calibration Date.....: 01/03/95

Instrument.....: 05586
Column ID.....: 75m x 0.45mm ID J&W Scientific DB-VRX

Compound	Laboratory Standard ID: 2.5-500 +12.5 Rf STD 1	4-200 +20 Rf STD 2	10-200 +20 Rf STD 3	10-50 +10 Rf STD 4	35-50 +20 Rf STD 5	70-50 +30 Rf STD 6	AVE RF	%RSD	QC Limit		RT	ID Windc
Chloromethane	0.001411	0.001668	0.001307	0.001949	0.001585	0.001394	0.001553	15.2	20		2.10	+/- 0.3 min
Bromomethane	0.003348	0.003280	0.002448	0.002366	0.002576	0.001980	0.002666	20.3	20	*	3.52	+/- 0.3
Vinyl chloride	0.001178	0.001732	0.001443	0.001537	0.001511	0.001294	0.001449	13.4	20		4.50	+/- 0.3
Chloroethane	0.001072	0.001265	0.001076	0.001064	0.001078	0.001009	0.001094	8.0	20		5.75	+/- 0.3 min
Methylene chloride	0.000695	0.000732	0.000751	0.000729	0.000712	0.000688	0.000718	3.4	20		8.24	+/- 0.2 min
1,1-Dichloroethene	0.001042	0.000907	0.000716	0.000736	0.000684	0.000727	0.000802	17.6	20		11.14	+/- 0.2
1,1-Dichloroethane	0.000989	0.000870	0.000791	0.000740	0.000730	0.000754	0.000812	12.4	20		12.46	+/- 0.2
cis-1,2-Dichloroethene	0.001391	0.001111	0.000886	0.000852	0.000824	0.000784	0.000974	24.0	20	*	13.22	+/- 0.2 min
Chloroform	0.000770	0.000666	0.000611	0.000565	0.000571	0.000570	0.000626	12.9	20		13.78	+/- 0.2
1,2-Dichloroethane	0.001341	0.001096	0.000936	0.000834	0.000825	0.000788	0.000970	22.0	20	*	14.65	+/- 0.2
1,1,1-Trichloroethane	0.001002	0.000982	0.000835	0.000809	0.000798	0.000806	0.000872	10.8	20		15.90	+/- 0.2
Carbon tetrachloride	0.000845	0.000718	0.000621	0.000597	0.000593	0.000584	0.000660	15.7	20		16.30	+/- 0.2 min
Bromodichloromethane	0.001315	0.001202	0.001001	0.000922	0.000893	0.000836	0.001028	18.5	20		16.85	+/- 0.2
1,2-Dichloropropane	0.001566	0.001361	0.000994	0.000985	0.000990	0.000944	0.001140	22.7	20	*	18.30	+/- 0.2
Trichloroethene	0.001007	0.000874	0.000717	0.000705	0.000676	0.000676	0.000776	17.4	20		19.06	+/- 0.2
Dibromochloromethane	0.002343	0.001957	0.001613	0.001590	0.001456	0.001384	0.001724	21.0	20	*	19.81	+/- 0.2 min
Bromoform	0.006067	0.004307	0.003584	0.003356	0.003115	0.002659	0.003848	31.6	20	*	22.61	+/- 0.4
Tetrachloroethene	0.000814	0.000805	0.000619	0.000677	0.000641	0.000646	0.000700	12.3	20		24.73	+/- 0.2
Chlorobenzene	0.003257	0.003025	0.002402	0.002333	0.002347	0.002104	0.002578	17.6	20		27.61	+/- 0.2 min
Benzene	0.026492	0.037147	0.033503	0.035573	0.031104	0.029908	0.032288	12.1	20		19.54	+/- 0.2 min
Toluene	0.038457	0.040149	0.037709	0.040768	0.036457	0.035646	0.038198	5.3	20		26.14	+/- 0.2
Ethylbenzene	0.071343	0.069934	0.070323	0.078276	0.069296	0.067727	0.071150	5.2	20		30.39	+/- 0.2

This initial calibration applies to samples:

2249687 UNSPK	2250495	2250499	Inj #494 BLK
2249688 MS	2250496	2250500	Inj #536 BLK
2249689 MSD	2250497	2250501	Inj #562 BLK
2249690	2250498	2250502	Inj #599 BLK

For initial calibration 01/03/95, the %RSD for bromomethane, cis-1,2-dichloroethene, 1,2-dichloroethene, 1,2-dichloropropane dibromochloromethane and bromoform is outside the QC limit as set by Lancaster Laboratories, Inc. However, EPA Method SW-84 5030A/8010A & 8020 does not specify QC limits for this parameter when a calibration curve is used. In addition, these compou-- were not detected in any of the samples analyzed under this method.

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Quality Control Summary

Continuing Calibration
Primary Run
Volatiles by GC

Calibration Date.....: 04/12/95
Batch Number.....: 95133/A12
Continuing Calibration Date...: 05/16/95
Inj #.....: 766

Instrument.....: 03819
Column.....: 1% SP-1000 on Carbopack B

	Compound	Amount Spiked	Laboratory Control Sample Result	% Recovery	Acceptance Range	Out of Range
2308195	Chloromethane	20.0	25.7	128.4	60 % - 141 %	
Inj #765 BLK	Bromomethane	20.0	20.4	102.1	59 % - 142 %	
	Vinyl chloride	20.0	22.7	113.4	69 % - 132 %	
	Chloroethane	20.0	22.3	111.4	77 % - 123 %	
	Methylene chloride	20.1	22.1	110.1	78 % - 123 %	
	Trichlorofluoromethane	20.1	20.6	102.7	67 % - 134 %	
	1,1-Dichloroethene	20.1	20.7	102.8	63 % - 137 %	
	1,1-Dichloroethane	20.1	23.3	116.1	84 % - 116 %	*
	1,2-Dichloroethene (c/t)	20.1	20.7	102.9	64 % - 136 %	
	Chloroform	20.1	20.7	103.0	75 % - 125 %	
	1,2-Dichloroethane	20.2	18.1	89.5	72 % - 129 %	
	1,1,1-Trichloroethane	20.0	21.5	107.5	71 % - 129 %	
	Carbon tetrachloride	20.1	22.8	113.3	69 % - 132 %	
	Bromodichloromethane	20.0	19.8	98.8	76 % - 124 %	
	1,2-Dichloropropane	20.1	20.2	100.3	74 % - 126 %	
	Trichloroethene	20.1	21.4	106.6	77 % - 123 %	
	Dibromochloromethane	20.1	18.8	93.6	66 % - 135 %	
	2-Chloroethyl vinyl ether	20.1	22.4	111.4	60 % - 140 %	
	Bromoform	20.1	19.8	98.7	74 % - 127 %	
	Tetrachloroethene	20.1	21.0	104.5	70 % - 130 %	
	Chlorobenzene	20.1	21.0	104.5	72 % - 128 %	

Check Standard Summary
Retention Time
Primary Run
Volatiles by GC - Water

Initial Calibration Date.....: 01/03/95
Sample Batch.....: 95061/A01
Injection Number.....: 056
Injection Date.....: 03/02/95
Method.....: EPA Method 601

Instrument.....: 02030
Column.....: 1% SP-1000 on Carbopack B

Sample Number	Compound	Retention Time	ID Window
2265581 UNSPK	Chloromethane	2.16	+/- 0.3 min
2265584 MS	Bromomethane	3.59	+/- 0.3 min
2265585 MSD	Vinyl chloride	4.62	+/- 0.3 min
Inj #063 BLK	Chloroethane	5.87	+/- 0.3 min
	Methylene chloride	8.29	+/- 0.2 min
	Trichlorofluoromethane	10.47	+/- 0.2 min
	1,1-Dichloroethene	11.18	+/- 0.2 min
	1,1-Dichloroethane	12.48	+/- 0.2 min
	cis-1,2-Dichloroethene	13.26	+/- 0.2 min
	Chloroform	13.79	+/- 0.2 min
	1,2-Dichloroethane	14.71	+/- 0.2 min
	1,1,1-Trichloroethane	15.92	+/- 0.2 min
	Carbon tetrachloride	16.32	+/- 0.2 min
	Bromodichloromethane	16.95	+/- 0.2 min
	1,2-Dichloropropane	18.44	+/- 0.2 min
	Trichloroethene	19.13	+/- 0.2 min
	Dibromochloromethane	19.92	+/- 0.2 min
	2-Chloroethyl vinyl ether	21.16	+/- 0.2 min
	Bromoform	22.62	+/- 0.4 min
	Tetrachloroethene	24.72	+/- 0.2 min
	Chlorobenzene	27.62	+/- 0.2 min

Retention Time Shift Summary
Primary Run
Volatiles by GC - Water

Initial Calibration Date .....: 01/03/95
Initial Calibration Batch.....: 95003/A01
Sample Batch.....: 95003/A01
Method .....: EPA Method SW-846 5030A/8010A

Instrument.....: 02030
Column.....: 1% SP-1000 on Carbopack B

SURROGATE RT FROM INITIAL CALIBRATION
MeBrCl: 11.62 1Cl3FBn (Hall): 28.76

LLI Sample No.	Sample Designation	Date Analyzed	Time Analyzed	RT (MeBrCl)	RT (1Cl3FBn) Hall Det.

(MeBrCl) = Bromochloromethane (Hall Det)
(1Cl3FBn) = 1-Chloro-3-fluorobenzene (Hall Det)

Matrix: WATER

LLI Sample No.	Sample Code	S1 (DCB)	S2 (TCX)	S3 (OXY)	S4 (DCAA)	OTHER
BLK6/9	BLK6/9	86	57 *			
LCS6/9	LCS6/9	100	71			
LCSD6/9	LCSD6/9	55 *	77			
BLK6/12	BLK6/12	83	95			
2326074	WPMW2	60	74			

		QC REC Limits Low	High
S1 (DCB)	Decachlorobiphenyl	60	120
S2 (TCX)	Tetrachlorometaxylene	60	120
S3 (OXY)	Oxychlordane		
S4 (DCAA)	2,4-Dichlorophenylacetic Acid		
S5 OTHER			

* = Surrogate Recovery is outside specifications.
# = No established limits
D = Surrogates diluted out I = Interferences present

Comments:

Matrix..: WATER

Sample Information		Blank Contamination Information					
LLI Sample No.	Sample Code	CAS Number	Compound	Analysis Date	Blank Result	Units	LOQ
BLK6/9	BLK6/9	319-84-6	alpha-BHC	06/14/95	ND	ug/l	0.01
LCS6/9	LCS6/9	319-85-7	beta-BHC	06/14/95	ND	ug/l	0.01
LCSD6/9	LCSD6/9	319-86-8	delta-BHC	06/14/95	ND	ug/l	0.01
		58-89-9	gamma-BHC (Lindane)	06/14/95	ND	ug/l	0.01
		76-44-8	Heptachlor	06/14/95	ND	ug/l	0.01
		309-00-2	Aldrin	06/14/95	ND	ug/l	0.01
		1024-57-3	Heptachlor epoxide	06/14/95	ND	ug/l	0.01
		959-98-8	Endosulfan I	06/14/95	ND	ug/l	0.01
		60-57-1	Dieldrin	06/14/95	ND	ug/l	0.01
		72-55-9	4,4'-DDE	06/14/95	ND	ug/l	0.01
		72-20-8	Endrin	06/14/95	ND	ug/l	0.01
		33213-65-9	Endosulfan II	06/14/95	ND	ug/l	0.01
		72-54-8	4,4'-DDD	06/14/95	ND	ug/l	0.01
		1031-07-8	Endosulfan sulfate	06/14/95	ND	ug/l	0.03
		50-29-3	4,4'-DDT	06/14/95	ND	ug/l	0.01
		72-43-5	Methoxychlor	06/14/95	ND	ug/l	0.05
		53494-70-5	Endrin ketone	06/14/95	ND	ug/l	0.1
		5103-71-9	alpha-Chlordane	06/14/95	ND	ug/l	0.01
		5103-74-2	gamma-Chlordane	06/14/95	ND	ug/l	0.01
		8001-35-2	Toxaphene	06/14/95	ND	ug/l	4
		12674-11-2	PCB-1016	06/14/95	ND	ug/l	1
		11104-28-2	PCB-1221	06/14/95	ND	ug/l	1
		11141-16-5	PCB-1232	06/14/95	ND	ug/l	1
		53469-21-9	PCB-1242	06/14/95	ND	ug/l	1
		12672-29-6	PCB-1248	06/14/95	ND	ug/l	1
		11097-69-1	PCB-1254	06/14/95	ND	ug/l	1
		11096-82-5	PCB-1260	06/14/95	ND	ug/l	1
		7421-39-4	Endrin aldehyde	06/14/95	ND	ug/l	0.1
		12789-03-6	Technical Chlordane	06/14/95	ND	ug/l	0.3

COMMENTS:

Abbreviation Key
--- = Analysis not requested
ND = None detected
J = Estimated value below LOQ
LOQ = Limit of Quantitation
* = Outside Specifications

Method Blank
Pesticides

Matrix..: WATER

Sample Information		Blank Contamination Information					
LLI Sample No.	Sample Code	CAS Number	Compound	Analysis Date	Blank Result	Units	LOQ
BLK6/12	BLK6/12	319-84-6	alpha-BHC	06/16/95	ND	ug/l	0.01
2326074	WPMW2	319-85-7	beta-BHC	06/16/95	ND	ug/l	0.01
		319-86-8	delta-BHC	06/16/95	ND	ug/l	0.01
		58-89-9	gamma-BHC (Lindane)	06/16/95	ND	ug/l	0.01
		76-44-8	Heptachlor	06/16/95	ND*	ug/l	0.01
		309-00-2	Aldrin	06/16/95	ND	ug/l	0.01
		1024-57-3	Heptachlor epoxide	06/16/95	ND	ug/l	0.01
		959-98-8	Endosulfan I	06/16/95	ND	ug/l	0.01
		60-57-1	Dieldrin	06/16/95	ND	ug/l	0.01
		72-55-9	4,4'-DDE	06/16/95	ND	ug/l	0.01
		72-20-8	Endrin	06/16/95	ND	ug/l	0.01
		33213-65-9	Endosulfan II	06/16/95	ND	ug/l	0.01
		72-54-8	4,4'-DDD	06/16/95	ND	ug/l	0.01
		1031-07-8	Endosulfan sulfate	06/16/95	ND	ug/l	0.03
		50-29-3	4,4'-DDT	06/16/95	ND	ug/l	0.01
		72-43-5	Methoxychlor	06/16/95	ND	ug/l	0.05
		53494-70-5	Endrin ketone	06/16/95	ND	ug/l	0.1
		5103-71-9	alpha-Chlordane	06/16/95	ND	ug/l	0.01
		5103-74-2	gamma-Chlordane	06/16/95	ND	ug/l	0.01
		8001-35-2	Toxaphene	06/16/95	ND	ug/l	4
		12674-11-2	PCB-1016	06/16/95	ND	ug/l	1
		11104-28-2	PCB-1221	06/16/95	ND	ug/l	1
		11141-16-5	PCB-1232	06/16/95	ND	ug/l	1
		53469-21-9	PCB-1242	06/16/95	ND	ug/l	1
		12672-29-6	PCB-1248	06/16/95	ND	ug/l	1
		11097-69-1	PCB-1254	06/16/95	ND	ug/l	1
		11096-82-5	PCB-1260	06/16/95	ND	ug/l	1
		7421-39-4	Endrin aldehyde	06/16/95	ND	ug/l	0.1
		12789-03-6	Technical Chlordane	06/16/95	ND	ug/l	0.3

COMMENTS:

Abbreviation Key
--- = Analysis not requested
ND = None detected
J = Estimated value below LOQ
LOQ = Limit of Quantitation
* = Outside Specifications

Lancaster Laboratories
Where quality is a science.

Quality Control Summary

Matrix Spike/Matrix Spike Duplicate
Pesticides

Unspiked Sample #....:2331041BKGD
Spiked Sample #.....:2331041MS
Spiked Dup Sample #..:2331041MSD

Matrix: water

This MS/MSD applies to the following samples	Compound	Spike Added (ug/l)	Sample Conc (ug/l)	MS Conc (ug/l)	MSD Conc (ug/l)	MS % REC	MSD % REC	QC Limits REC	RPD	QC Limits RPD
1322BLK6/22	alpha-BHC	0.200	ND	0.207	0.194	103	97	80 -132	6	30
2331034	beta-BHC	0.200	ND	0.204	0.200	102	100	74 -120	2	30
2331035	delta-BHC	0.200	ND	0.166	0.173	83	86	76 -126	4	30
2331038	gamma-BHC (Lindane)	0.200	ND	0.201	0.195	101	98	66 -120	3	30
2331039	Heptachlor	0.200	ND	0.190	0.186	95	93	60 -120	2	30
2331040	Aldrin	0.200	ND	0.142	0.141	71	70	58 -120	1	30
2331041BKGD	Heptachlor epoxide	0.200	ND	0.194	0.186	97	93	64 -120	4	30
2331041MS	Endosulfan I	0.200	ND	0.199	0.193	100	97	66 -120	3	30
2331041MSD	Dieldrin	0.200	ND	0.203	0.195	102	98	83 -120	4	30
	4,4'-DDE	0.200	ND	0.206	0.201	103	101	74 -120	2	30
	Endrin	0.200	ND	0.239	0.240	119	120	76 -120	0	30
	Endosulfan II	0.200	ND	0.212	0.212	106	106	67 -120	0	30
	4,4'-DDD	0.200	ND	0.227	0.228	114	114	75 -126	0	30
	Endosulfan sulfate	0.200	ND	0.213	0.210	106	105	74 -120	1	30
	4,4'-DDT	0.200	ND	0.204	0.204	102	102	71 -120	0	30
	Methoxychlor	0.200	ND	0.216	0.231	108	116	63 -120	7	30
	Endrin aldehyde	0.200	ND	0.209	0.208	104	104	68 -120	0	30
	Kepone	10.090	ND	2.937	3.053	29	30	22 -120	4	30

ABBREVIATION KEY
MS = Matrix Spike
MSD = Matrix Spike Duplicate
ND = None Detected
RPD = Relative Percent Difference
--- = Analysis not requested
= No established limits
* = Outside specifications
D = Detection Limit

COMMENTS:

Lancaster Laboratories
Where quality is a science.

Quality Control Summary

Lab Control Spike/Lab Control Spike Duplica
Pesticides

Unspiked Sample #....:BLK6/9
Spiked Sample #.....:LCS6/9
Spiked Dup Sample #..:LCSD6/9

Matrix: WATER

This LCS/LCSD applies to the following samples	Compound	Spike Added (ug/l)	BKGD Conc (ug/l)	LCS Conc (ug/l)	LCSD Conc (ug/l)	LCS % REC	LCSD % REC	QC Limits REC	RPD	QC Lf
BLK6/9	alpha-BHC	0.205	ND	0.169	0.183	82	89	80 -132	8	30
LCS6/9	gamma-BHC	0.202	ND	0.169	0.175	84	87	66 -120	3	0
LCSD6/9	beta-BHC	0.192	ND	0.179	0.190	93	99	74 -120	6	0
BLK6/12	Heptachlor	0.182	ND	0.153	0.158	84	87	60 -120	3	30
2326074	delta-BHC	0.186	ND	0.166	0.178	89	96	76 -126	7	30
	Aldrin	0.192	ND	0.154	0.159	80	83	58 -120	3	0
	Heptachlor epoxide	0.196	ND	0.169	0.164	86	84	64 -120	3	0
	Endosulfan I	0.200	ND	0.190	0.196	95	98	66 -120	3	30
	4,4'-DDE	0.211	ND	0.180	0.186	85	88	74 -120	3	30
	Dieldrin	0.202	ND	0.176	0.181	87	90	83 -120	3	0
	Endrin	0.232	ND	0.219	0.228	94	98	76 -120	4	0
	4,4'-DDD	0.233	ND	0.199	0.201	85	86	75 -126	1	30
	Endosulfan II	0.199	ND	0.184	0.192	92	96	67 -120	4	30
	4,4'-DDT	0.201	ND	0.192	0.191	96	95	71 -120	1	0
	Endrin aldehyde	0.224	ND	0.185	0.200	83	89	68 -120	8	0
	Endosulfan sulfate	0.215	ND	0.194	0.209	90	97	74 -120	7	30
	Methoxychlor	0.242	ND	0.203	0.219	84	90	63 -120	8	30

ABBREVIATION KEY

LCS = Lab Control Spike LCSD = Lab Control Spike Duplicate
ND = None Detected --- = Analysis not requested
RPD = Relative Percent Difference
# = No established limits * = Outside Specifications

COMMENTS:

QUALITY ASSURANCE SUMMARY

COVER PAGE - INORGANIC ANALYSES DATA PACKAGE

Lab Name: LANCASTER_LABORATORIES_____

SDG No.: TEST___

Client Sample ID	Lab Sample ID

Were ICP interelement corrections applied ? Yes/No YES

Were ICP background corrections applied ? Yes/No YES

If yes - were raw data generated before application of background corrections ? Yes/No NO_

LEGEND:

FLAGS: (indicate matrix interference)
- N = Matrix Spike OOS
- * = Duplicate OOS
- W = Method F Analytical Spike Recovery <85% or >115% when the sample conc. is <50% of the spike conc.
- S = Analysis Determined by MSA
- + = MSA Correlation Coefficient <0.995
- E = Matrix Effects exist as proven by Serial Dilution or Spiked Dilution

Presence of FLAGS does not invalidate data

U = Below MDL B = Below LOQ

METHODS:
- A = Flame Atomic Absorption
- P = Inductively Coupled Plasma
- F = Graphite Furnace
- AS = Hydride Generation
- CV = Cold Vapor
- NR = Not Required

TERMS:
- MDL = Method Detection Limit
- LOQ = Limit Of Quantitation
- OOS = Out Of Specification
- MSA = Method Of Standard Addition

I certify that this data package is in full compliance with the terms and conditions as specified by the NJDEP, both technically and for completeness, other than the conditions detailed above. Release of the data contained in this data package has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature:_____ Name:_____

Date:_____ Title:_____

QUALITY ASSURANCE SUMMARY

INORGANIC ANALYSES DATA SHEET

CLIENT SAMPLE NO.

Lab Name: LANCASTER_LABORATORIES
SDG No.: TEST
Matrix (soil/water): WATER
Level (low/med): LOW
% Solids: 0

Lab Sample ID:
Date Received: 10/19/92

Concentration Units (ug/L or mg/kg dry weight): UG/L

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum				NR
7440-36-0	Antimony				NR
7440-38-2	Arsenic				NR
7440-39-3	Barium				NR
7440-41-7	Beryllium				NR
	Boron				NR
7440-43-9	Cadmium				NR
7440-70-2	Calcium				NR
7440-47-3	Chromium				NR
7440-48-4	Cobalt				NR
7440-50-8	Copper				NR
7439-89-6	Iron				NR
7439-92-1	Lead				P
	Lithium				NR
7439-95-4	Magnesium				NR
7439-96-5	Manganese				NR
7439-97-6	Mercury				NR
	Molybdenu				NR
7440-02-0	Nickel				NR
7440-09-7	Potassium				NR
7782-49-2	Selenium				NR
	Silicon				NR
7440-22-4	Silver				NR
7440-23-5	Sodium				NR
	Strontium				NR
7440-28-0	Thallium				NR
	Tin				NR
	Titanium				NR
7440-62-2	Vanadium				NR
7440-66-6	Zinc				NR

Color Before:
Color After:
Clarity Before:
Clarity After:
Texture:
Artifacts:

Comments:

QUALITY ASSURANCE SUMMARY

BLANKS

Lab Name: LANCASTER_LABORATORIES_____

SDG No.: TEST__

Preparation Blank Matrix (soil/water): _____

Preparation Blank Concentration Units (ug/L or mg/kg): _____

Analyte	Initial Calib. Blank (ug/L)	C	Continuing Calibration Blank (ug/L) 1	C	2	C	3	C	Prepa-ration Blank	C	M
Aluminum											NR
Antimony											NR
Arsenic											NR
Barium											NR
Beryllium											NR
Boron											NR
Cadmium											NR
Calcium											NR
Chromium											NR
Cobalt											NR
Copper											NR
Iron											NR
Lead											P
Lithium											NR
Magnesium											NR
Manganese											NR
Mercury											NR
Molybdenu											NR
Nickel											NR
Potassium											NR
Selenium											NR
Silicon											NR
Silver											NR
Sodium											NR
Strontium											NR
Thallium											NR
Tin											NR
Titanium											NR
Vanadium											NR
Zinc											NR

QUALITY ASSURANCE SUMMARY

SPIKE SAMPLE RECOVERY

CLIENT SAMPLE NO.

Lab Name: LANCASTER_LABORATORIES_____

SDG No.: TEST
Matrix: WATER
% Solids for Sample: __0.0

Level (low/med): LOW

Concentration Units (ug/L or mg/kg dry weight): UG/L_

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									NR
Antimony									NR
Arsenic									NR
Barium									NR
Beryllium									NR
Boron									NR
Cadmium									NR
Calcium									NR
Chromium									NR
Cobalt									NR
Copper									NR
Iron									NR
Lead	75-125	28.0000		28.0000		2000.00	0.0	N	P
Lithium									NR
Magnesium									NR
Manganese									NR
Mercury									NR
Molybdenu									NR
Nickel									NR
Potassium									NR
Selenium									NR
Silicon									NR
Silver									NR
Sodium									NR
Strontium									NR
Thallium									NR
Tin									NR
Titanium									NR
Vanadium									NR
Zinc									NR

NOTE: An (N) in column "Q" indicates a spike recovery that is not within the control limits. The data are considered to be valid because the laboratory control sample is within the control limits. See the Laboratory Control Sample page of the Quality Assurance Summary.

Comments:

QUALITY ASSURANCE SUMMARY

DUPLICATES

CLIENT SAMPLE NO.

Lab Name: LANCASTER_LABORATORIES

SDG No.: TEST

Matrix (soil/water): WATER

Level (low/med): LOW

% Solids for Sample: 0

% Solids for Duplicate: 0

Concentration Units (ug/L or mg/kg dry weight): UG/L

Analyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	M
Aluminum								NR
Antimony								NR
Arsenic								NR
Barium								NR
Beryllium								NR
Boron								NR
Cadmium								NR
Calcium								NR
Chromium								NR
Cobalt								NR
Copper								NR
Iron								NR
Lead								P
Lithium								NR
Magnesium								NR
Manganese								NR
Mercury								NR
Molybdenu								NR
Nickel								NR
Potassium								NR
Selenium								NR
Silicon								NR
Silver								NR
Sodium								NR
Strontium								NR
Thallium								NR
Tin								NR
Titanium								NR
Vanadium								NR
Zinc								NR

NOTE: An asterisk(*) in column "Q" indicates poor duplicate precision. The data are considered to be valid because the laboratory control sample is within the control limits. See the Laboratory Control Sample page of the Quality Assurance Summary.

QUALITY ASSURANCE SUMMARY

STANDARD ADDITION RESULTS

Lab Name: LANCASTER_LABORATORIES_________

SDG No.: TEST__

Concentration Units: ug/L

EPA Sample No.	An	0 ADD	1 ADD		2 ADD		3 ADD		Final Conc.	r	Q
		ABS	CON	ABS	CON	ABS	CON	ABS			

ICP SERIAL DILUTIONS

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix (soil/water): _____ Level (low/med): _____

Concentration Units: ug/L

Analyte	Initial Sample Result (I)	C	Serial Dilution Result (S)	C	% Difference	Q	M
Aluminum							
Antimony							
Arsenic							
Barium							
Beryllium							
Cadmium							
Calcium							
Chromium							
Cobalt							
Copper							
Iron							
Lead							
Magnesium							
Manganese							
Mercury							
Nickel							
Potassium							
Selenium							
Silver							
Sodium							
Thallium							
Vanadium							
Zinc							

QUALITY ASSURANCE SUMMARY

LABORATORY CONTROL SAMPLE

ab Name: LANCASTER_LABORATORIES_____

DG No.: TEST__

olid LCS Source: _____

queous LCS Source: LLI_____

Analyte	Aqueous (ug/L) True	Found	%R(1)	Solid (mg/kg) True	Found	C	Control Limits		%R
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Boron									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Lithium									
Magnesium									
Manganese									
Mercury									
Molybdenu									
Nickel									
Potassium									
Selenium									
Silicon									
Silver									
Sodium									
Strontium									
Thallium									
Tin									
Titanium									
Vanadium									
Zinc									

(1) Control Limits: All Metals 80-120

ICP INTERFERENCE CHECK SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

ICP ID Number: _____ ICS Source: _____

Concentration Units: ug/L

Analyte	True Sol. A	True Sol. AB	Initial Found Sol. A	Initial Found Sol. AB	Initial Found %R	Final Found Sol. A	Final Found Sol. AB	Final Found %R
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								

QUALITY ASSURANCE SUMMARY

INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: LANCASTER_LABORATORIES_____

SDG No.: TEST__

Initial Calibration Source: LLI_____

Continuing Calibration Source: LLI_____

Concentration Units: ug/L

Analyte	Initial Calibration True	Found	%R(1)	Continuing Calibration True	Found	%R(1)	Found	%R(1)	M
Aluminum									I
Antimony									N
Arsenic									NR
Barium									I
Beryllium									I
Boron									NR
Cadmium									NR
Calcium									I
Chromium									I
Cobalt									NR
Copper									I
Iron									I
Lead	20.0	1.00	5.0	20.0	1.00	5.0			I
Lithium									NR
Magnesium									I
Manganese									I
Mercury									I
Molybdenu									NR
Nickel									I
Potassium									I
Selenium									I
Silicon									NR
Silver									I
Sodium									I
Strontium									NR
Thallium									NR
Tin									
Titanium									
Vanadium									NR
Zinc									NR

(1) Control Limits: Mercury 80-120; Other Metals 90-110; Cyanide 85-115

QUALITY ASSURANCE SUMMARY

Method Detection Limits (Annually)

Lab Name: LANCASTER_LABORATORIES
SDG No.: TEST
ICP Method No.:
Other AA Method No.:
Furnace AA Method No.: GF_1,2,3_AQUEOU

Date: 01/15/92

Analyte	Wave-length (nm)	Back-ground	LOQ ** (ug/L)	MDL (ug/L)	M
Aluminum			200		NR
Antimony			200		NR
Arsenic			5		NR
Barium			100		NR
Beryllium			10		NR
Boron			40		NR
Cadmium			10		NR
Calcium			200		NR
Chromium			50		NR
Cobalt			50		NR
Copper			20		NR
Iron			100		NR
Lead	283.30	BD	3	1.0	F
Lithium			20		NR
Magnesium			100		NR
Manganese			10		NR
Mercury			0.2		NR
Molybdenu			100		NR
Nickel			50		NR
Potassium			500		NR
Selenium			3		NR
Silicon			300		NR
Silver			20		NR
Sodium			400		NR
Strontium			10		NR
Thallium			10		NR
Tin			300		NR
Titanium			10		NR
Vanadium			10		NR
Zinc			40		NR

** The LOQ must be adjusted for % Solids and Sample Weight for samples reporting in mg/Kg.

Comments:

PREPARATION LOG

Lab Name: LANCASTER_LABORATORIES_________

SDG No.:_TEST__

Method: P_

Client Sample No.	* Preparation Date	Weight (gram)	Volume (mL)

ANALYSIS RUN LOG

Lab Name: ____________________ Contract: __________

Lab Code: ______ Case No.: ______ SAS No.: ______ SDG No.: ______

Instrument ID Number: ____________ Method: __

Start Date: ________ End Date: ________

EPA Sample No.	D/F	Time	% R	Analytes																							
				AL	SB	AS	BA	BE	CD	CA	CR	CO	CU	FE	PB	MG	MN	HG	NI	K	SE	AG	NA	TL	V	ZN	CN

Method Blank Analysis — Matrix: SOIL

Method Blank Designation	LLI Sample No.	Client Designation	Method	Analysis Date	Batch Number	Blank Result	Units	LOQ
BLANK	2302108	Q17T1	TOC	5/04/95	95118-201	< LOQ	mg/kg	50
	2302109	Q17T2						
	2302110	Q17T3						
	2302111	Q17T4						
	2302112	Q16T2						
	2302113	Q16T3						
	2302114	Q16T4						
	2302115	Q16T5						
	2302116	Q1652						
	2302108	SPK, DUP						
BLANK	2303628	S20-8	TOC	5/04/95	95122-201	< LOQ	mg/kg	50
	2303629	S20-9						
	2303630	S2010						
	2303631	S2011						
	2303633	S19S5						
BLANK	2303632	R19S4	TOC	5/18/95	95122-201 *	< LOQ	mg/kg	50
	2303634	S19S4						
	2303635	S1942						

Comments: The blank is acceptable when the result is less than the limit of quantitation.

* The blank prepped with the repeated samples retains the original batch number.

ABBREVIATION KEY

IC = Ion Chromatography	--- = Analysis Not Requested
D = Distillation	ND = Not Detected
TOC = Total Organic Carbon	AK = AlpKem
TOX = Total Organic Halogens	LOQ = Limit of Quantitation
* = Out of Specification	NA = Not Applicable

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Quality Control Summary

~~Matrix Spike Analysis~~

Instrumental Analysis

Sample Information		Matrix Spike Analysis										Matrix: SOIL	
LLI Sample No.	Client Designation	Parameter	Meth	Analysis Date	Unspiked Desig.	Unspiked Result	LOQ	Spiked Desig.	Spike Added	Spiked Result	Units	%REC	
2302108	Q17T1	Total											
2302109	Q17T2	Organic											
2302110	Q17T3	Carbon	TOC	5/04/95	BKG	33056	4000	SPIKE	17857	51843	mg/kg	105.2	
2302111	Q17T4												
2302112	Q16T2												
2302113	Q16T3												
2302114	Q16T4												
2302115	Q16T5												
2302116	Q1652												
2303628	S20-8												
2303629	S20-9												
2303630	S2010												
2303631	S2011												
2303632	S19S4												
2303633	S19S5												
2303634	S19S4												
2303635	S1942												

% Recovery Control Limit 75

% Recovery Control Limit 125

Comments:

ABBREVIATION KEY	
IC = Ion Chromatography	--- = Analysis Not Requested
D = Distillation	ND = Not Detected
TOC = Total Organic Carbon	AK = AlpKem
TOX = Total Organic Halogens	LOQ = Limit of Quantitation
* = Out of Specification	NA = Not Applicable

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Quality Control Summary

~~Duplicate Analysis~~

Instrumental Analysis

Duplicate Analysis — Matrix: SOIL

LLI Sample No.	Client Designation	Parameter	Meth	Analysis Date	1st Dup Desig.	1st Dup Result	LOQ	2nd Dup Desig.	2nd Dup Result	Units	RPD (%)	Control Limit %
2302108	Q17T1	Total Organic Carbon	TOC	5/04/95	BKG	32439	4000	DUP	33673	mg/kg	3.7	35
2302109	Q17T2											
2302110	Q17T3											
2302111	Q17T4											
2302112	Q16T2											
2302113	Q16T3											
2302114	Q16T4											
2302115	Q16T5											
2302116	Q1652											
2303628	S20-8											
2303629	S20-9											
2303630	S2010											
2303631	S2011											
2303632	S19S4											
2303633	S19S5											
2303634	S19S4											
2303635	S1942											

Comments: If one or more sample values are less than the limit of quantitation, the RPD is not calculated.

ABBREVIATION KEY

IC = Ion Chromatography	--- = Analysis Not Requested
D = Distillation	ND = Not Detected
TOC = Total Organic Carbon	AK = AlpKem
TOX = Total Organic Halogens	LOQ = Limit of Quantitation
* = Out of Specification	NA = Not Applicable

Sample Information		Laboratory Control Standard							Matrix: SOIL
LLI Sample No.	Client Designation	Parameter	Meth	Analysis Date	True LCS Value	LCS Result	LOQ	Units	%REC
2302108	Q17T1	Total							
2302109	Q17T2	Organic							
2302110	Q17T3	Carbon	TOC	5/04/95	25	26.74	50	mg/kg	107.0
2302111	Q17T4	RPD= 5.1%	TOC	5/04/95	25	28.14	50	mg/kg	112.6
2302112	Q16T2								
2302113	Q16T3								
2302114	Q16T4	RPD= 1.7%	TOC	5/04/95	25	26.66	50	mg/kg	106.6
2302115	Q16T5		TOC	5/04/95	25	26.23	50	mg/kg	104.9
2302116	Q1652								
2303628	S20-8	RPD= 3.6%	TOC	5/04/95	25	26.66	50	mg/kg	106.6
2303629	S20-9		TOC	5/04/95	25	26.23	50	mg/kg	104.9
2303630	S2010								
2303631	S2011								
2303632	S19S4								
2303633	S19S5								
2303634	S19S4								
2303635	S1942								

Comments: The recovery range for the LCS is +/- 20%.

ABBREVIATION KEY

IC = Ion Chromatography	--- = Analysis Not Requested
D = Distillation	ND = Not Detected
TOC = Total Organic Carbon	AK = AlpKem
TOX = Total Organic Halogens	LOQ = Limit of Quantitation
* = Out of Specification	NA = Not Applicable

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Quality Control Summary
Initial Calibration & Linearity Check
~~Instrumental Analysis~~
TOTAL ORGANIC CARBON
Instrument Identification: 5214
Calibration Date: 5/04/95
MATRIX: SOIL

Std mass: 25.0000 ug
Scaling Factor: 0.04080 Cl mV

Batch Numbers: 95118-201

Blank:	41.1680 mv	Standard:	624.385 mv
Blank:	30.9270 mv	Standard:	610.960 mv
Blank:	41.3370 mv	Standard:	602.709 mv
Average:	37.8107 mv	Average:	612.685 mv

This IC applies to samples:	Client Designation	Analysis #	Method Blank	ICV/ 5	ICV/ 10	ICV/ 25	ICV/ 50	ICV/ 75	Units= ug
2302108	Q17T1	138	0.238	4.954	10.441	25.448	51.842	74.583	
2302109	Q17T2								
2302110	Q17T3								
2302111	Q17T4								
2302112	Q16T2								
2302113	Q16T3								
2302114	Q16T4								
2302115	Q16T5								
2302116	Q1652								
2302108	SPK, DUP								
BLANK									
LCSS									
LCSSD									
2303628	S20-8								
2303629	S20-9								
2303630	S2010								
2303631	S2011								
2303633	S19S5								
BLANK									
LCSS									
LCSSD									

Continuing Calibration Dates: 5/04/95
Concentration units: mg/l

Parameter TOC	Reference Concentration	Result Cont. Cal	% Rec.	Acceptance Range -/+ 10%		Out of Specification
CCV	25.0	25.623	102.5	22.5	27.5	
CCV	25.0	25.250	101.0	22.5	27.5	
CCV	25.0	26.023	104.1	22.5	27.5	
CCV	25.0	26.262	105.0	22.5	27.5	

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Quality Control Summary

Method Blank
Miscellaneous Wet Chemistry

Method Blank Analysis — Matrix:

Method Blank Designation	LL Sample No.	Sample Code	Method	Analysis Date	Batch Number	Blank Result	Units	LOQ

Comments: The blank is acceptable when the result is less than the limit of quantitation.

ABBREVIATION KEY

TI = Titration	ND = Not Detected
CO = Colorimetric	J = Estimated Value < LOQ
IR = Infrared Spectrophotometry	< = Less Than
OD = Oven Dried	LOQ = Limit of Quantitation
DI = Distillation	NA = Not Applicable
G = Gravimetric	M = Meter
U = Under Method Detection Limit	* = Out of Specification

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Quality Control Summary

Matrix Spike Analysis
Miscellaneous Wet Chemistry

Sample Information		Matrix Spike Analysis											Matrix:		
LL Sample No.	Sample Code	Parameter	Meth	Analysis Date	Unspiked Desig.	Unspiked Result	LOQ	Spiked Desig.	Spike Added	Spiked Result	Units	Rec (%)	Accept Window (%)

Comments: Sample values shown may be rounded to be consistent with the limit of quantitation.

ABBREVIATION KEY

TI = Titration	ND = Not Detected
CO = Colorimetric	J = Estimated Value < LOQ
IR = Infrared Spectrophotometry	< = Less Than
OD = Oven Dried	LOQ = Limit of Quantitation
DI = Distillation	NA = Not Applicable
G = Gravimetric	M = Meter
U = Under Method Detection Limit	* = Out of Specification

Duplicate Analysis
Miscellaneous Wet Chemistry

Sample Information		Duplicate Analysis										Matrix:	
LL Sample No.	Sample Code	Parameter	Meth	Analysis Date	1st Dup Desig.	1st Dup Result	LOQ	2nd Dup Desig.	2nd Dup Result	Units	RPD (%)	Control Limit % </=

Comments: If one or more sample values are less than the limit of quantitation, the RPD is not required.

Sample values shown may be rounded to be consistent with the limit of quantitation.

ABBREVIATION KEY

TI = Titration	ND = Not Detected
CO = Colorimetric	J = Estimated Value < LOQ
IR = Infrared Spectrophotometry	< = Less Than
OD = Oven Dried	LOQ = Limit of Quantitation
DI = Distillation	NA = Not Applicable
G = Gravimetric	M = Meter
U = Under Method Detection Limit	* = Out of Specification

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Quality Control Summary

Laboratory Control Standard
Laboratory Control Standard Duplicate
Miscellaneous Wet Chemistry

Sample Information		Laboratory Control Standards									Matrix:	
LL Sample No.	Sample Code	Parameter	Analysis Date	True LCS/D Value	LCS Result	LCSD Result	LOQ	Units	LCS/D Acceptance Range	% RPD Result	% RPD Accept < /	e

Comments: LCS/LCSD values shown may be rounded to be consistent with the limit of quantitation.

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Quality Control Summary

Initial Calibration
Miscellaneous Wet Chemistry
Instrument Identification:

Calibration Date:

Batch Number:

This IC applies to samples:	Sample Code	Parameter *	Blank 0.0000	STD 1 0.1000	STD 2 0.2000	STD 3 0.6000	STD 4 1.0000	STD 5 2.0000	STD 6 4.0000	Corr. Coef.

Calibration Date:

Batch Number:

Parameter	Reference Concentration	Result Cont. Cal	% Rec.	Acceptance Range -/+ 10%	Out of Specification

APPENDIX C

LANCASTER LABORATORIES ANALYTICAL SOPS

Purgeable Aromatics in High-Level Soils

References:

Methods 8000B, 8020, 8021B, 5030B, 5035B, *Test Methods for Evaluating Solid Waste*, SW-846, U.S. EPA, December 1996.

Scope:

This method is based on a purge and trap (P&T) gas chromatography (GC) procedure and may be utilized to determine the concentration of BTEX (benzene, toluene, ethylbenzene, and xylenes*), MTBE (methyl *t*-butyl ether), isopropylbenzene (IPB), and naphthalene by use of a photoionization detector (PID). This method can be used in conjunction with Method 8015B and API for the quantitation of gasoline range organics using a flame ionization detector (FID). The limits of quantitation for these analyses are as follows:

B, T, E: 5 µg/kg
Xylenes*: 15 µg/kg
MTBE: 20 µg/kg
IPB: 10 µg/kg
Naphthalene: 20 µg/kg

NOTE: Xylenes is a cumulative result based on the concentrations of *p*-, *m*-, and *o*-xylenes.

Summary:

Samples are prepared in methanol (MeOH) according to LL Analyses #2379 and #8390. An aliquot of this methanolic solution is analyzed by P&T GC following this method. Detection is achieved by a PID.

Apparatus:

1. Gas chromatograph - Hewlett-Packard 5890 Series II or any other commercially available GC capable of temperature programming that is equipped with a PID and FID that provide proper sensitivity and linearity.

2. GC column - DB-624 capillary GC column, or equivalent, that is 75 m in length with a 0.53 ID and a 3-µm film thickness. Equivalent column must be capable of resolving 2-methylpentane from the solvent (MeOH) front and ethylbenzene from *m/p* xylene.

3. Purge and trap concentrator - O.I. Analytical Model 4560, or equivalent, with the following specifications:

   a. A purging chamber (sparge) that is designed to accept 25 mL of sample and whose water column is at least 12-cm deep. The gaseous headspace of the sparge is <15 mL total volume.

   b. The trap must be at least 25-cm long and have an inside diameter of at least 0.105". The trap is prepacked and purchased from instrument supplies vendor.

   c. The desorber should be capable of rapidly heating the trap to at least 180°C for desorption. The trap should not be heated beyond the trap's maximum temperature. See manufacturer's recommendations for the maximum temperature allowed for specific traps.

   d. Syringes - 25-mL Luer-Lok gastight syringe; 5-mL Luer-Lok gastight glass syringe

   e. Microsyringes - 10-, 25-, 100-, 250-, 500-, and 1000-µL gastight glass syringes

f. Glass vials - 1.5 mL with screw caps fitted with septa

g. 15-mL glass vials with Teflon-lined screws

h. Stainless steel spatula

i. Balances

(1) Mettler analytical balance, or equivalent, capable of accurately weighing to nearest 0.0001 g

(2) Mettler top-loading balance, or equivalent, capable to weighing to the nearest 0.01 g

j. Volumetric flasks (Class A)

Materials:

Laboratory deionized water is used to prepare all sample dilutions and injections.

Methanol, P&T grade or equivalent, is used to extract all solid samples for analysis and to prepare stock solutions not purchased from an analytical vendor. Methanol is also utilized to prepare all secondary dilution standards required for this method. All standards that are obtained from suppliers must indicate purity of compounds in purchased solution. No correction for purity is made if the listed purity is ≥96%.

Purchased standards must have the concentration(s) of the component(s) documented by the supplier. If the purity of the standard is <96%, a calculation to correct for standard purity is performed and documented in the standards notebook.

Safety Precautions:

The toxicities of all compounds used in the method have not been established. However, several of the compounds are considered carcinogens. Each compound should be treated as a potential health hazard. The major route of exposure is inhalation during handling of any neat materials while preparing stock standards. Therefore, these stocks must be prepared in a hood to eliminate the risk of inhaling the vapors. After stock standards are diluted, the potential for exposure is reduced significantly. Nevertheless, care must be taken in handling any standard. Information concerning the known toxicity, properties, or any special handling precautions can be found in the material safety data sheets (MSDS) available from the Safety Officer. Safety glasses and lab coats are required as personal protective wear. Gloves are optional.

Standards:

1. Stock standards

   a. Custom BTEX/GRO mix - 13 individual component stock standard that is purchased from an analytical supplier. The concentration of each component is listed below:

Component	Concentration (ppm)
2-Methylpentane	15000
MTBE	10000
Benzene	5000
Iso-octane (2,2,4-trimethylpentane)	15000
n-Heptane	5000
Toluene	15000
Ethylbenzene	5000
p-Xylene	10000
m-Xylene	10000
o-Xylene	10000
1,2,4-Trimethylbenzene	10000
Isopropylbenzene (cumene)	10000
Naphthalene	10000

b. Custom VOA Surrogate Mix #2 and #3 - Stock surrogate/internal standard are purchased from an analytical supplier. The following tables lists each component in their respective mix at their representative concentrations:

(1) Custom VOA Surrogate Mix #2:

Component	Concentration (ppm)
1-Chloro,3-Fluorobenzene (1Cl,3FIB)	12500
α,α,α-Trifluorotoluene (TFT)	12500
4-Bromochlorobenzene (4-BrClB)	12500

(a) 1-Cl,3-FIB is utilized as the internal standard on the PID. TFT is utilized as a surrogate on both the PID and the FID. 4-BrClB is utilized as a retention time marker for both detectors.

(b) Custom VOA Surrogate Mix #2 is utilized in all method blank, LCS, calibration verification standard, and detection limit standard injections.

(2) Custom VOA Surrogate Mix #3:

Component	Concentration (ppm)
1-Chloro,3-Fluorobenzene (1-Cl,3FIB)	12500
4-Bromochlorobenzene (4-BrClB)	12500

(a) Custom VOA Surrogate Mix #3 is utilized in all sample, calibration, matrix spike, and matrix spike duplicate injections. TFT is introduced to the sample during the methanol extraction. See LL Method #2379 for specific details.

(b) For Method 8021B, surrogate standard is spiked into the vial after methanolic extraction has occurred. The surrogate (TFT) solution used for this spiking is prepared by diluting 0.5 mL of a 15,000 ppm stock solution into 10 mL of methanol. The concentration of this surrogate solution is 750 ppm.

For MeOH preserved containers, add 1 µL of the above solution for every 1 mL of solvent that was submitted to the client. For example, if a vial was submitted to the client with 5 mL of MeOH, then 5 µL of the above solution shall be added to the vial after it has been reweighed after submittal.

c. PA DEP UST standard - A 9-component stock standard purchased from an analytical supplier at the following concentrations:

Component	Concentration (ppm)
MTBE	2000
Benzene	2000
Toluene	2000
Ethylbenzene	2000
p-Xylene	2000
m-Xylene	2000
o-Xylene	2000
Isopropylbenzene (cumene)	2000
Naphthalene	2000

NOTE: Another stock standard is purchased that may be used in place of the above standard. "Custom PA Spike Mix" has the identical concentrations as does the "PA DEP UST Standard," except the former contains TBA for utilization in Department 34 water scans.

d. 5500-ppm certified BTEX/gasoline matrix spiking standard - Purchased from an analytical chromatography supplier. Gasoline is quantitated at a concentration of 5500 ppm and the BTEX constituents are certified at the following concentrations:

Component	Concentration (ppm)
MTBE	112
Benzene	65.2
Toluene	398
Ethylbenzene	89
Total Xylenes*	467

*Total xylenes is a summation of the concentrations of *m*-, *p*-, and *o*-xylenes.

This standard may be used to quantitate BTEX QC if running in conjunction with API or Method 8015B.

e. TFT calibration stock solution - A standard that is purchased from an analytical supplier at a concentration of 15,000 ppm.

All of the above standards are stored in an explosion-proof freezer at -10° to -20°C. Those standards that are purchased from analytical vendors have expiration dates determined by the vendor. Those standards that are prepared from neat solutions are considered stable for 6 months. Proper documentation on all standards must be demonstrated for tracking purposes.

2. Secondary dilution standards

   a. Matrix spike

      (1) PA component "intermediate" matrix spike - Prepared by adding 1.0 mL PA DEP UST stock standard to methanol in a 10-mL volumetric flask.

      (2) PA component "working" matrix spike standard - Prepared by adding 1.0 mL PA intermediate to methanol in a 10-mL volumetric flask.

   b. Calibration

      (1) BTEX/GRO calibration Standard "B" - Prepared by adding 0.5 mL custom BTEX/GRO stock standard to methanol in a 25-mL volumetric flask.

      (2) BTEX/GRO calibration Standard "A" - Prepared by adding 1.0 mL of B to methanol in a 10-mL volumetric flask.

      (3) TFT calibration Standard "B" - Prepared by adding 0.5 mL TFT calibration stock to methanol in a 10-mL volumetric flask.

      (4) TFT calibration Standard "A" - Prepared by adding 1.0 mL of B to methanol in a 10-mL volumetric flask.

   c. Calibration verification ("check") standards

      (1) Intermediate BTEX/GRO check standard - Prepared by adding 0.5 mL custom BTEX/GRO standard to methanol in a 10-mL volumetric flask.

(2) Working BTEX/GRO check standard - Prepared by adding 1.0 mL of the intermediate to methanol in a 10-mL volumetric flask.

d. Detection limit (LOQ) standard - Prepared by adding 25 µL of PA component intermediate matrix spike to methanol in a 10-mL volumetric flask.

e. "3-Component" surrogate standard - Prepared by adding 300 µL of custom VOA surrogate Mix #2 to methanol in a 50-mL volumetric flask.

f. "2-Component" surrogate standard - Prepared by adding 300 µL of custom VOA surrogate Mix #3 to methanol in a 50-mL volumetric flask.

All of the above secondary dilution standards are stored in an explosion-proof freezer at -10° to -20°C for up to 1 month. Proper documentation must be demonstrated on all standards for tracking purposes.

Sample Collection:

Samples are to be collected using the recommended sampling protocol in SW-846. Samples collected should be cooled at 2° to 6°C (36° to 43°F) at the time of collection until analysis. All samples must be analyzed within 14 days of collection.

Personnel Training and Qualifications:

Analysts are considered proficient when they have successfully completed a quad study for the analyses. A quad study consists of four QA standards that are carried through all steps of the analyses and that meet the defined acceptance criteria. Documentation for these studies are in each analyst's training records.

Instrument Operating Parameters:

1. GC conditions - See Appendix I

2. Purge and trap conditions

   The purge cycle consists of an 11-minute purge at ambient temperature followed by an optional dry purge. Desorb the sample at 180°C for 4 minutes and, if necessary, desorb preheat at 175°C. The bake cycle should be 10 minutes at a temperature of at least 180°C. Longer bake cycles may be necessary to facilitate baking off heavier compounds that may remain on the trap after the desorb cycle has concluded.

   Please note that after the desorb cycle has begun, the water remaining in the sparge is automatically drained from the concentrator. No manual draining is required.

3. Calibration

   All calibration standards are prepared in the 25-mL syringe, injecting the required amount of standards into reagent water and methanol (see Appendix II). To these standards, 10 µL of **2-component** surrogate standard is added.

   Attaching the Luer-Lok end of the syringe to the sparge port, inject this final solution into the purge vessel. Proceed with the purge and trap procedure.

   The calibration standards are run at six levels with the first level being at or above the method detection limit (MDL), but below the limit of quantitation (LOQ) for each component. The calibration mimics the normal working range of typical samples submitted for analysis.

Calibration is performed using internal standardization for PID detection and an average calibration factor (CF) curve is utilized. The CF is calculated as indicated below:

$$CF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

Where:

Ax = Peak area of the compound to be measured

Ais = Peak area of the internal standard

Cis = Concentration of the internal standard.
NOTE: For our calculation, Cis had been arbitrarily chosen as 1.

Cx = Concentration of the compound to be measured

If the relative standard deviation (RSD) of the CFs for any analyte is <20% and all defined QC is within specification, then the average calibration factor may be used. If the %RSD criteria are exceeded, the calibration should be evaluated to determine if reanalyzing one (or more) level would bring the %RSD within specifications. Alternately, a curve fit (linear or quadratic) may be used if the %RSD criteria are exceeded, requiring a minimum of six levels for Method 8021B. The alternate fit must have a coefficient of determination (r^2) of $\geq$0.99 and may not force the origin through zero. For every initial calibration that is performed, an MDL standard must be analyzed to ensure that all compounds of interest are detectable.

Procedure:

NOTE: The syringe and syringe plunger should be rinsed at least 3× with deionized water between samples.

1. Set the P&T and GC to the conditions specified under the Apparatus section of this method.

2. To start, analyze a deionized water/setup blank if the instrument has been sitting for more than one analytical shift. This water blank consists of only 25 mL of deionized. No surrogate or any other standard is required.

3. After the P&T has returned to "Purge Ready," a calibration verification standard or check standard must be analyzed. This standard must be analyzed every 12 hours or after 10 field samples have been analyzed. A field sample is a client submitted sample, matrix spike, or matrix spike duplicate. Method blanks, cleanup blanks, LCSs, and LOQ standards are NOT considered field samples. A check standard is comprised of 24 mL of deionized, 1 mL of methanol, 10 µL of **3-component** surrogate, and 10 µL of working check standard solution. See Appendix III for specific levels.

   The check standard is considered within specification if the percent difference for each component is within ±15%. If any analyte is outside acceptance criteria, the standard is repeated with freshly poured solutions. If the repeated standard is still not within acceptance criteria and each injection was properly prepared and analyzed on properly functioning instrumentation, the system must be recalibrated. For Method 8021B, calibration verification involves the calculation of the percent difference of the instrument response between the initial calibration and the verification standard using the following equation:

$$\%\ Difference = \frac{CFV - Avg\ CF}{Avg\ CF}$$

Where CFV and Avg CF are the calibration factors from the calibration verification and the average of the initial calibration, respectively. The percent difference for the calibration verification must be within ±15% for each analyte. If any analytes are outside the 15% difference acceptance window, the % difference for all analytes may be averaged. If the averaged % difference is <15%, then the calibration verification is determined to be in specification. If a continuing calibration standard is utilized in which an average % difference measurement was required to meet specifications, the client must be provided with a list of all analytes that exceed the 15% difference for that standard. If the averaged % difference is <15%, then the calibration verification is determined to be in specification. Alternately, a % drift calculation may be used in place of % difference by using the following equation:

$$\%\ Drift = \frac{Calculated\ Concentration - Theoretical\ Concentration}{Theoretical\ Concentration} \times 100$$

4. After a check standard has been analyzed within specifications, a method blank needs to be analyzed to ensure the system is clean before any sample is analyzed. A method blank is considered within specification if the concentration of each compound of interest is less than that compound's MDL (method detection limit).

   This method blank consists of 24 mL deionized, 1 mL MeOH, and 10 µl of **3-component** surrogate standard. If the method blank is the first one on a batch, it is considered the batch blank for data package purposes.

5. If a new batch is being started, a detection limit standard (DLS) must be analyzed to demonstrate the system's sensitivity at each component's LOQ. This standard consists of 24 mL deionized, 10 µL of **3-component** surrogate, and 10 µL of LOQ standard. Note that there is no percent recovery window for this standard. The mere presence of a component proves the system's sensitivity. If a component is not detected in this standard, maintenance and possibly recalibration may need to be performed.

6. For each batch of field samples (20 maximum per batch), one sample is required to be spiked with known concentrations of target analytes. This sample, known as the background, is extracted 3×. Two of the extracts are spiked and referred to as the MS and MSD (matrix spike and matrix spike duplicate). Please refer to LL Analysis #2379 for the sample preparation and spiking procedure. (See Appendix IV.)

   The background, MS, and MSD are analyzed by adding 1 mL of the samples' methanol extract to 24 mL deionized and 10 µL of **2-component** surrogate. Percent recovery windows for the MS and MSD are component specific and vary based on statistical calculations. Component recoveries are updated annually. Each component, including gasoline if applicable, must have an RPD calculation performed on their respective MS and MSD data. See the Calculations section below for the MS/MSD RPD calculation. The maximum RPD between MS and MSDs is 30%. If any component recovery on the MS or MSD is not within specifications, an LCS must be analyzed to demonstrate that matrix interferences are to blame for the non-conformance(s) in the MS and/or the MSD. Method 8021B requires that an LCS be analyzed per analytical batch. The LCS is prepared by adding 20 µL of the "550 ppm Certified BTEX in Gasoline" standard or, depending upon batch content, 25 µL of the "working PA component standard" to 24 mL deionized, 1 mL of MeOH, and 10 µL of **3-component** surrogate in a 25-mL syringe. The percent recovery windows are component specific and vary based on statistical calculations. Component recoveries are updated annually.

   Collectively, the background, MS, MSD, and the LCS are referred to as the quality control (QC). Each batch requires that QC is analyzed and within specifications. QC should be run before any samples are analyzed.

   NOTE: If the analyst is not beginning a new batch, steps 5 and 6 above are not required before analyzing samples.

7. Prepare methanolic extractions for the samples as per LL Analysis #2379 and #8390.

8. Samples are analyzed by adding 1 mL of the sample's methanolic extract to 24 mL deionized and 10 µL of **2-component** surrogate in a 25-mL syringe. If the odor of the extract indicates high levels of organics, a dilution should be made. However, when preparing dilutions, a constant amount of methanol (i.e., 1 mL) should be maintained.

9. Continue analysis of samples in this manner for either 10 field samples or 12 hours, whichever comes first. At this time, a new check standard must be analyzed within specification before any more samples may be analyzed. All data must have in-spec check standards to be considered valid.

Identification of Analytes:

Identification of analytes - Comparison of sample peak retention times to standard peak retention times is used to tentatively identify compounds. In addition, retention time windows of ±3× the standard deviation of the average retention time for compounds in standards analyzed over a 3-day period can be used. Where this window is close to zero, a default window of 0.03 minutes will be used for all compounds. In many cases, the experience and discretion of the analyst should weigh heavily in the interpretation of the chromatogram. If the identification of a compound is in doubt due to the possible presence of coelutors, the sample is reanalyzed.

Dilution of Samples:

A sample which contains levels of analytes above the dynamic range of the system (i.e., the highest calibration standard) must be reanalyzed at a dilution. A sample whose matrix contains interferences with the internal standard and/or the surrogate should also be analyzed at a dilution. If highly concentrated samples are being run along with "clean" samples, a "cleanup blank" may need to be analyzed to assure that the system is contaminant-free for each sample.

Calculations:

1. The concentration of target compounds for internal standard quantitation, use the following calculation:

$$Concentration\ (\mu g\,/\,kg) = \frac{(Ax)\,(Cis)}{(Ais)\,(CF)} \times DF$$

Where:

Ax = Peak area of the compound to be measured

Ais = Peak area of the internal standard

Cis = Concentration of the internal standard.
NOTE: For out calculation, Cis had been arbitrarily chosen as 1.

CF = Calibration factor

DF = Dilution factor

2. The calculation for the RPD of MS/MSD data is as follows:

$$RPD = \left| \frac{MSR - MSDR}{MSR + MSDR\,/\,2} \right| \times 100$$

Where:

MSR = Matrix spike concentration

MSDR = Matrix spike duplicate concentration

Quality Control:

In order to monitor both the performance of the analytical system and the effectiveness of the method, each field sample, calibration standard, and QC standard is spiked with internal standard/surrogate solution. A matrix spike (MS) and matrix spike duplicate (MSD) are performed on one sample in each batch of up to 20 samples. The recovery for each analyte of interest should be within the statistically derived windows that are calculated on an annual basis for 8000, Update II, and on a semi-annual basis for 8000, Update III.

A. For 8000, Update II Series Methods, the following criteria apply regarding a laboratory control sample (LCS):

1. If the MS/MSD is analyzed first and meets criteria, then the LCS does not need to be analyzed. If the LCS is analyzed, it must be evaluated.

2. If the MS/MSD fails criteria, then the LCS must be analyzed and must meet criteria for the compounds that failed in the MS/MSD.

3. If the LCS fails for compounds that met criteria in the MS/MSD, a nonconformance form (form #2586) must be initiated. Unless the LCS failure is severe (indicative of a preparation problem), passing results of the MS/MSD and/or LCS would meet method requirements.

4. A comment will be added to the analytical report for compounds that failed in the LCS, but met criteria in the MS/MSD.

B. For 8000, Update III Series Methods, the following criteria apply for a laboratory control sample (LCS):

1. Analysis of one LCS per analytical batch is required.

2. If the LCS does not meet criteria, a nonconformance form (form #2586) must be initiated. Corrective action may include instrument maintenance, re-extraction, and/or reanalysis of samples, or data qualification. Corrective action is determined on a case-by-case basis.

Surrogate recoveries (TFT) should be within statistically derived windows per detector. If insufficient data is available to determine these windows, then a window of 70% to 130% should be utilized. The internal standard recovery (1-Cl,3-FlB) should be 50% to 150%.

As stated above, TFT is added to a sample at the time of extraction. If a sample is analyzed **undiluted** and the TFT is not within specifications, the sample is reextracted. If the TFT is not within specifications in the reextraction, proper commenting at the time of reporting is required. The data is reported according to client-specific requirements.

If a dilution is required to analyze a sample, certain criteria must be met for data acceptance:

1. The internal standard must be within specifications (if data is reported from the PID).

2. All, or the majority, of the target compounds must be reportable and within calibration.

Waste Disposal:

Expired standards in methanol are disposed of as hazardous waste. Aqueous standards may be disposed of as non-hazardous waste due to the low concentration of volatile organics. For disposal procedures of methanolic extractions, please refer to LL Analysis #2379. Raw soil samples are collected by Sample Storage until they are disposed of by incineration.

Appendix I: GC Conditions

1. Flow rates (mL/min)

Carrier gas (He)	11 ± 2
Makeup gas (He)	20 ± 2
Hydrogen	35 ± 4*
Air	160 ± 10*

*Settings will vary according to manufacturer's specifications

2. Temperature program and ramp rates

Initial Temp	50°C	for 0.5 minutes
Ramp Rate #1	1.5°C/minute	-
Ending Temp #1	65°C	-
Ramp Rate #2	10°C/minute	-
Ending Temp #2	210°C	-
Ramp Rate #3	35°C/minute	-
Ending Temp #3	240°C	for 2 minutes

3. Miscellaneous GC settings

Injector Temp	220°C
Detector Temp	260°C

NOTE: Conditions may be altered to improve resolution of BTEX and/or GRO, or to facilitate column cleanup after samples containing heavier compounds are analyzed.

Appendix II: Calibration levels

PID

Component	Level 1 2.5 µL (A) 25 mL	Level 2 5 µL (A) 25 mL	Level 3 10 µL (A) 25 mL	Level 4 25 µL (A) 25 mL	Level 5 5 µL (B) 25 mL	Level 6 10 µL (B) 25 mL
MTBE	2	4	8	20	40	80
Benzene	1	2	4	10	20	40
TFT*	7.5	15	30	75	150	300
Toluene	3	6	12	30	60	120
Ethylbenzene	1	2	4	10	20	40
m/p-Xylene	4	8	16	40	80	160
o-Xylene	2	4	8	20	40	80
Isopropylbenzene	2	4	8	20	40	80
Naphthalene	2	4	8	20	40	80

*TFT levels are based upon a 15,000 mg/kg stock.

NOTE: All levels above are in µg/kg.

Appendix III: Check standard amounts

PID

Component	Level (µg/kg)
MTBE	20
Benzene	10
Toluene	30
Ethylbenzene	10
m/p-Xylene	40
o-Xylene	20
Isopropylbenzne	20
Naphthalene	20

Appendix IV: Spiking Levels for MS/MSD and LCS

1. 550 ppm BTEX/gasoline

Component	Level (µg/kg)
MTBE	224
Benzene	130.4
Toluene	796
Ethylbenzene	178
Total Xylenes*	934
Gasoline	11,000

*Total Xylenes is a cumulative total of *m*-, *p*-, and *o*-xylenes

NOTE: Above levels are listed after a DF of 25 is applied.

2. PA UST spike

Component	Level (µg/kg)
MTBE	500
Benzene	500
Toluene	500
Ethylbenzene	500
Total Xylenes*	1500
Isopropylbenzene	500
Naphthalene	500

*Total Xylenes is a cumulative total of *p*-, *m*-, and *o*-xylenes

NOTE: Above levels are listed after a base DF of 25 is applied.

Revision Log:

Initiated Date: 04/03/91

Ver. #	Effective Date	Change
00	05/14/96	Previous Issue
01	12/26/97	Major changes are as follows: • Changes and revisions due to the implementation of method 8000B and 8021B • Changes to Reference section • Revision to Standard section • Additions to Instrument Operation Parameters section • Changes and additions to Procedure section • Addition and deletion to Calculation section • Changes to Quality Control section • Reformatting and editing of Tables
02	09/17/98	Major changes are as follows: • Method number changed from Analysis #6175, 1837, 4262, 2213, 6998, 2464, 6868 to Analysis #8179, 8180, 8181, 8182, 2213, 6998, 2464, 6868
03	01/21/99	Major changes are as follows: • Deleted reference to longer retention time window for methyl *t*-butyl ether in "Identification of Analytes" section
04	FEB 16 1999	Major changes are as follows: • Calibration - Inserted references to acceptance criteria for initial calibrations that utilize a linear or quadratic fit • Calibration - Included section on uses the average of all analytes %D for determining the acceptance of a continuing calibration standard and that the client must be notified of all analytes that exceed the individual %D requirement

Ver. #	Effective Date	Change

- Quality Control - Addressed the location of statistically derived acceptance windows for surrogate and QC recoveries
- Quality Control - Inserted verbage on action taken upon failing an LCS
- Calculations - Modified sub-section #1 that includes calculation of concentration of target analytes; updated the equation to address current practices

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Prepared by: _____ Date: 2-15-99

Approved by: _____ Date: 2/15/99

Approved by: Dorothy McLove Date: 2/16/99

CONFIDENTIAL

Analysis #1862, 1861, 1632
Revision 02
Supersedes Date: 02/27/98
Effective Date: OCT 0 5 1998

Polynuclear Aromatic Hydrocarbons in Soils, Sludges, Water, and Wastewater

Reference:

1. Method 3510B/8310, *Test Methods for Evaluating Solid Waste*, SW-846, EPA, September 1986.

2. EPA Method 610.

Scope:

This method is applicable to the measurement of the following polynuclear aromatic hydrocarbons (PAHs) in soil, sludges, water, and wastewater.

Analyte	Limit of Quantitation (µg/kg)	µg/L
Napthalene	270	8
Acenapthylene	270	8
Acenaphthene	270	8
Fluorene	27	0.8
Phenanthrene	11	0.3
Anthracene	5.3	0.2
Fluoranthene	5.3	0.2
Pyrene	27	0.8
Benzo (a) anthracene	2.7	0.08
Chrysene	11	0.3

Analyte	Limit of Quantitation (µg/kg)	µg/L
Benzo (b) fluoranthene	2.1	0.06
Benzo (k) fluoranthene	2.1	0.06
Benzo (a) pyrene	2.7	0.08
Dibenzo (a,h) anthracene	5.3	0.2
Benzo (g, h, i) perylene	16	0.5
Indeno (1, 2, 3-cd) pyrene	11	0.3
1-methylnapthalene	--	10
2-methylnapthalene	--	10

This method is used for analyzing soil and sludge samples scheduled for Analysis #1862. It is also used for analyzing water and wastewater samples scheduled for Analysis #1861.

Basic Principles:

A 30-g portion of homogenized sample is dried with sodium sulfate and extracted with 50% methylene chloride in acetone. The extract is filtered, dried, concentrated by evaporation, diluted into ACN and put through silica gel, if necessary. A 1-L sample of water or wastewater is extracted with methylene chloride. The extract is dried concentrated by evaporation and solvent exchanged to ACN. The PAHs are identified and quantitated using reverse phase HPLC with both UV and Fluorescence detection.

Apparatus:

1. Amber glass screw cap vial - 12-mL capacity
2. HPLC gradient pumping system
3. 20-µL injection loop

4. UV spectrophotometric detector

5. Fluorescence detector, specific excitation, broad range emission.

6. Dual channel integration system such as Chrom Perfect by Justice Innovations or equivalent

7. Supelco LC-PAH, 10 cm × 4.6 mm, 3 µm or equivalent

Reagents and Standards:

1. Methylene chloride, HPLC grade

2. Acetone, HPLC grade

3. Acetonitrile, HPLC grade

Safety Precautions:

Avoid inhaling the solvents or getting them on the skin. Wear gloves when handling methylene chloride as well as the samples. Avoid contact with the standards. Wear gloves, a laboratory coat, and safety glasses while handling neat materials.

Sample Collection, Preservation, and Handling:

Samples must be collected in glass with Teflon-lined lids. The samples must be maintained cool, 4° ± 2°C. Samples must not be collected in plastic due to the possibility of sample contamination from hydrocarbons within the plastic. Samples should not be collected in the presence of exhaust fumes. Soil samples must be

extracted within 14 days of collection and analyzed within 40 days of extraction. Water samples must be extracted within 7 days of collection and analyzed within 40 days of extraction.

Standards:

Neat Stock Standards:

Stock 1: Nitrobenzene: Approximately 0.25 g to 25 mL Acetonitrile 10,000 ppm

Stock 2: Triphenylene: Approximately 0.025 g to 25 mLs Acetonitrile 200 ppm

Purchased Stock Standards:

Stock A: Custom Mix A PAH, Ultra Scientific Catalog No: CUS-827A
Stock B: Custom Mix B PAH, Ultra Scientific Catalog No: CUS-1017A

Standard ID	Stock	Initial Vol. (mL)	Final Vol. (mL)	Solvent
PAH Surrogate	Stock 1	2	200	Acetonitrile
	Stock 2	1		
Mix A Intermediate	Stock A	2.5	25	Acetonitrile
Level 5	Mix A Interm.	4	100	1:5 $MeCl_2$/ACN
	Stock B	10		
	Stock 1	2		
	Stock 2	1		
Level 4	Mix A Interm.	2	100	1:5 $MeCl_2$/ACN
	Stock B	5		
	Stock 1	1		
	Stock 2	0.5		

Standard ID	Stock	Initial Vol. (mL)	Final Vol. (mL)	Solvent
Level 3	Mix A Interm.	1.6	200	1:5 $MeCl_2$/ACN
	Stock B	4		
	Stock 1	0.5		
	Stock 2	0.2		
Level 2	Level 5	0.5	10	1:5 $MeCl_2$/ACN
Level 1	Level 5	1	50	1:5 $MeCl_2$/ACN
MDL STD	Level 1	2	10	1:5 $MeCl_2$/ACN

Standards Database Entry:

Enter each standard into the system as follows:

Level 1: PAHX197A (PAHX PREFIX)
Level 2: PAHX297A
Level 3: PAHX397A
Level 4: PAHX497A
Level 5: PAHX597A

Example Sequence:

Sample Name	ID	File Name	Method	Sample Wt.	Dil F
CONDITIONER	AA	1C4P001.01R	PAH.MET	1.0	1.0
CONDITIONER	AA	1C4P001.02R	PAH.MET	1.0	1.0
PAHX197A	AA	1C4P001.03R	PAH.MET	1.0	1.0
PAHX297A	AA	1C4P001.04R	PAH.MET	1.0	1.0
PAHX397A	AA	1C4P001.05R	PAH.MET	1.0	1.0

Sample Name	ID	File Name	Method	Sample Wt.	Dil F
PAHX497A	AA	1C4P001.06R	PAH.MET	1.0	1.0
PAHX597A	AA	1C4P001.07R	PAH.MET	1.0	1.0
BLANKA	AA	1C4P001.08R	PAHW.MET	1000	2
LCSA	AA	1C4P001.09R	PAHW.MET	1000	2
LCSDA	AA	1C4P001.10R	PAHW.MET	1000	2
SAMPLE1	AA	1C4P001.11R	PAHW.MET	1000	2
SAMPLE2	AA	1C4P001.12R	PAHW.MET	1000	2
BLANKB	AA	1C4P001.14R	PAHS.MET	30.0	2
LCSB	AA	1C4P001.15R	PAHS.MET	30.0	2
LCSDB	AA	1C4P001.16R	PAHS.MET	30.0	2
SAMPLE1	AA	1C4P001.17R	PAHS.MET	30.0	2
SAMPLE2	AA	1C4P001.18R	PAHS.MET	30.0	2
PAHX397A	AB	1C4P001.19R	PAHCC.MET	1.0	1.0
TEN SAMPLES	AA	1C4P001.20R	PAHS.MET	30.0	2
PAHX397A	AC	1C4P001.21R	PAHCC.MET	1.0	1.0

Personnel Training and Qualifications:

Each analyst performing the instrumental analysis will work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set-up sequences, perform calculations, interpret the chromatograms, and enter the data into the LIMS. They will also follow the department training manual for analysts. Proficiency is measured through documented audits of the tasks listed and overchecking of data.

Chromatographic Procedure:

1. HPLC setup

   Column - Supelco LC-PAH, 250 mm × 4.6 mm × 5 µm

   Mobile phase - A = H_2O
   B = Acetonitrile

   Gradient - 40% B-1 min 100% at 8 min hold 6 min.
   Or a gradient which provides acceptable resolution for all 16 PAHs on the UV detector.

   Flow - 2.0 mL/minute

   Temperature - 35°C, regulated by a column oven

   Injection loop size - 20 µL

   UV - 254 nm

   Fluorescence - Kratos: 280 nm excitation
   370 nm emission
   0.01 µA PMT signal

   H.P. - 280 nm excitation
   0 nm emission for full range response

2. Both mobile phases must be degassed prior to use. A second redegassing before seven days is usually not necessary.

3. The system should be set up and checked as described below:

   a. Set all HPLC parameters to those listed in Sections 1 through 3 of the method.

   b. Pump mobile phase at the initial gradient conditions for 10 minutes. After 10 minutes, check the entire system for leaks (i.e., all connections, injection loop, Shimadzu pump heads, detector inlets and outlets, etc.).

4. External calibration is performed by injecting working mixes at five concentration levels.

5. Calculate response factors (RF) for the first six compounds listed in this method from the UV data (RF = peak height divided by concentration [ng/mL]). Calculate RFs for the last ten compounds from the fluorescence data in the same fashion. RFs for the last ten compounds on UV and first six compounds on fluorescence need not be calculated unless second detector confirmation is required. If the RSD for any compound is <20%, average response factor quantitation can be used (10% for Method 610). If only a few compounds exceed 20% RSD, an average RSD for the entire list of compounds can be determined. If this average is under 20%, average response factor quantitation can be used. This applies to SW-846 samples only. Final data comments should contain a comment stating this option was used when quantitation for that compound was based on this option. As an alternative, linear calibration may be used for quantitation.

6. Run an MDL standard prior to each ICAL to confirm sensitivity on the primary detector.

7. For SW-846 8310 analyses, the working calibration curve or RF must be verified at least once per day (after every ten injections is recommended) by injecting the Level 3 calibration mix. The concentration for each analyte must

show ≤15.0% RPD as quantitated from the initial calibration. If only a few compounds exceed this criteria, the average RPD for all compounds can be determined. If the average RPD is ≤15%, analysis can continue. Quantitation performed using this option must be flagged as such.

For EPA Method 610 analyses, the working calibration curve or RF must be verified at least once per day by injecting a level 3 standard. The concentration for all analytes must be within ±15% RPD as quantitated from the initial calibration.

For SW-846 analyses, all samples must be bracketed by an acceptable continuing standard. All samples must be re-injected which were injected since the last acceptable standard.

8. If the problem is corrected, it must be verified by showing all RPDs ≤15.0% (this applies to RFs to be used for primary quantitation only) and sample analysis may proceed. If the problem can not be corrected, then the system must be recalibrated. If no samples were injected after the system was deemed out of control then no sample reanalysis is necessary. The option of averaging the RPDs for all compounds may be used. The average RPD cannot exceed ±15%. If this option is used, final data comments should reflect that this option was used.

9. Retention time windows (for a given column) are generated by making three mid-level standard injections over a 72-hour period. Calculate the average retention time for each analyte. Each subsequent window is calculated as the average ±3× the standard deviation. Retention time windows may by updated as needed by modifying the midpoint retention times.

10. Any analyte concentration above the working range of the standards must be diluted to within the range.

11. Silica gel cleanup (as per Method 3630A SW-846) may be used for messy samples. See Appendix V of the Department 24 Methods Manual.

12. Any time the injection sequence is discontinued for more than 8 hours, the mid-level calibration mix must be injected just before and right after the break in the injection sequence. Before sample analysis proceeds, the requirements listed in Step 8 must be met.

13. All standards and samples must be warmed to room temperature prior to being injected.

Data Interpretation:

1. Quantitation of individual PAH compounds will be performed using the primary detector for that compound. Napthalene, acenapthene, acenapthylene, fluorene, phenanthrene, and anthracene using UV Other 10 PAH compounds on fluorescence.

2. Disparity in quantitative results between sides will not be used to perform worst case analysis.

3. Confirmation is based on retention time only. If a compound is detected in the retention time window for both detectors, and the same peak is being called on both sides, the primary result is reported. The retention time for the fluorescence detector should be within 0.05 min. of the UV detector retention time to consider the peak in the same window. If peak retention times do not agree, perform a worst case evaluation.

4. Due to differences in sensitivity, results below the LOQ cannot be confirmed. In these cases, the results will be reported from the primary detector without retention time confirmation.

5. Primary detector calibration data must meet Update III criteria. See updated calibration SOP.

Calculations:

$$\frac{Pk\ Ht \times FV \times DF}{RF} = Concentration\ (\mu g/kg)$$

Where

Pk Ht = Peak height found in the sample

RF = Response factor (peak ht/ppb) of the analyte in the standard

FV = Final volume of the sample extract (mL)

AF = Additional factor

DF = Dilution factor (where applicable)

IW = Initial weight of the sample extracted (g)

Quality Assurance:

1. A reagent blank (using sodium sulfate) is extracted with every batch of 20 samples or less.

2. A laboratory control sample (a spiked reagent blank) is extracted with every batch of 20 samples or less.

3. A matrix spike (MS) and a matrix spike duplicate (MSD) is extracted for every 20 samples or every 14 days, whichever comes first.

4. See SOP-PP-002, "QC Data Acceptability and Corrective Action," for QC acceptance criteria and SOP-PP-025, "Monitoring QC Data Acceptance Limits," for monitoring QC limits.

Revision Log:

Initiated Date: 10/26/90

Ver. #	Effective Date	Change
00	03/21/95	Previous Issue
01	02/27/98	Major changes are as follows: • Combined Analysis #1861 and #1862 • Took out preparation section • Changed LOQs to reflect final volume of 2 mL • Added standard preparation section • Added data interpretation section
02	OCT 0 5 1998	Major changes are as follows: • Added 1- and 2-methylnapthalene, Method 610 requirements, Analysis 1632, and column dimensions • Changed holdtime for water to 7 days • Corrected RF reference

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Prepared by: _____ Date: 9/28/98

Approved by: _____ Date: 9/30/98

Approved by: Susan B. Shorter Date: 10/2/98

Figure 1

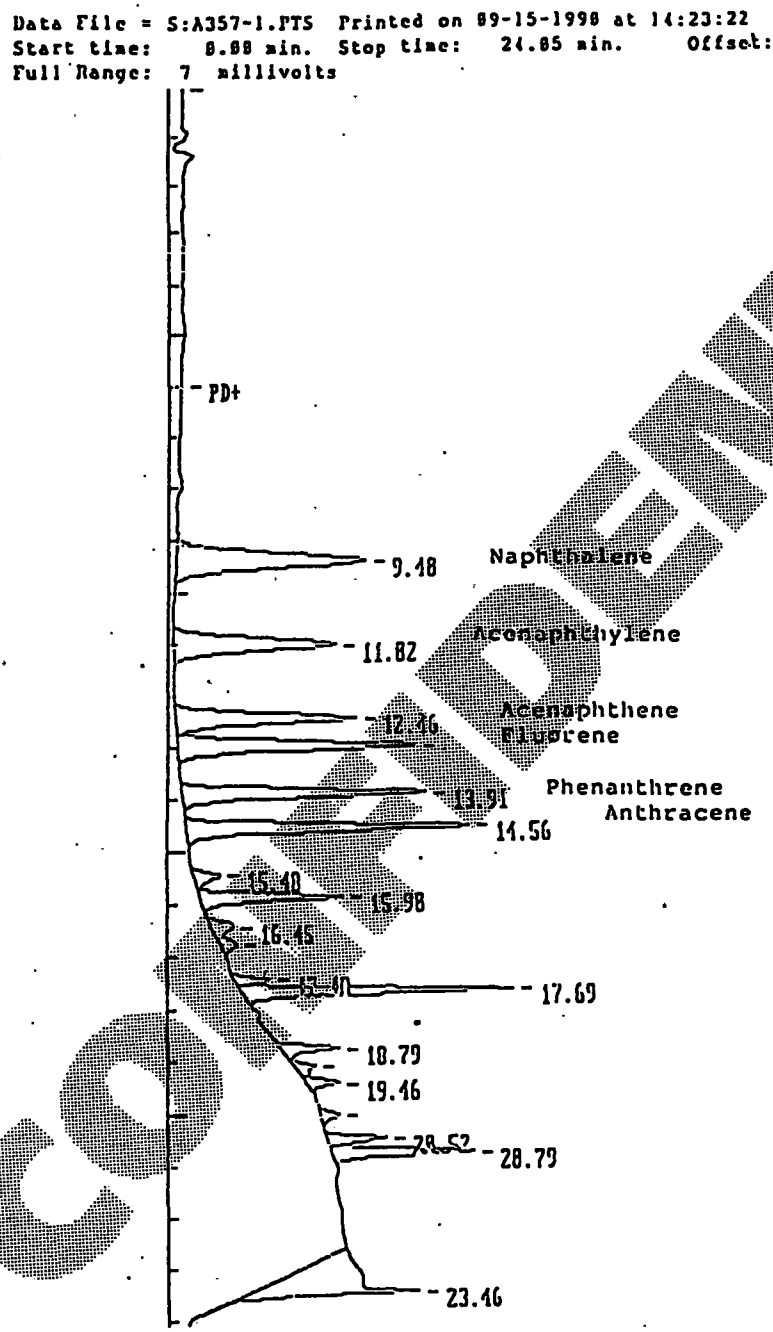

FIGURE 1: PAH's which are determined using UV detection as the primary mode of detection.

Figure 1 - continued

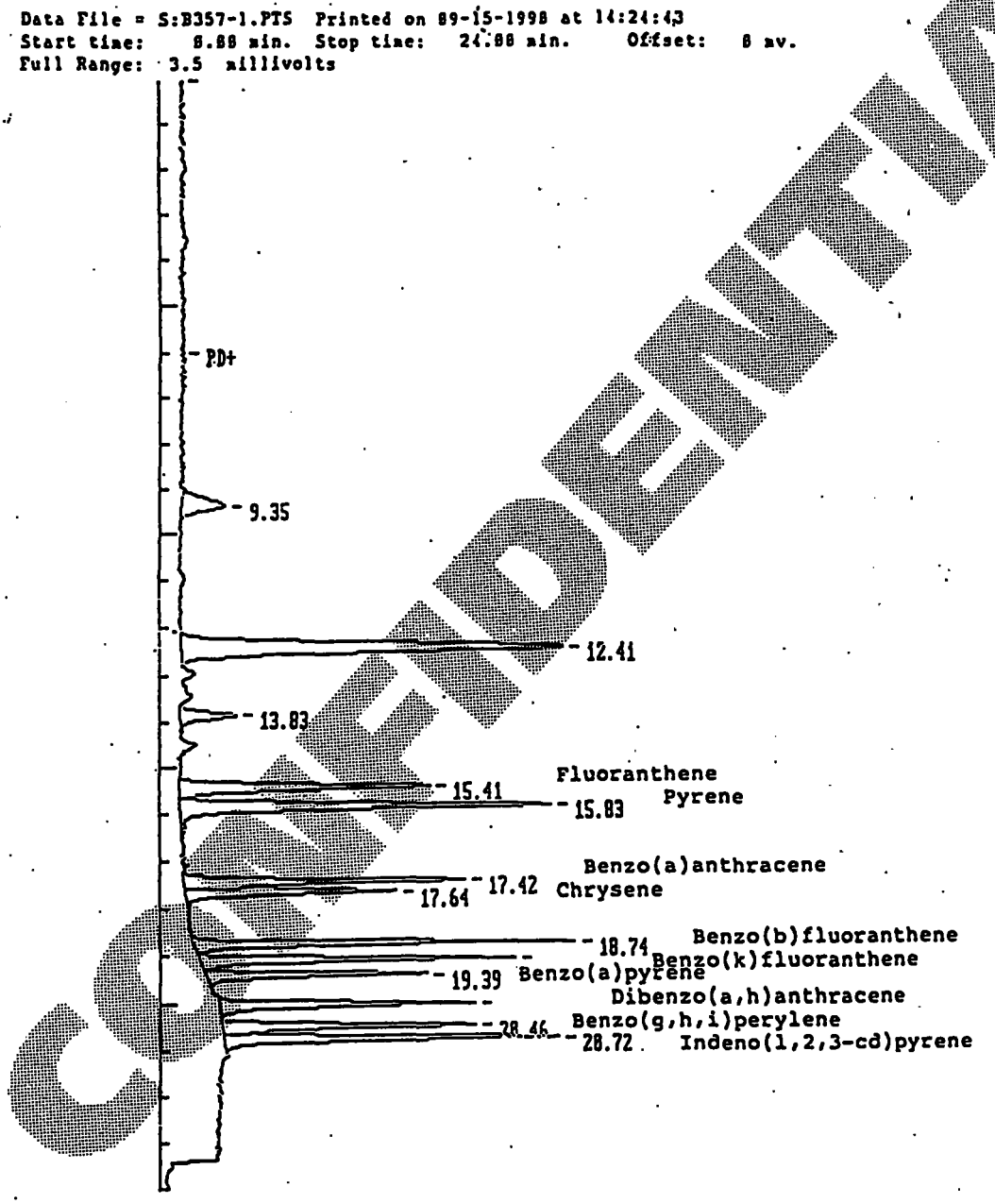

FIGURE 2: PAH's which are determined using Fluorescence detection as the primary mode of detection.

Analysis #0949, 0968, 1198, 1199, 1200, 1309, 1310, 1311, 1312, 1424, 1425, 1426, 4688, 4689, 4678, 4679, 4615, 4616, 4617, 4618, 3349, 7820, 7821, 7822, 7823, 7804, 7805, 5749, 5750, 7357, 7358, 7437, 7438, 7588, 7589, 7804, 7805

Revision 03
Supersedes Date: 01/16/98
Effective Date: MAR 1 1 1999

Determination of Semivolatile Organic Compounds by Method 8270C

Reference:

1. Method 8270C, USEPA SW-846
2. Method 8000B, USEPA SW-846
3. *Federal Register*, Vol. 57, No. 227, November 24, 1992, p. 55114 (TCLP).
4. *Federal Register*, Vol. 57, No. 160, August 18, 1992, p. 37203 (CCW).

Scope:

This method is suitable for the determination of the concentration of certain semivolatile organic compounds (priority pollutant list, target compound list, Appendix IX list, TCLP list, and CCW list) found in soils, waters, and leachates.

Personnel Training and Qualifications:

Education Requirement: Degree in science or relevant experience.

Each new chemist will train with an experienced chemist for the first 12 weeks depending on the individual and their previous experience. The first 12 weeks are spent working one-on-one with the trainer. This time may be less if the new chemist

has prior experience in the GC/MS Semivolatiles area or relevant analytical chemistry background. Each new chemist receives a training manual outlining the basics of operating the GC/MS and data work up.

During the training period, the new chemist will learn daily maintenance, column and source changing procedures, calibration techniques, data and library search review, and forms generation. They are also required to read all relevant SOPs and EPA methods.

To measure the proficiency of each chemist, several checks have been established. The first is the ability to calibrate for each method. The chemist will run a series of at least five calibration standards and perform the calibration routine. The curve will then be reviewed by a departmental data validator. They will confirm that relative retention times (RRT) and response factors (RF) match throughout the calibration and ID list. Secondly, each analyst must perform a quad study. This will consist of serial dilutions on a known concentration mixture and analyzing four back-to-back replicates of these dilutions. This process will measure accuracy in dilution preparation as well as reproducibility of results. All data and forms generated by each chemist must pass through a thorough technical review by a departmental data validator. Any errors will be corrected by the chemist and these corrections will again be reviewed by the data validator.

Basic Principles:

A 1-microliter mixture of organic compounds in methylene chloride is injected onto a 0.25-mm (internal diameter) fused silica capillary column coated with a relatively non-polar stationary phase, which is enclosed in a temperature controlled oven. A carrier gas, which is ultra pure helium, passes continuously through the column (note that the rate of the helium flow is usually controlled by an internal or external pressure control unit). The GC oven is temperature programmed and the organic mixture separates into its individual components as it moves along the length of the column. This separation is a function of the polarity and boiling point of the individual compounds. The column empties into a mass selective detector. When a compound

reaches the detector, it is bombarded by high-energy electrons (70 eV). This causes the compounds to fragment, forming ions. By applying various voltages to plates in the area where the ions are formed, the positive ions are thrust into a quadruple mass analyzer which selects for a given mass fragment at a given time. These selected fragments reach an electron multiplier which amplifies the signals and sends them to a computer making storage and manipulation of the data possible. Target compounds are identified on the basis of relative retention times and spectral match to standards which are injected every 12 hours on the same system.

Quantitation is achieved by the internal standard method and the average response factor of a calibration using at least five concentration levels.

Apparatus:

1. Hewlett-Packard Model 5890 (Series I and II) or 6890 Gas Chromatograph or equivalent

2. Hewlett-Packard Models 5971, 5972, and 5973 Mass Selective Detector or equivalent

3. Hewlett-Packard HP-1000 RTE-A Data System and/or Chem Station or equivalent

Reagents:

1. Decafluorotriphenylphosphine (DFTPP)

2. Methylene chloride, pesticide grade

3. Internal standard mix

Safety Precautions:

The toxicity of each reagent used has not been precisely determined. However, each reagent should be treated as a potential health hazard. Safety measures would include the use of fume hoods, safety glasses, lab coats, and gloves when working directly with reagents. Refer to the Lancaster Laboratories *Chemical Hygiene Plan* for specific details.

Procedure:

A. Standard preparation - These solutions are used to standardize the GC/MS system every 12 hours and are prepared weekly or more frequently if needed. See SOP-EX-001, "Semivolatile Spiking and Calibration Standards," for standard preparation and validation. Calibration standard solutions shall be used for 1 week or until component degradation is observed.

B. Daily maintenance - Refer to MC-EX-001, "GC/MS Daily Routine Maintenance," for this procedure.

C. Tuning

Frequency	Criteria Acceptance	Corrective Action
Every 12 hours	1. Criteria in Table I 2. DDT breakdown ≤20%* 3. Tailing factors: Benzidine ≤3 Pentachlorophenol ≤5	1. Retune. Analysis cannot proceed until tune meets criteria. 2. More aggressive injection port maintenance. 3. Clean the source. 4. Change the column.

* DDT breakdown ≤20% may be acceptable if you are calibrating for polynuclear aromatic hydrocarbon compounds only. Consult supervisor when this situation occurs.

You are only permitted to use a background subtracted spectrum of the following in evaluating the DFTPP:

1 The apex of the scan.

2. The apex of the scan -1.

3. The apex of the scan +1.

4. A three scan average of the above three scans.

5. A five scan average.

NOTE: All standards, samples, and associated quality control samples with a particular tune must use the identical conditions of the mass spectrometer.

D. Initial Calibration

Standardization is performed by analyzing at least five levels of calibration standards from which an average response factor is generated for each compound. Refer to the *GC/MS Semivolatile Training Manual* for more specific information. A method detection limit (MDL) standard must be analyzed with each initial calibration. This standard is prepared at the departmental MDL and is not to be included in the calibration curve. All compounds must be detected in the MDL standard.

Frequency	Acceptance Criteria	Corrective Action
Initially and then when CCCs and/or SPCCs in the daily calibration standard fail criteria. Initially establish with at least five levels of standards and an MDL standard. See Table 2 for a list of the SPCC and CCC compounds.	1. Ave RRF for each SPCC ≥0.05. 2. %RSD for each CCC ≤30%. 3. %RSD for non-CCCs ≤50%.* 4. All compounds of interest must be detected in the MDL standard. 5. The relative retention times of the target compounds must agree within 0.06 relative retention time units. The exception would be for the case of system maintenance.	1. Any target analyte with a %RSD >15%, use a first degree fit if the correlation coefficient is ≥ 0.99. If <0.99, use a second order fit. If both the first and second order fits have a correlation coefficient ≥0.99, then use the fit with the smallest negative y-intercept. When using a second order fit, if the y-intercept quantitates to be greater than the MDL, consult your supervisor immediately or recalibrate. 2. If a compound is not detected in the MDL standard, then report to the level of the lowest standard detected. All compounds manually integrated in this standard must be checked for in each sample analyzed under this initial calibration.* 3. More aggressive system maintenance and recalibrate.

***If these situations occur, your supervisor is to be consulted immediately.**

E. Continuing calibrations

Frequency	Acceptance Criteria	Corrective Action
1. Every 12 hours. 2. Check standard must be at the mid-point of the calibration range.	1. RRF for each SPCC ≥0.05. 2. %Drift for each CCC ≤20%. 3. %Drift for all non-CCCs ≤50%. * 4. The relative retention times of the target compounds must agree within 0.06 relative retention time units. The exception would be for the case of system maintenance. 5. The EICP area for each internal standard must fall within the window of -50% to +100% from the last initial calibration.	1. If the CCC or SPCC compounds do not meet criteria but all compounds of interest have a %Drift ≤20%, the calibration may be used. ** 2. More aggressive system maintenance or recalibrate

***If these situations occur, your supervisor is to be consulted immediately**

**** Notification to the data user will occur in the case narrative that is submitted with the data package. Your supervisor must be consulted.**

F. Qualitative analysis

A compound is identified by comparison of the following parameters with those of a standard of this suspected compound (standard reference spectra). In order to verify identification, the following criteria must be met:

1. The intensities of the characteristic ions of the compound must maximize in the same scan or within one scan of each other.

2. The sample component relative retention time should compare within ±0.06 RRT units of the RRT of the standard component.

3. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum.

4. The relative intensities of the characteristic ions should agree within 30% of the relative intensities of these ions in the reference spectrum. Analyst discretion is used to determine compound identification. (Example: for an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

5. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

6. The above criteria apply to hits greater than or equal to the LOQ. For hits between the MDL and the LOQ, the above criteria and analyst discretion are used to determine compound identification.

G. Quantitative analysis

Once a compound has been identified, quantitation will be based on the internal standard technique and the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion.

Waters:

$$Concentration\ (\mu g\ /\ L) = \frac{A(x) \times I(s) \times V(t) \times D_f}{A(is) \times RRF \times V(o) \times V(i)}$$

Where:

A(x) = Area of characteristic ion for compound being measured

I(s) = Amount of internal standard injected (ng)

V(t) = Volume of concentrated extract in microliters (µL)

D_f = Dilution factor

A(is) = Area of characteristic ion for the internal standard

RRF = Relative response factor for the compound being measured

V(i) = Volume of extract injected (µL)

V(o) = Volume of water extracted (mL)

Soils:

$$Concentration\ (\mu g\ /\ kg) = \frac{A(x) \times I(s) \times V(t) \times G \times D_f}{A(is) \times RRF \times W(s) \times V(i) \times D}$$

Where:

A(x) = Area of characteristic ion for compound being measured

I(s) = Amount of internal standard injected (ng)

V(t) = Volume of concentrated extract in microliters

D_f = Dilution factor

A(is) = Area of characteristic ion for the internal standard

RRF = Relative Response factor for the compound being measured

V(i) = Volume of extract injected (µL)

W(s) = Weight of sample extracted or diluted in grams

D = The percent solids (100 - % moisture)/100

G = 1 if extract did not require GPC cleanup

= 2 if extract required GPC cleanup

1. Calculation of the relative response factor (RRF):

$$RRF = \frac{[A(x) \times C(is)]}{[A(is) \times C(x)]}$$

Where:

A(x) = Area of the characteristic ion for the compound being measured

A(is) = Area of the characteristic ion for the specific internal standard

C(x) = Concentration of the compound being measured

C(is) = Concentration of specific internal standard

2. Calculation of the percent drift:

$$\%\ Drift = \frac{C(i) - C(c)}{C(i)} \times 100$$

Where:

C(i) = Calibration check compound standard concentration

C(c) = Measured concentration using selected quantitation method

H. Quality Assurance:

Each extraction batch must contain a method blank, a laboratory control reference sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), a matrix spike duplicate (MSD) or a laboratory control sample/laboratory control sample duplicate (LCS/LCSD). Additional QC samples may be required to meet project or state certification requirements.

Quality Control Item	Acceptance Criteria	Corrective Action
Internal Standards	1. Peak area within -50% to +100% of the area in the associated reference standard. 2. Retention time(RT) within 30 seconds of RT for associated reference standard.	1. Check instrument for possible problems and then reanalyze samples. 2. If reinjection meets the criteria, report this injection. 3. If reinjection still shows same problem, report first injection and qualify data with a comment.
Method Blank	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. All target compounds must be less than the reporting limit for the associated samples.	1. Inspect system for possible problems and reanalyze. 2. If one surrogate is out of spec high and all associated sample surrogates are in spec, data can be used. (Unless project requirements dictate otherwise). * 3. If the method blank contains target analytes and the associated samples do not contain these compounds, no corrective action is required. If the target compounds in the blank are also in the associated samples, the samples should be reextracted unless it does not interfere with project data requirements.

Quality Control Item	Acceptance Criteria	Corrective Action
Laboratory Control Sample/Laboratory Control Sample Duplicate	1. All percent recoveries within QC limits. Refer to the GC/MS Semivolatile SOP manual for QC windows. These are updated on a semiannual basis. 2. RPDs within QC limits.	1. If non-compliant, check for calculation or preparation errors. 2. If no errors found, check system for problems and reanalyze. 3. If MS/MSD is within QC limits for the analytes out of spec in the LCS and/or LCSD, fill out Non-conformance Form #2586. 4. If no MS/MSD or analyte(s) in MS/MSD also out of spec, consult supervisor immediately. Samples may need to be reextracted.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1. % Recoveries within QC limits. Refer to the GC/MS Semivolatile SOP manual for QC windows. These are updated on a semiannual basis. 2. RPDs within QC limits.	1. If LCS within QC limits, proceed with sample analysis. 2. If most recoveries or RPDs out of spec, consult supervisor.

Quality Control Item	Acceptance Criteria	Corrective Action
Surrogates	All recoveries must be within QC limits. Refer to the GC/MS Semivolatile SOP manual for surrogate windows. These are updated on a semiannual basis.	1. If non-compliant, check for calculation or preparation errors. 2. If no errors found, check system for problems and reanalyze. 3. If no problem is found, reextract and reanalyze the sample.

*** Requires approval of supervisor and completion of Non-Conformance Form #2586.**

I. Dilution criteria

1. Initial dilutions

a. More than three internal standard areas are less than -50%.

b. Either of the last two internal standard areas are less than -80%.

c. Prescreen data or analyst's judgement of a sample extract's color or viscosity indicates possible matrix interference.

2. Secondary dilutions

Are required to bring all target compounds in the calibration range of the GC/MS.

Revision Log:

Initiated Date: 06/09/89

Ver. #	Effective Date	Change
00	07/03/95	Previous Issue
01	07/07/97	Major changes are as follows: • Added section on Personnel Training and Qualifications • Updated method references • Deleted Additional TCLP Requirements section • Removed QC Windows from tables
02	01/16/98	Major changes are as follows: • Changed method number from Analysis #0949, 0968, 1198, 1199, 1200, 1309, 1310, 1311, 1312, 1424, 1425, 1426, 4688, 4689, 4678, 4679, 4615, 4616, 4617, 4618, 3349, 7820, 7821, 7822, 7823, 7804, 7805, 5749, 5750, 7357, 7358, 7437, 7438, 7588, 7589 to Analysis #0949, 0968, 1198, 1199, 1200, 1309, 1310, 1311, 1312, 1424, 1425, 1426, 4688, 4689, 4678, 4679, 4615, 4616, 4617, 4618, 3349, 7820, 7821, 7822, 7823, 7804, 7805, 5749, 5750, 7357, 7358, 7437, 7438, 7588, 7589, 7804, 7805 • Changed title • Made changes to Reference section • Made changes to Procedure C., Standardization
03	MAR 1 1 1999	Major changes are as follows: • Rewrote QA section • Rewrote Procedure section

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Prepared by: Christine M. Ratcliff /412 Date: 3/10/99

Approved by: [signature] /112 Date: 3/10/99

Approved by: Darcys K. Clark Date: 3/10/99

Table I

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30% to 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40% to 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5% to 9% of mass 198
275	10% to 30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17% to 23% of mass 442

Table II

CCCs

Acenaphthene
1,4-Dichlorobenzene
Hexachlorobutadiene
Diphenylamine*
Di-n-octylphthalate
Fluoranthene
Benzo(a)pyrene
4-Chloro-3-methylphenol
2-Nitrophenol
Phenol
Pentachlorophenol
2,4,6-Trichlorophenol

Note: Diphenylamine cannot be separated from N-nitroso-di-phenylamine under the chromatographic conditions used for sample analysis.

SPCCs

N-Nitroso-di-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol
2,4-Dichlorophenol

Analysis #0520, 1131, 1387, 1388,
4601, 4604, 6374, 6376
Initiated Date: 09/18/85
Effective Date: MAR 1 0 1997

The Analysis of Water for Purgeable Organics by Purge and Trap Gas Chromatography/Mass Spectrometry

Reference:

Method 624, *Federal Register*, Revision 7-1-87.

Scope:

This method is suitable for the determination of compounds listed in USEPA Method 624 for aqueous matrices. The method also is appropriate for acquiring data files required to perform a search for tentatively identified compounds (TIC) in volatile organics GC/MS analyses. This analysis should be performed by or under the direct supervision of an operator experienced in the analysis of volatile organics by GC/MS purge and trap methodologies.

Personnel Training and Qualifications:

Each new chemist will train with an experienced chemist for the first 12 weeks depending on the individual and his or her previous experience. The first 12 weeks is spent working one-on-one with the trainer. This time may be less if the new chemist has prior experience in the GC/MS volatiles area.

During the training period, the new chemist will learn daily maintenance, column and source changing procedures, calibration techniques, data and library search review, and forms generation. They are also required to read all relevant SOPs and EPA methods. They also participate in an on-line GC/MS tutorial.

All data and forms generated by each chemist must pass through a thorough technical review by a departmental data validator. Any errors will be corrected by the chemist and these corrections will again be reviewed by the data validator.

Basic Principles:

A 5-mL sample or a dilution of a sample is placed in a specifically designed purge vessel. The sample is purged with an inert gas and the effluent gas passed through a sorbent trap where the volatile organics are trapped.

After purging, the sorbent trap is rapidly heated and backflushed onto the head of a gas chromatographic column. The gas chromatographic column is temperature programmed to separate the volatile compounds which are subsequently detected and identified using mass spectrometric techniques.

Apparatus:

1. Micro syringes - 10 μl and larger
2. 5-mL gastight syringe
3. Analytical balance, capable of accurately weighing ±0.001 g
4. Glassware - Volumetric flasks, Class A with ground-glass stopper
5. Purge and trap device, consisting of the sample purger, the trap, and desorber. The OI Analytical 4560, Tekmar LSC 2000, or equivalent meets the requirements of this method.
6. GC/MS system - The HP 5970 MSD, Finnigan Incos 50, or equivalent GC/MS system meets the requirements for this method.

Reagents:

1. Reagent water is defined as water in which an interferant is not observed at or above the reporting limit for parameters of interest. In general, the deionized water supplied at the taps in the laboratory will meet this criteria. If the reagent water does not meet the requirements, see your supervisor for further instructions.

2. Methanol, for purge and trap analysis or equivalent

3. Stock standard solutions - Prepared from assayed standard materials or purchased. Stock solutions must be prepared in methanol.

   a. Place about 9.8 mL methanol into a tared 10.0-mL glass-stoppered volumetric flask. Weigh the flask to the nearest 0.1 mg.

   b. Add the liquids using a syringe or a pipet by adding two or more drops of the assayed material to the flask, being careful that no drop hits the side of the flask. Bring the volume of methanol in the flask to 10.0 mL. Calculate the concentration of the standard.

   c. The gases bromomethane, chloromethane, chloroethane, and vinyl chloride are purchased from an outside vendor and used as received.

   d. The stock standard solutions are stored in Teflon-sealed screw-capped vials at -10° to -20°C. The compound name, concentration, date prepared, and expiration date must appear on the bottle label.

   e. Replace stock standard solutions every 6 months.

4. Secondary dilution standards - Using the stock standard solutions, prepare secondary stock solutions in methanol containing the compounds listed in Tables 1A through 1F. Each standard is prepared by calculating the volume of each stock standard required to produce a given volume of a mixed working standard with a known concentration of each analyte. The working standard is tested according to SOP-MS-006, "GC/MS Volatile Standards Traceability." The verified working standard is pipetted into 1.5-mL Teflon-lined screw-capped vials which are stored at -10° to -20°C. A designator indicating the standard, month, and day of preparation must be on the sample label. The designator and the calculation are to be recorded in the standards logbook. Replace secondary dilution standards every 6 months.

5. Matrix spiking solution - Prepare solution(s) in methanol that contain(s) all the compounds listed in Tables 1A through 1F at a known concentration.

   This solution will be used for the spike, spike duplicate, and QC reference sample.

6. Surrogate standard spiking solutions - Prepare stock standard solutions for fluorobenzene, *p*-bromofluorobenzene, and 1,2-dichloroethane-d4 in methanol. Prepare the surrogate standard spiking solutions from the stock standards at concentrations of 10 µg/mL and 100 µg/mL in methanol for use in initial calibration standards. Prepare a surrogate spiking solution from the stock standards at a concentration of 15 µg/mL in methanol for use in check samples, blanks, and samples. Replace the surrogate standard spiking solutions every 6 months.

7. Internal standards solution - Prepare a solution in methanol containing bromochloromethane, 1,4-difluorobenzene, and 2-bromo-1-chloropropane at a concentration of 15 µg/mL using stock standards as described in Item 3 above. Replace the internal standards solution every 6 months.

8. Bromofluorobenzene (BFB) standard - Prepare a 25-µg/mL solution of BFB in methanol as described in Item 4 above. Replace the bromofluorobenzene standard solution every 6 months.

9. Store all standard solutions at -10° to -20°C.

Preparation of Glassware:

All glassware is washed with soapy water, rinsed with tap water, then rinsed with deionized water, and baked in a drying oven for at least 4 hours.

Safety Precautions:

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; therefore, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as the use of fume hoods, lab coats, gloves, and safety glasses.

Sample Handling:

The samples to be analyzed using EPA Method 624, should be stored in a refrigerator between 2° and 6°C. They should be preserved to a pH of <2 in order to prevent degradation of aromatic compounds that may be present in the sample. 1 + 1 HCl is the recommended preservative. In addition, all samples must be analyzed within 14 days of collection. Unpreserved samples must be run within 7 days of collection.

Procedure:

1. The purge and trap device should have the trap conditioned at 180° to 220°C at a flow rate of 20 to 60 mL/min prior to initial use.

2. An example of typical purge and trap conditions are listed below:

Purge gas	Helium
Purge flow	35 mL/min
Purge temperature	Ambient temperature
Purge time	11 min
Desorb temperature	220°C
Desorb time	4 min
Bake temperature	180°C
Bake time	8 min

Purge and trap conditions may be changed to optimize instrument operations. A record of actual purge and trap conditions for each instrument may be found in the appropriate instrument maintenance log.

3. The gas chromatograph must be operated using conditions equivalent to those listed below:

	Packed Column	Capillary Column
Column packing	SP-1000 on Carbopack B	DB-624
Column dimensions	6 ft. × 0.1 in. ID	75 m, 0.53-mm ID 3-μm film thickness
Carrier gas	Helium	Helium
Carrier gas flow	30 mL/min	9 to 10 mL/min
Makeup gas flow		20 mL/min
Initial temperature	45°C	35°C
Initial hold time	3 min	5 min
Temperature ramp	8°C/min	10°C/min
Final temperature	220°C	180°C
Final hold time	15 min	3 min or until all targ compounds elute

4. The suggested mass spectrometer operating conditions are listed below:

Ions	Positive
Electron energy	70 volts
Mass range	35 to 300 amu
Scan time	To give at least 5 scans per peak but not to exceed 7 s per scan

5. Tune the GC/MS system to meet the criteria in Table 2 following a 2-μL direct injection of BFB. The chromatographic conditions must be the same as those under which the samples will be analyzed except that the initial temperature and the temperature ramp may be increased. The BFB tune must be verified every 24 hours.

6. Internal standard calibration consists of analyzing standards at five distinct levels of analyte and producing response factors for each compound. The relative standard deviation of the response factors determine the suitability of the average relative response factor for calculation of the analyte concentration.

   a. All aqueous matrices should be prepared in the following manner. Remove the plunger from a 5 mL glass Luer-Lok syringe and fill the syringe barrel to overflowing with aqueous matrix. Replace the plunger and compress the aqueous matrix such that no air is trapped in the syringe. Adjust the syringe volume to 5.0 mL. Add an appropriate volume of the surrogate spiking solution and the internal standard solution to the syringe through the syringe tip.

   b. Add the spiked aqueous matrix to the purge vessel through the Luer-Lok assembly at the top of the impinging assembly. Do these steps quickly to minimize the loss of volatiles from the aqueous matrix and to minimize the possibility of airborne contamination of the aqueous matrix.

   c. Prior to loading another aqueous matrix into the vessel, empty and rinse the purging chamber twice with reagent water to minimize the possibility of carryover contamination.

   d. Continue collecting GC/MS data until the end of the GC run.

   e. Prepare the calibration standards at the levels of 4, 20, 50, 100, and 300 μg/L.

Due to poor purging efficiency or poor chromatography, certain analytes (Tables 1A through 1F) require calibration at higher concentrations. To prevent confusion and assure proper calibration, a Theoretical Standard Concentration (TSC) sheet is completed for each calibration (Figure 2).

The TSC sheet contains the theoretical concentration for each analyte in the calibration at each of the five levels.

f. Each level is analyzed as described above by substituting 5 mL of the calibration standard as the aqueous matrix (see Items a. through d. above).

Surrogate standard compounds should be at the standard concentration level for each initial calibration standard. The amount of surrogate standard spiking solution for each calibration standard is listed on Figure 2.

g. Next tabulate the area response of the characteristic ions against concentration for each analyte, surrogate standard, and internal standard, and calculate relative response factors (RRF) for each compound using the calculation below:

$$RRF = \frac{Ax}{Ais} \times \frac{Cis}{Cx}$$

Where:

Ax = Area of the characteristic ion for the analyte (Table 5)

Ais = Area of the characteristic ion of the appropriate internal standard (Table 5)

Cis = Concentration of the internal standard

Cx = Concentration of the analyte to be measured

h. Calculate the average relative response factors for all analytes. The %RSD for all analytes should be less than or equal to 35%. A first or second order regression curve may be generated and used if the %RSD for an analyte exceeds 35%. Failure to meet the criteria for any of the compounds invalidates the calibration. The calibration must be redone after proper corrective action to the system is taken.

i. The average RRF values for the initial calibration are used to calculate values for calibration check standards, method blanks, and sample analyses. The calibration is valid for 24 hours, at which time a tune check and a calibration check must occur.

j. The calibration check involves analyzing the 20-μg/L standard and comparing it to the values listed in Table 4. If any of the compounds fail to meet the criteria listed, the calibration is considered invalid. Corrective action must be taken and the calibration redone. If the criteria are met, the average RRF is used for the sample calculations during the 24-hour tune period. At the end of the tune period another tune check and calibration check must be done.

7. A method blank is then analyzed using reagent water as the aqueous matrix. (See steps 6.a. through 6.d.) The method blank is examined for interfering peaks. Any target compound peaks are calculated as described under the Calculations section of this procedure. Methylene chloride and toluene must not exceed five times the quantitation limit in the blank. All other compounds may not exceed the quantitation limit. If the blank values exceed these values, corrective action must be taken and the method blank reanalyzed until the criteria are met. Also, all surrogate compound recoveries must meet the criteria listed in Table 3.

8. Sample analysis

a. Sample analysis for water and wastewater proceeds using the sample as the aqueous matrix. (See steps 6.a. through 6.d.)

b. For each set of 20 field samples, one sample shall be analyzed in triplicate, spiking the duplicate and triplicate with 10 µL of the matrix spiking solution(s). Results of the unspiked and spiked duplicate and triplicate shall be compared to Table 4.

Due to poor purging efficiency or poor chromatography, certain analytes require spiking at higher concentrations. To prevent confusion and assure proper recovery calculations, a TSC sheet is completed for each set of matrix spikes analyzed (Figure 3). The TSC sheet contains the theoretical concentration for each analyte in the matrix spike, matrix spike duplicate, and QC reference sample.

c. All samples must also meet the surrogate recoveries listed in Table 3. If a sample surrogate recovery falls outside the criteria, the sample should be reanalyzed. If the reanalysis shows the same surrogate results, the sample matrix is assumed to be interfering and the initial results are reported. If the reanalysis meets the recovery criteria, the first analysis is assumed to have been outside of limits due to a laboratory error and the second analysis is reported.

9. Data analysis

a. Sample chromatograms are analyzed both qualitatively and quantitatively. First, a determination of the identity of a sample peak as a compound (Table 1) is made through the use of computerized analysis. Guidelines for the qualitative determination are as follows.

(1) The relative retention time (RRT) of the sample peak is within 0.06 of the RRT of the most recent check standard.

(2) Each ion with a relative intensity of greater than 10% of the base ion of the mass spectra of the standard must be present in the sample spectrum produced on the same mass spectrometer.

(3) The relative intensities of the ions in the sample mass spectrum must be within 20% of the intensities of the standard mass spectrum.

(4) Peaks greater than 10% relative intensity in the sample mass spectrum, but not present in the standard mass spectrum from the same mass spectrometer, must be accounted for by the analyst.

(5) If a compound fails any of the criteria listed above but, in the judgment of the mass spectral interpretation specialist, is a correct identification, the identification is used and the quantitation of the peak is performed.

b. Quantitation of identified target compounds is performed using the equations listed in the Calculations section of this procedure. All calculations should report concentrations in values of µg/L. Any analyte with a calculated concentration above the highest calibration standard must be reanalyzed at a dilution which should bring the concentration in solution into the upper half of the calibration curve.

Calculations:

$$Concentration\ (\mu g/L) = \frac{(Ax)\,(Is)}{(Ais)\,(RRF)\,(Vo)}$$

Where:

Ax = Area of the quantitation ion peak for the compound to be measured

Ais = Area of the quantitation ion peak for the appropriate internal standard

Is = Amount of internal standard added in nanograms

Vo = Volume of sample purged

RRF = Relative response factor from the current initial calibration

Quality Assurance:

1. The GC/MS system must be tuned to meet the criteria in Table 2 following the BFB injection. The chromatographic conditions must be the same as those under which the samples will be analyzed except that the initial temperature and rate of temperature ramping may be increased. The BFB tune must be verified every 24 hours.

2. Internal standard calibration requires analysis of a minimum of three levels of analyte concentration. Response factors for each compound are calculated. The relative standard deviation of the response factors determine the suitability of the average relative response factor for calculation of the analyte concentration.

   a. All compounds are evaluated on the basis of the percent relative standard deviation of the RRF values (%RSD). %RSD values on the initial calibration must be less than 35%.

   b. Alternatively, a first or second order regression curve may be used if the correlation coefficient achieves a minimum value of 0.995.

   c. For continuing calibration to be valid, the 20-μg/L check must meet the criteria listed in Table 4.

3. Surrogate recoveries are calculated for each sample and blank analyzed. Surrogate recovery limits are listed in Table 3. If any surrogate is outside of limits, the sample is to be reanalyzed. If surrogates in the reanalysis are within limits, the reanalysis data are reported. If surrogate recoveries are again outside of limits on the reanalysis, the first analysis is reported.

4. The method blank must meet the above criteria for surrogate recoveries. In addition, the method blank may not contain any target compound above the quantitation limit except that methylene chloride and toluene may be present up to five times the quantitation limit. All method blanks must meet these criteria; otherwise, the system is considered out of control and corrective action must be taken.

5. The matrix spike and matrix spike duplicate pair are analyzed for each set of 20 field samples. The recoveries and relative percent differences of the recoveries for each spiked compound are calculated and compared to the values in Table 4 or from the statistical results of multiple analysis of the spiked samples. The results of the matrix spike/matrix spike duplicate must be within the windows outlined in Table 4. For samples with results outside the statistical windows, analysis of a QC reference sample is required. The QC reference sample is a method blank spiked with the appropriate volume of the matrix spiking solutions which yields a known concentration of all analytes (Figure 3). The QC reference sample must yield recoveries for all analytes within the specification of Table 4. If the QC reference sample fails to meet the criteria, the analysis must stop and the cause of the problem found. If the analysis meets the values in the table, the results are reported.

6. All data is reviewed by a data review specialist with respect to quality assurance and data interpretation prior to release of the data from the analytical laboratory. The data review specialist will return data to the analyst for correction as necessary. The specialist will also report the results of the review and corrections on a form to be kept with the raw data. Any noncompliant data that cannot be corrected will be referred to the group leader or supervisor for further action.

Additional Analyses:

The following analyses are run by EPA Method 624: 0520, 1131, 1387, 1388, 4601, 4604, 6374, and 6376. Any extra compounds should be added to the theoretical standards concentration sheets when performing these analyses.

Method Modification:

EPA Method 624 indicates use of packed-column chromatography. While packed-column chromatography may be used, capillary-column chromatography will often be used for EPA Method 624 analyses. Capillary-column chromatography is equivalent to packed-column chromatography in its ability to meet method requirements.

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Prepared by: Alita Nelly Date: 3/10/97

Approved by: Duane A Suchsdell Date: 3/10/97

Approved by: Susan S. Shorta Date: 3/10/97

Table 1A

Practical Quantitation Limits (PQL) for Volatile Organics
Analysis #0520

Compounds	Practical Quantitation Limits Water/Wastewater (ug/L)
Acrolein	100
Acrylonitrile	50
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	5
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	5
2-Chloroethyl vinyl ether	10
Chloroform	5
Chloromethane	5
Dibromochloromethane	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
cis-1,2-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	5
Vinyl chloride	5

The PQLs listed herein are provided for guidance and may not always be achievable.

Table 1B

Practical Quantitation Limits (PQL) for Volatile Organics
Analysis #1131

Compounds	Practical Quantitation Limits Water/Wastewater (µg/L)
Benzene	5
Toluene	5
Ethylbenzene	5
Methyl Tertiary Butyl Ether	5
Tertiary Butyl Alcohol	100
Xylene (total)	5

The PQLs listed herein are provided for guidance and may not always be achievable.

Table 1C

Practical Quantitation Limits (PQL) for Volatile Organics
Analysis #1387

Compounds	Practical Quantitation Limits Water/Wastewater (µg/L)
Acrolein	100
Acrylonitrile	50
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	5
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	5
2-Chloroethyl vinyl ether	10
Chloroform	5
Chloromethane	5
Dibromochloromethane	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
cis-1,2-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	5
Vinyl chloride	5
t-Butyl alcohol	100
Methyl t-butyl ether	5
Xylene (total)	5

The PQLs listed herein are provided for guidance and may not always be achievable.

Table 1D

Practical Quantitation Limits (PQL) for Volatile Organics
Analysis #1388

Compounds	Practical Quantitation Limits Water/Wastewater (µg/L)
Acrolein	100
Acrylonitrile	50
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	5
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	5
2-Chloroethyl vinyl ether	10
Chloroform	5
Chloromethane	5
Dibromochloromethane	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
cis-1,2-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	5
Vinyl chloride	5
Xylene (total)	5

The PQLs listed herein are provided for guidance and may not always be achievable.

Table 1E

Practical Quantitation Limits (PQL) for Volatile Organics
Analysis #4601

Compounds	Practical Quantitation Limits Water/Wastewater (µg/L)
Benzene	5
Toluene	5
Ethylbenzene	5
Xylene (total)	5

The PQLs listed herein are provided for guidance and may not always be achievable.

Table 1F

Practical Quantitation Limits (PQL) for Volatile Organics
Analysis #4604

Compounds	Practical Quantitation Limits Water/Wastewater (µg/L)
Chloromethane	5
1,2-Dichloroethane	5
1,2-Dichloropropane	5
Tetrachloroethene	5
Toluene	5
Chlorobenzene	5
Ethylbenzene	5

The PQLs listed herein are provided for guidance and may not always be achievable.

Table 1G

Practical Quantitation Limits (PQL) for Volatile Organics
Analysis #6374

Compounds	Practical Quantitation Limits Water/Wastewater (µg/L)
Trichlorofluoro methane	5
Acrolein	100
Acrylonitrile	50
2-Chloroethyl Vinyl Ether	10
Chloromethane	5
Bromoethane	5
Vinyl Chloride	5
Chloroethane	5
Methylene Chloride	5
1,1-Dichloroethene	5
1,1-Dichloroethane	5
Chloroform	5
1,2-Dichloroethane	5
1,1,1-Trichloroethane	5
Carbon Tetrachloride	5
Bromodichloromethane	5
1,2-Dichloropropane	5
trans-1,3-Dichloropropene	5
Trichloroethene	5
Benzene	5
cis-1,3-Dichloropropene	5
1,1,2-Trichloroethane	5
Dibromochloromethane	5
Bromoform	5
Tetrachloroethene	5
1,1,2,2-Tetrachloroethane	5
Toluene	5
Chlorobenzene	5
Ethylbenzene	5
trans-1,2-Dichloroethene	5
Xylene (total)	5
Acetone	20
Carbon Disulfide	5
2-Butanone	10
Vinyl Acetate	10
4-Methyl-2-pentanone	10
2-Hexanone	10
Styrene	5

The PQLs listed herein are provided for guidance and may not always be achievable.

Table 1H

Practical Quantitation Limits (PQL) for Volatile Organics
Analysis #6376

Compounds	Practical Quantitation Limits Water/Wastewater (µg/L)
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
1,2-Dichlorobenzene	5

The PQLs listed herein are provided for guidance and may not always be achievable.

Table 2

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of mass 95
75	30% to 60% of mass 95
95	base peak, 100% relative abundance
96	5% to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5% to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5% to 9% of mass 176

Table 3

Surrogate Spike Recovery Limits for Water and Wastewater Samples

Surrogate Compound	WateWastewater
4-Bromofluorobenzene	86-115
1,2-Dichloroethane-d4	76-114
Fluorobenzene	80-120

Table 4

Calibration and QC Acceptance Criteria[a]

Parameter	Range for Q (µg/L)	Range p, p2 (%)
Acrolein	Not defined	22 - 169
Acrylonitrile	Not defined	51 - 138
Benzene	12.8 - 27.2	37 - 151
Bromodichloromethane	13.1 - 26.9	35 - 155
Bromoform	14.2 - 25.8	45 - 169
Bromomethane	2.8 - 37.2	D - 242
Carbon tetrachloride	14.6 - 25.4	70 - 140
Chlorobenzene	13.2 - 26.8	37 - 160
Chloroethane	7.6 - 32.4	14 - 230
2-Chloroethyl vinyl ether	D - 44.8	D - 305
Chloroform	13.5 - 26.5	51 - 138
Chloromethane	D - 40.8	D - 273
Dibromochloromethane	13.5 - 26.5	53 - 149
1,1-Dichloroethane	14.5 - 25.5	59 - 155
1,2-Dichloroethane	13.6 - 26.4	49 - 155
1,1-Dichloroethene	10.1 - 29.9	D - 234
cis-1,2-Dichloroethene	13.9 - 26.1	54 - 156
trans-1,2-Dichloroethene	13.9 - 26.1	54 - 156
1,2-Dichloropropane	6.8 - 33.2	D - 210
cis-1,3-Dichloropropene	4.8 - 35.2	D - 227
trans-1,3-Dichloropropene	10.0 - 30.0	17 - 183
Ethylbenzene	11.8 - 28.2	37 - 162
Methylene chloride	12.1 - 27.9	D - 221
1,1,2,2-Tetrachloroethane	12.1 - 27.9	46 - 157
Tetrachloroethene	14.7 - 25.3	64 - 148
Toluene	14.9 - 25.1	47 - 150
1,1,1-Trichloroethane	15.0 - 25.0	52 - 162
1,1,2-Trichloroethane	14.2 - 25.8	52 - 150
Trichloroethene	13.3 - 26.7	71 - 157
Trichlorofluoromethane	9.6 - 30.4	17 - 181
Vinyl chloride	0.8 - 39.2	D - 251
t-Butyl alcohol	Not defined	25 - 195
Methyl t-butyl ether	Not defined	80 - 123
Xylene (total)	Not defined	61 - 165

Q = Concentration measured in QC check sample in µg/L.
p, p2 = Percent recovery measured (20 µg/L spike)

[a]Criteria from 40 CRF Part 136 for Method 624 and were calculated assuming a QC check sample concentration of 20 µg/L.

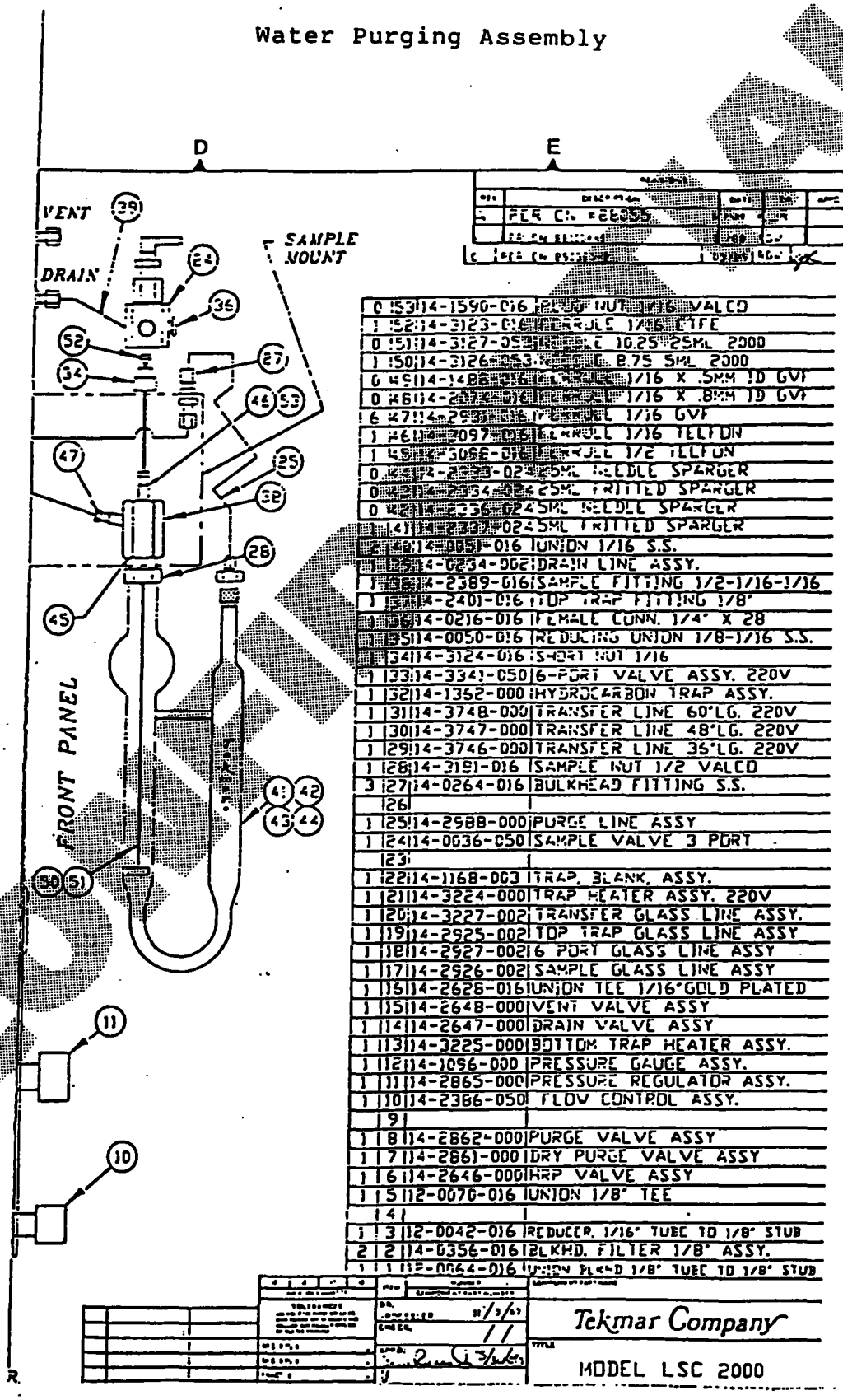
cis-1,3-Dichloropropene and *trans*-1,3-Dichloropropene are available only as an isomeric mixture. Thus the amount of *trans*-1,3,-Dichloropropene in standards, QC check samples, and matrix spikes will vary.

Table 5

Compound	Primary m/z	Secondary Masses
4-Bromofluorobenzene (SS)	95	174,176
1,2-Dichloroethane d-4 (SS)	102	
Bromochloromethane (IS)	128	49,130,51
Chlorobenzene-d5 (IS)	117	82,119
1,4-Difluorobenzene (IS)	114	63,88
Toluene-d8 (SS)	98	70,100
Chloromethane	50	52
Bromomethane	94	96
Vinyl chloride	62	64
Chloroethane	64	66
Methylene chloride	84	49,51,86
Trichlorofluoromethane	101	103
1,1-Dichloroethene	96	61,98
1,1-Dichloroethane	63	65,83,85,98,100
cis-1,2-Dichloroethene	96	61,98
trans-1,2-Dichloroethene	96	61,98
Chloroform	83	85
1,2-Dichloroethane	98	62,64,100
1,1,1-Trichloroethane	97	99,117,119
Carbon tetrachloride	117	119,121
Bromodichloromethane	127	83,85,129
1,2-Dichloropropane	112	63,65,114
trans-1,3-Dichloropropene	75	77
Trichloroethene	130	95,97,132
Benzene	78	
Dibromochloromethane	127	129,208,206
1,1,2-Trichloroethane	97	83,85,99,132,134
cis-1,3-Dichloropropene	75	77
2-Chloroethyl vinyl ether	106	63,65
Bromoform	173	171,175,250,252,254,256
1,1,2,2-Tetrachloroethane	168	83,85,131,133,166
Tetrachloroethene	164	129,131,166
Toluene	92	91
Chlorobenzene	112	114
Ethylbenzene	106	91
Acrolein	56	55,58
Acrylonitrile	53	52,51
t-Butyl alcohol	59	41
Methyl t-butyl ether	73	57
Xylene (total)	106	91

Figure 1

Water Purging Assembly



0	53	14-1590-016	PLUG NUT 1/16 VALCO	
1	52	14-3123-016	FERRULE 1/16 TFE	
0	51	14-3127-052	[illegible] 10.25" 25ML 2000	
1	50	14-3126-063	[illegible] 8.75 5ML 2000	
0	49	14-1488-016	[illegible] 1/16 X .5MM ID GVF	
0	48	14-2074-016	[illegible] 1/16 X .8MM ID GVF	
6	47	14-2930-016	FERRULE 1/16 GVF	
1	46	14-2097-016	FERRULE 1/16 TELFON	
1	45	14-3088-016	FERRULE 1/2 TELFON	
0	44	14-2333-024	25ML NEEDLE SPARGER	
0	43	14-2334-024	25ML FRITTED SPARGER	
0	42	14-2336-024	5ML NEEDLE SPARGER	
1	41	14-2337-024	5ML FRITTED SPARGER	
2	40	14-0851-016	UNION 1/16 S.S.	
1	39	14-0234-002	DRAIN LINE ASSY.	
1	38	14-2389-016	SAMPLE FITTING 1/2-1/16-1/16	
1	37	14-2401-016	TOP TRAP FITTING 1/8"	
1	36	14-0216-016	FEMALE CONN. 1/4" X 28	
1	35	14-0050-016	REDUCING UNION 1/8-1/16 S.S.	
1	34	14-3124-016	SHORT NUT 1/16	
1	33	14-3341-050	6-PORT VALVE ASSY. 220V	
1	32	14-1362-000	HYDROCARBON TRAP ASSY.	
1	31	14-3748-000	TRANSFER LINE 60"LG. 220V	
1	30	14-3747-000	TRANSFER LINE 48"LG. 220V	
1	29	14-3746-000	TRANSFER LINE 36"LG. 220V	
1	28	14-3191-016	SAMPLE NUT 1/2 VALCO	
3	27	14-0264-016	BULKHEAD FITTING S.S.	
	26			
1	25	14-2988-000	PURGE LINE ASSY	
1	24	14-0036-050	SAMPLE VALVE 3 PORT	
	23			
1	22	14-1168-003	TRAP, BLANK, ASSY.	
1	21	14-3224-000	TRAP HEATER ASSY. 220V	
1	20	14-3227-002	TRANSFER GLASS LINE ASSY.	
1	19	14-2925-002	TOP TRAP GLASS LINE ASSY	
1	18	14-2927-002	6 PORT GLASS LINE ASSY	
1	17	14-2926-002	SAMPLE GLASS LINE ASSY	
1	16	14-2628-016	UNION TEE 1/16"GOLD PLATED	
1	15	14-2648-000	VENT VALVE ASSY	
1	14	14-2647-000	DRAIN VALVE ASSY	
1	13	14-3225-000	BOTTOM TRAP HEATER ASSY.	
1	12	14-1096-000	PRESSURE GAUGE ASSY.	
1	11	14-2865-000	PRESSURE REGULATOR ASSY.	
1	10	14-2386-050	FLOW CONTROL ASSY.	
	9			
1	8	14-2862-000	PURGE VALVE ASSY	
1	7	14-2861-000	DRY PURGE VALVE ASSY	
1	6	14-2646-000	HRP VALVE ASSY	
1	5	12-0070-016	UNION 1/8" TEE	
	4			
1	3	12-0042-016	REDUCER, 1/16" TUBE TO 1/8" STUB	
2	2	14-0356-016	BLKHD. FILTER 1/8" ASSY.	
1	1	12-0064-016	UNION PLATED 1/8" TUBE TO 1/8" STUB	

Tekmar Company

MODEL LSC 2000

Figure 2

THEORETICAL STANDARD CONCENTRATIONS
INITIAL CALIBRATION
PURCHASED STANDARDS
HP CAPILLARY COLUMN
EPA METHOD 624

DATE:

INSTRUMENT:

VOA1 = 1 to 10 dilution of CS#1, CS#2 and CS#4
VOA3 = 1 to 10 dilution of CS#3
VOA2 = 1 to 10 dilution of CS#2

stock mix name	VOA1	VOA3	VOA2	VOA4	Restek GASES (2000 ppm) Lt#	FLASK ml
300 ppb std	37.5 ul	15 ul		7.5 ul	3.75 ul	25
100 ppb std	25 ul	10 ul		5 ul	2.5 ul	50
50 ppb std	25 ul	10 ul		5 ul	2.5 ul	100
20 ppb std	2.0 ml of 50 ppb std into 5 ml syringe					
4 ppb std	20 ul	8 ul	60 ul	4 ul	2.0 ul	200*

*Dilute 1.0 ml of 200 ml flask into 5 ml syringe

compound name	std mix	stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	4 ppb
Benzene	CS	2000	300	100	50	20	4
Bromobenzene	#1	2000	300	100	50	20	4
Bromodichloromethane		2000	300	100	50	20	4
Bromoform		2000	300	100	50	20	4
n-Butylbenzene		2000	300	100	50	20	4
sec-Butylbenzene		2000	300	100	50	20	4
tert-Butylbenzene		2000	300	100	50	20	4
Carbon tetrachloride		2000	300	100	50	20	4
Chlorobenzene		2000	300	100	50	20	4
Chloroform		2000	300	100	50	20	4
2-Chlorotoluene		2000	300	100	50	20	4
4-Chlorotoluene		2000	300	100	50	20	4
Dibromochloromethane		2000	300	100	50	20	4
1,2-Dibromo-3-chloropropane		2000	300	100	50	20	4
1,2-Dibromoethane (EDB)		2000	300	100	50	20	4
Dibromomethane		2000	300	100	50	20	4
1,2-Dichlorobenzene		2000	300	100	50	20	4
1,3-Dichlorobenzene		2000	300	100	50	20	4
1,4-Dichlorobenzene		2000	300	100	50	20	4
1,1-Dichloroethane		2000	300	100	50	20	4
1,2-Dichloroethane		2000	300	100	50	20	4
1,1-Dichloroethene		2000	300	100	50	20	4
cis-1,2-Dichloroethene		2000	300	100	50	20	4
trans,1,2-Dichloroethene		2000	300	100	50	20	4

Figure 2 (Continued)

EPA METHOD 624
INITIAL CALIBRATION

compound name	std mix	stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	4 ppb
1,2-Dichloropropane	CS	2000	300	100	50	20	4
1,3-Dichloropropane	#1	2000	300	100	50	20	4
2,2-Dichloropropane		2000	300	100	50	20	4
1,1-Dichloropropene		2000	300	100	50	20	4
cis-1,3-Dichloropropene		2000	300	100	50	20	4
trans-1,3-Dichloropropene		2000	300	100	50	20	4
Ethylbenzene		2000	300	100	50	20	4
Hexachlorobutadiene		2000	300	100	50	20	4
Isopropylbenzene (Cumene)		2000	300	100	50	20	4
p-Isopropyltoluene		2000	300	100	50	20	4
Methylene Chloride		2000	300	100	50	20	4
Naphthalene		2000	300	100	50	20	4
n-Propylbenzene		2000	300	100	50	20	4
Styrene		2000	300	100	50	20	4
1,1,1,2-Tetrachloroethane		2000	300	100	50	20	4
1,1,2,2-Tetrachloroethane		2000	300	100	50	20	4
Tetrachloroethene		2000	300	100	50	20	4
Toluene		2000	300	100	50	20	4
1,2,3-Trichlorobenzene		2000	300	100	50	20	4
1,2,4-Trichlorobenzene		2000	300	100	50	20	4
1,1,1-Trichloroethane		2000	300	100	50	20	4
1,1,2-Trichloroethane		2000	300	100	50	20	4
Trichloroethene		2000	300	100	50	20	4
1,2,3-Trichloropropane		2000	300	100	50	20	4
1,2,4-Trimethylbenzene		2000	300	100	50	20	4
1,3,5-Trimethylbenzene		2000	300	100	50	20	4
m-Xylene		2000	300	100	50	20	4
o-Xylene		2000	300	100	50	20	4
p-Xylene		2000	300	100	50	20	4
Bromomethane	GAS	2000	300	100	50	20	4
Chloroethane	MIX	2000	300	100	50	20	4
Chloromethane		2000	300	100	50	20	4
Dichlorodifluoromethane		2000	300	100	50	20	4
Trichlorofluoromethane		2000	300	100	50	20	4
Vinyl Chloride		2000	300	100	50	20	4
Methacrylonitrile	CS	5000	750	250	125	50	40
Propionitrile	#2	10000	1500	500	250	100	80
trans-1,4-Dichloro-2-Buten		5000	750	250	125	50	40
t-Butyl Alcohol		10000	1500	500	250	100	80
2-Propanol		10000	1500	500	250	100	80
Isobutyl Alcohol		25000	3750	1250	625	250	200
n-Butanol		25000	3750	1250	625	250	200
1-Propanol		25000	3750	1250	625	250	200
1,4-Dioxane		25000	3750	1250	625	250	200
Cyclohexanone		25000	3750	1250	625	250	200
Monochloroacetone		25000	3750	1250	625	250	200
2-Butanone	CS	10000	600	200	100	40	8
2-Hexanone	#3	10000	600	200	100	40	8
4-Methyl-2-Pentanone		10000	600	200	100	40	8
Acetone		10000	600	200	100	40	8
Acrolein		50000	3000	1000	500	200	40
Acrylonitrile		50000	3000	1000	500	200	40
2-Chloroethyl-vinyl-ether		10000	600	200	100	40	8
2-Nitropropane		10000	600	200	100	40	8

Figure 2 (Continued)

EPA METHOD 624
INITIAL CALIBRATION

compound name	std mix	stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	4 ppb
Methyl-t-butyl Ether	CS	2000	300	100	50	20	4
t-Butyl Formate	#4	2000	300	100	50	20	4
Ethyl Methacrylate		2000	300	100	50	20	4
Methyl Methacrylate		2000	300	100	50	20	4
Ethyl Ether		2000	300	100	50	20	4
Freon 113		2000	300	100	50	20	4
Hexane		2000	300	100	50	20	4
Heptane		2000	300	100	50	20	4
Cyclohexane		2000	300	100	50	20	4
Benzyl Chloride		2000	300	100	50	20	4
Isopropyl Acetate		2000	300	100	50	20	4
Methyl Iodide		2000	300	100	50	20	4
Carbon Disulfide		2000	300	100	50	20	4
Vinyl Acetate		2000	300	100	50	20	4
n-Propyl Acetate		2000	300	100	50	20	4
Tetrahydrofuran		2000	300	100	50	20	4
2-Chloro-1,3-butadiene		2000	300	100	50	20	4
Pentachloroethane	VOA4	1000	300	100	50	20	4
Allyl Chloride		1000	300	100	50	20	4

ppb of analytical standard = stock ppm x ul stock / flask ml

Calibration Standard Conc. (ppb)	300	100	50	20	4
IH624SS (100 ug/mL) (ul)*	15	5	---	---	---
IL624SS (10 ug/mL) (ul)*	----	---	25	10	2

Note: The surrogate used for spiking samples is 624SS (15 ug/mL) and is spiked at 10 ul directly into the syringe.

ANALYST_____

DATE_____

(saved as file NEW624::DB) editted 06/19/96

Figure 3

THEORETICAL STANDARD CONCENTRATIONS
QUALITY CONTROL
PURCHASED STANDARDS
HP CAPILLARY COLUMN
EPA SW846 METHOD 8240B AND
EPA METHOD 624

DATE:

INSTRUMENT:

QVOA1 = 1 to 20 dilution of QCS#1, QCS#2 QCS#3 and QCS#4

stock mix name	QVOA1 _____	QVOA4 _____	Restek Q Gas Mix Lt#_____	SYRINGE ml
20 ppb std	10 ul	5 ul	5 ul	5

compound name	std mix	stock ppm	20 ppb
Benzene	QCS #1	200	20
Bromobenzene		200	20
Bromodichloromethane		200	20
Bromoform		200	20
n-Butylbenzene		200	20
sec-Butylbenzene		200	20
tert-Butylbenzene		200	20
Carbon tetrachloride		200	20
Chlorobenzene		200	20
Chloroform		200	20
2-Chlorotoluene		200	20
4-Chlorotoluene		200	20
Dibromochloromethane		200	20
1,2-Dibromo-3-chloropropane		200	20
1,2-Dibromoethane (EDB)		200	20
Dibromomethane		200	20
1,2-Dichlorobenzene		200	20
1,3-Dichlorobenzene		200	20
1,4-Dichlorobenzene		200	20
1,1-Dichloroethane		200	20
1,2-Dichloroethane		200	20
1,1-Dichloroethene		200	20
cis-1,2-Dichloroethene		200	20
trans,1,2-Dichloroethene		200	20

Figure 3 (Continued)

EPA SW846 METHOD 8240B
QC

compound name	std mix	stock ppm	20 ppb
1,2-Dichloropropane	QCS	200	20
1,3-Dichloropropane	#1	200	20
2,2-Dichloropropane		200	20
1,1-Dichloropropene		200	20
cis-1,3-Dichloropropene		200	20
trans-1,3-Dichloropropene		200	20
Ethylbenzene		200	20
Hexachlorobutadiene		200	20
Isopropylbenzene (Cumene)		200	20
p-Isopropyltoluene		200	20
Methylene Chloride		200	20
Naphthalene		200	20
n-Propylbenzene		200	20
Styrene		200	20
1,1,1,2-Tetrachloroethane		200	20
1,1,2,2-Tetrachloroethane		200	20
Tetrachloroethene		200	20
Toluene		200	20
1,2,3-Trichlorobenzene		200	20
1,2,4-Trichlorobenzene		200	20
1,1,1-Trichloroethane		200	20
1,1,2-Trichloroethane		200	20
Trichloroethene		200	20
1,2,3-Trichloropropane		200	20
1,2,4-Trimethylbenzene		200	20
1,3,5-Trimethylbenzene		200	20
m-Xylene		200	20
o-Xylene		200	20
p-Xylene		200	20
Bromomethane	GAS	20	20
Chloroethane	MIX	20	20
Chloromethane		20	20
Dichlorodifluoromethane		20	20
Trichlorofluoromethane		20	20
Vinyl Chloride		20	20
Methacrylonitrile	QCS	1500	150
Propionitrile	#2	1500	150
trans-1,4-Dichloro-2-Buten		1500	150
t-Butyl Alcohol		2000	200
2-Propanol		1500	150
Isobutyl Alcohol		5000	500
n-Butanol		5000	500
1-Propanol		5000	500
1,4-Dioxane		5000	500
Cyclohexanone		5000	500
Monochloroacetone		5000	500
2-Butanone	QCS	1500	150
2-Hexanone	#3	1500	150
4-Methyl-2-Pentanone		1000	100
Acetone		1000	100
Acrolein		1500	150
Acrylonitrile		1500	150
2-Chloroethyl-vinyl-ether		200	20
2-Nitropropane		200	20

Figure 3 (Continued)

EPA SW846 METHOD 8240B
QC

compound name	std mix	stock ppm	20 ppb
Methyl-t-butyl Ether	QCS	200	20
t-Butyl Formate	#4	200	20
Ethyl Methacrylate		200	20
Methyl Methacrylate		200	20
Ethyl Ether		200	20
Freon 113		200	20
Hexane		200	20
Heptane		200	20
Cyclohexane		200	20
Benzyl Chloride		200	20
Isopropyl Acetate		200	20
Methyl Iodide		200	20
Carbon Disulfide		1500	150
Vinyl Acetate		1500	150
n-Propyl Acetate		200	20
Tetrahydrofuran		200	20
2-Chloro-1,3-butadiene		200	20
Pentachloroethane	Q	20	20
Allyl Chloride	VOA4	20	20

ppb of analytical standard = stock ppm x ul stock / syringe ml

ANALYST_____

DATE_____

page 3 of 3

(saved as file QC824N::DB) editted 07/24/96

CONFIDENTIAL

Procedural Amendment #1

Number: Analysis #0520, 1131, 1387, 1388, 4601, 4604, 6374, 6376

Title: The Analysis of Water for Purgeable Organics by Purge and Trap Gas Chromatography/Mass Spectrometry

Effective Date (listed on procedure): 03/10/97

Section(s) affected by change: Apparatus

Reason for addition(s) or change(s): Audit response

Change will be effective from (date): 10/06/97

Samples or project affected: All

List change(s) or addition(s) (specify which section):

Apparatus: (*Add as second sentence*)

5. The purging chamber should have the purge gas passing through the sample as finely divided bubbles and minimize the gaseous headspace between the sample and the trap to <15 mL.

05201131.DOC
100797

Prepared by: Robert E. Mellinger Date: 10/9/97

Approved by: Jeanne A. Fuchsbill Date: 10/9/97

Approved by: Susan B. Shorter Date: 10/10/97

CONFIDENTIAL

Analysis #0552, 0553, 0554, 7357, 7358
Revision 01
Supersedes Date: 04/15/96
Effective Date: MAY 06 1997

Determination of Priority Pollutants by Method 625

Reference:

1. *Federal Register*, Friday, October 26, 1984, pp. 153 - 162.
2. Method 3510A, SW 846, USEPA, 3rd Edition, July 1992.
3. Method 3520A, SW 846, USEPA, 3rd Edition, July 1992.
4. *Hewlett-Packard Operations Manuals.*

Scope:

This method is suitable for the determination of the concentration of priority pollutants in waters. The list of compounds is presented in Table I. The limit of quantitation (LOQ) for most semivolatile compounds in a solution of methylene chloride is 10.0 µg/L. However, the LOQ is highly dependent on the sample matrix. Interferences may also be caused by solvent contamination, reagent contamination, or glassware contamination. These sources of interference may lead to false-positive identification of target compounds in the sample. A method blank must be analyzed to ensure that all materials are free of contamination.

Personnel Training and Qualifications:

Education Requirement: Degree in science or relevant experience

Each new chemist will train with an experienced chemist for the first 12 weeks depending on the individual and his or her previous experience. The first 12 weeks is spent working one-on-one with the trainer. This time may be less if the new chemist has prior experience in the GC/MS Semivolatiles area. Each new chemist receives a training manual outlining the basis of operating the GC/MS and data work up.

During the training period, the new chemist will learn daily maintenance, column, and source changing procedures, calibration techniques, data and library search review, and forms generation. They are also required to read all relevant SOPs and EPA methods. They also participate in an on-line GC/MS tutorial.

To measure the proficiency of each chemist, several checks have been established. The first is the ability to calibrate for each method. The chemist will run a series of at least five calibration standards and perform the calibration routine. The curve will then be reviewed by a departmental data validator. They will confirm that relative retention times (RRT) and response factors (RF) match throughout the calibration and idlist. Secondly, each analyst must perform a quad study. This will consist of serial dilutions on a known concentration mixture and analyzing four back-to-back replicates of these dilutions. This process will measure accuracy in dilution preparation as well as reproducibility of results. All data and forms generated by each chemist must pass through a thorough technical review by a departmental data validator. Any errors will be corrected by the chemist and these corrections will again be reviewed by the data validator.

Basic Principles:

The sample is extracted by use of either a separatory funnel or a liquid-liquid extractor as per EPA Method #3520B or Method #3510B (see Analysis #0813). The extract is concentrated to a final volume of 1.0 mL and analyzed by GC/MS. A 1-µL mixture of organic compounds in methylene chloride is injected onto a 0.25-mm (internal diameter) fused silica capillary column, which is enclosed in a temperature-controlled

oven. A carrier gas, helium, passes continuously through the column. The GC oven is temperature programmed and the organic mixture separates into its individual components as it moves along the length of the column. This separation is a function of the polarity and boiling point of the individual compounds. The column empties into a mass selective detector. When a compound reaches the detector, it is bombarded by high-energy electrons (70eV). This causes the compounds to fragment, forming ions. By applying various voltages to plates in the area where the ions are formed, the positive ions are thrust into a quadrupole mass analyzer which selects for a given mass fragment at a given time. These selected fragments reach an electron multiplier which amplifies the signals and sends them to a computer making storage and manipulation of the data possible. Target compounds are identified on the basis of relative retention times and spectral match to standards which are injected every 24 hours on the same system.

Quantitation is achieved by the internal standard method and the average response factor of a five-point calibration.

Apparatus:

1. Hewlett-Packard Model 5890 Gas Chromatograph or equivalent
2. Hewlett-Packard Model 5970 or 5971 Mass Selective Detector or equivalent
3. Hewlett-Packard HP-1000 or RTE-A Data System or equivalent

Reagents:

1. Decafluorotriphenylphosphine (DTFPP)
2. Methylene chloride, pesticide grade

Safety Precautions:

The toxicity of each reagent used has not been precisely determined. However, each reagent should be treated as a potential health hazard. Safety measures would include the use of fume hoods, safety glasses, lab coats, and gloves when working directly with reagents. Refer to the *Lancaster Laboratories' Chemical Hygiene Plan* for specific details.

Procedure:

A. Standard preparation

These solutions are used to standardize the GC/MS system every 24 hours and are prepared weekly or more frequently if needed. See SOP-EX-001, "Semivolatile Spiking and Calibration Standards," for standard preparation and validation. Calibration standard solutions shall be used for 1 week or until component degradation is observed.

B. Daily maintenance

Refer to MC-EX-001, "GC/MS Daily Routine Maintenance," for this procedure.

C. Tuning

1. Enter the appropriate tune file MT0x using program MTUNE and the appropriate soft keys.

2. Enter profile and spectrum scan.

3. Use the ramp parameters (ion focus, entrance lens) to adjust the abundances to roughly match the following (these numbers may vary depending on specific instrument performance):

Mass	Abundance
69	100%
131	slightly greater than 219
219	30%
502	0.8%

4. Adjust the mass axis and widths if necessary. Adjust until an accurate and repeatable axis and width are obtained.

5. Save the tune as MT0x, where x is defined as the instrument number.

6. Exit the program.

7. Using the BEDIT program, create an autosampler run with DFTPP as the first sample or, using the BAMON program, create a single-sample DFTPP run.

8. Set up the run, injecting DFTPP.

9. After the run is complete, check the DFTPP.

   a. TR,TUNCHK,name of data file

   b. If the DFTPP meets specifications (see Table II), the spectrum and mass listing will print. Standardization may begin at this point. If the DFTPP fails to meet specifications, the tune must be modified and the DFTPP reinjected until acceptable criteria are obtained.

D. Standardization

Standardization is performed by analyzing five levels of standards (5, 50, 80, 120, and 160 ppm) and creating an average response factor which is then used in quantitation. The relative standard deviation (%RSD) for each compound should

be less than 35%. If the %RSD is >35%, a first degree or second degree fit must be used for quantitation. All compounds should meet %RSD criteria ≤75%. **If these criteria are exceeded, consult with supervisor immediately.**

1. Standardization is accomplished as follows:

   a. Using the BEDIT program, create a BLIST with the standards described above.

   b. Set up the run, injecting the standards.

2. Review the quantitation reports. Use the SEMCHK program to determine if any compounds were missed in quantitation. Use the QAREA program to integrate any missing compounds or to check the integration of problem compounds, then use the GRHCPY program to print a graphics copy of the manual integration. The analyst should determine why compounds were missed in quantitation and must either adjust quant parameters or QAREA for missed compounds in all samples.

3. Run CBUPD,calibration file,C to clear the old calibration curve.

4. Run CBRPT,calibration file,TRD to generate a hard copy of the results.

5. If all %RSDs are <35%, run CBCAL,calibration file,ID list,,A to update the response factors in the library with the average response factors from the five-point calibration. For any compound which has a %RSD >35%, run CBCAL, calibration file, ID list, # (where # is defined as the number of the errant compound) and select either a first or second degree curve fit. Which curve fit to select is determined by plotting the curves using CBPLT, calibration file, # (where # is the number of the compound with %RSD >35%).

6. Update the retention times in the library by running: QCAL,output file of the 80 ppm standard,ID list,T.

7. Archive all output, calibration, and ID files. At this point, the calibration is complete and analysis of samples may begin and continue for 24 hours after the injection of the DFTPP. The average response factor of the five-point calibration is then used in sample quantitation.

E. Continuing calibrations

After 24 hours have elapsed from the injection of DFTPP, a tune check and calibration check may be performed.

1. DFTPP must be reanalyzed and meet the criteria described in Table II.

2. A calibration check standard of 80 ppm is analyzed. All compounds must have a percent difference of <20%. If this criteria is not met, a new five-point calibration must be generated.

F. Analysis of samples

1. Internal standard must be added to all samples.

   a. Sonicate an ampule of internal standard mix for 15 minutes.

   b. Add internal standard mix to each sample for an in-extract concentration of 40 µg/mL. If the sample is subsequently diluted, additional internal standard must be added to maintain the concentration of 40 µg/mL.

2. Using the BEDIT program, create a BLIST containing the samples to be analyzed.

3. Set up the run, injecting the samples.

G. Qualitative analysis

A compound is identified by comparison of the following parameters with those of a standard of this suspected compound (standard reference spectra). In order to verify identification, the following criteria must be met:

1. The sample component relative retention time must compare within ±0.06 RRT units of the RRT of the standard component.

2. All ions in the reference spectra at >10% of the most abundant ion must be in the sample mass spectra.

3. The relative intensities of the ions mentioned in G.2. must agree within ±20% between the sample spectra and the reference spectra.

4. The above criteria apply to hits greater than or equal to the LOQ. For hits between the method detection limit (MDL) and the LOQ, the above criteria and analyst's discretion are used to determine compound identification.

H. Quantitative analysis

Once a compound has been identified, quantitation will be based on the internal standard technique and the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion.

$$Concentration\ (\mu g / L) = \frac{A(x) \times I(s) \times V(t) \times D_f}{A(is) \times RRF \times V(o) \times V(i)}$$

Where:

A(x) = Area of characteristic ion for compound being measured

I(s) = Amount of internal standard injected (ng)

V(t) = Volume of concentrated extract, in microliters (µL)

D_f = Dilution factor

A(is) = Area of characteristic ion for the internal standard

RRF = Relative response factor for the compound being measured

V(i) = Volume of extract injected (µL)

V(o) = Volume of water extracted (mL)

Calculations:

1. Calculation of the relative response factor (RRF):

$$RRF = [A(x) \times C(is)] / [A(is) \times C(x)]$$

Where:

A(x) = Area of the characteristic ion for the compound being measured

A(is) = Area of the characteristic ion for the specific internal standard

C(x) = Concentration of the standard compound being measured

C(is) = Concentration of specific internal standard

2. Calculation of the percent difference:

$$\%\,Difference = \frac{RRF(i) - RRF(c)}{RRF(i)} \times 100$$

Where:

RRF(i) = Average response factor from the initial calibration

RRF(c) = Response factor from current check standard

Quality Assurance:

Each day samples are extracted, a method blank and a laboratory control sample (LCS) must be extracted. In addition, for each batch of samples, a matrix spike and matrix spike duplicate must be extracted. A batch is defined as the samples to be extracted over a 14-day period but not to exceed 20 field samples. Additional QC samples may be required to meet project or state certification requirements. Referencing QC will be based on project requirements.

1. The method blank will be evaluated for each extraction batch. It will be used to determine if there are any interferences from the analytical system, glassware, and reagents. The method blank should not contain any analyte of interest above the method detection limit. If this criteria is exceeded, the entire batch must be reextracted and reanalyzed, or the data must be qualified.

2. The MS/MSD will be analyzed to assess accuracy and precision. The recovery of each individual analyte must be in the range listed in Table I. If any analyte falls outside the range, an LCS must be analyzed for that analyte. If after analysis of the LCS the analyte is still outside the range, the entire batch must be reextracted and reanalyzed, or the data must be qualified. Poor precision (>30% RPD between MS and MSD) should be brought to supervisor's attention.

3. Each sample is spiked with surrogate standards. Acceptance criteria are listed in Table III. If any one surrogate recovery per fraction does not meet the criteria listed in Table III, but is >10%, the data must be qualified. If more than one surrogate recovery per fraction does not meet criteria but is >10%, or if any one surrogate recovery does not meet the criteria listed in Table III and is <10%, the sample should be reextracted and reanalyzed.

4. Each sample is spiked with six internal standards. If any one of these internal standard areas differs from the area of the internal standards in the 80 ppm standard by a factor of two (-50% to +100%), the sample should be reanalyzed. If this criteria is not met upon reinjection, the analyst may submit the original injection with a comment. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration, the batch run must be terminated and analytical system inspected for possible problems.

5. In some circumstances, an initial dilution may be necessary. Initial dilutions are justified when:

   a. More than three internal standard areas are <-50%.

   b. Either of the last two internal standard areas are <-80%.

   c. Prescreen data indicates significant matrix interference.

   The level of initial dilution performed is based on analyst's judgment.

Revision Log:

Initiated Date: 03/05/90

Ver. #	Effective Date	Change
00	04/15/96	Previous Issue
01	MAY 0 6 1997	Major changes are as follows: • Added Personnel Training and Qualifications section • Basic Principles - Updated method references Part D - Added information on alternate fits Part F - Reworded this section • Quality Assurance - Added section on batching

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Prepared by: Lynn Wallace Date: 4/30/97

Approved by: Christine M. Ratcliff Date: 4/30/97

Approved by: Susan S. Shorba Date: 5/5/97

Table I

Priority Pollutant Compound List

Name	Water LOQ (µg/L)	C Windows
phenol	10.	5.0 - 112.0
2-chlorophenol	10.	23.0 - 134.0
4-chloro-3-methylphenol	10.	22.0 - 147.0
4-nitrophenol	25.	1.0 - 132.0
pentachlorophenol	25.	14.0 - 176.0
2-nitrophenol	10.	29.0 - 182.0
2,4-dimethylphenol	10.	32.0 - 119.0
2,4-dichlorophenol	10.	39.0 - 135.0
2,4,6-trichlorophenol	10.	37.0 - 144.0
2,4-dinitrophenol	25.	1.0 - 191.0
4,6-dinitro-2-methylphenol	25.	1.0 - 181.0
1,4-dichlorobenzene	10.	20.0 - 124.0
N-nitrosodi-*n*-propylamine	10.	1.0 - 230.0
1,2,4-trichlorobenzene	10.	44.0 - 142.0
acenaphthene	10.	47.0 - 145.0
2,4-dinitrotoluene	10.	39.0 - 139.0
N-nitrosodimethylamine	10.	35.0 - 100.8
bis (2-chloroethyl) ether	10.	12.0 - 158.0
1,3-dichlorobenzene	10.	1.0 - 172.0

Priority Pollutant Compound List

Name	Water LOQ (µg/L)	C Windows
1,2-dichlorobenzene	10.	32.0 - 129.0
bis (2-chloroisopropyl) ether	10.	36.0 - 166.0
hexachloroethane	10.	40.0 - 113.0
nitrobenzene	10.	35.0 - 180.0
isophorone	10.	21.0 - 196.0
bis (2-chloroethoxy) methane	10.	33.0 - 184.0
naphthalene	10.	21.0 - 133.0
hexachlorobutadiene	10.	24.0 - 116.0
hexachlorocyclopentadiene	10.	1.0 - 100.0
2-chloronaphthalene	10.	60.0 - 118.0
acenaphthylene	10.	33.0 - 145.0
dimethylphthalate	10.	1.0 - 112.0
2,6-dinitrotoluene	10.	50.0 - 158.0
fluorene	10.	59.0 - 121.0
4-chlorophenyl-phenylether	10.	25.0 - 158.0
diethylphthalate	10.	1.0 - 114.0
1,2-diphenylhydrazine	10.	25.7 - 124.9
N-nitrosodiphenylamine	10.	37.8 - 147.0
4-bromophenyl-phenylether	10.	53.0 - 127.0
hexachlorobenzene	10.	1.0 - 152.0
phenanthrene	10.	54.0 - 120.0

Priority Pollutant Compound List

Name	Water LOQ (µg/L)	C Windows
pyrene	10.	52.0 - 115.0
anthracene	10.	27.0 - 133.0
di-*n*-butylphthalate	10.	1.0 - 118.0
fluoranthene	10.	26.0 - 137.0
benzidine	50.	1.0 - 155.0
butylbenzylphthalate	10.	1.0 - 152.0
benzo (a) anthracene	10.	33.0 - 143.0
chrysene	10.	17.0 - 168.0
3,3'-dichlorobenzidine	20.	1.0 - 262.0
bis (2-ethylhexyl) phthalate	10.	8.0 - 158.0
di-*n*-octylphthalate	10.	4.0 - 146.0
benzo (b) fluoranthene	10.	24.0 - 159.0
benzo (k) fluoranthene	10.	11.0 - 163.0
benzo (a) pyrene	10.	17.0 - 163.0
indeno (1,2,3-cd) pyrene	10.	1.0 - 171.0
dibenz (a,h) anthracene	10.	1.0 - 227.0
benzo (ghi) perylene	10.	1.0 - 219.0

Table II

DFTPP Key Ions and Ion Abundance Criteria

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% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Table III

Surrogate Spike Recovery Limits for Water Samples

Surrogate Compound	% Recovery Low/Medium Water
Nitrobenzene-d_5	35-114
2-Fluorobiphenyl	43-116
p-Terphenyl-d_{14}	33-141
Phenol-d_6	10-94
2-Fluorophenol	21-100
2,4,6-Tribomophenol	10-123

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Analysis #0821, 5713, 5714
Revision 01
Supersedes Date: 10/04/95
Effective Date: FEB 2 5 1999

Sample Preparation of Potable Water and Wastewater for Total Mercury Analysis by Cold Vapor Technique

Reference:

1. Method 7470A, *Test Methods for Evaluating Solid Waste*, USEPA SW-846, modified (5713), September 1994.

2. Method 245.1, USEPA CLP SOW No. ILM04.0, Exhibit D, CLP-M, D47-51, modified (0821).

3. Method 245.1, *Methods for Chemical Analysis of Water and Wastes*, USEPA 600/4-79-020, March 1979.

Purpose:

This digestion procedure is used to prepare potable water and wastewater samples for measurement of mercury by atomic absorption cold vapor technique.

Reference Modifications:

Manual digestions – To increase efficiency, Erlenmeyer flasks are used in place of BOD bottles. Prior to analysis, after excess potassium permanganate is reduced with sodium chloride/hydroxylamine hydrochloride solution, samples are adjusted to 100 mL in volumetric flasks. This allows aliquots to be taken as required for analysis; aliquots cannot be taken when BOD bottles are used.

Automated digestions – To increase efficiency and reduce waste, mercury digestions are performed using a Leeman Labs AP200II Automated Mercury Preparation System. All solutions/reagents are added in appropriate proportions to the methods.

Comparison of data between manual and automated digestions have yielded similar results.

Scope:

The digestion procedure is used by the Metals Department of the Environmental Sciences Division.

Basic Principles:

The samples are digested with nitric acid, sulfuric acid, potassium permanganate, and potassium persulfate to oxidize mercury compounds to mercuric ions. Mercuric ions are reduced to mercury metal using stannous chloride. Mercury measurement is performed using the mercury cold vapor technique.

Apparatus and Reagents:

For reagent preparation, shelf life, and storage conditions, see SOP-IO-007, "Preparation of Standards and Solutions."

1. 100-mL graduated cylinders or other appropriate graduated cylinders (manual digestion)
2. 25-mL graduated cylinders or appropriate graduated cylinders (Leeman Labs digestion)
3. Leeman Labs 15-mL sample test tubes or equivalent (Leeman Labs digestion)
4. Leeman Labs 50-mL standard test tubes or equivalent (Leeman Labs digestion)
5. 10-mL adjustable pipette, set at 6.0 mL (±1%) (Leeman Labs digestion)
6. Leeman Labs AP200II, automated mercury preparation system (Leeman Labs digestion)

7. 250-mL Erlenmeyer flasks or other appropriate Erlenmeyer flasks (manual digestion)

8. Nitric acid, HNO_3, 70.0% to 71.0%, Baker Instra-Analyzed reagent, 1.428 g/mL, or equivalent

9. Sulfuric acid, 95.0% to 98.0%, H_2SO_4, 36 N, Fisher, reagent A.C.S., 1.84 g/mL, or equivalent

10. Potassium permanganate, $KMnO_4$, Baker Analyzed reagent, A.C.S., or equivalent

11. Potassium persulfate, $K_2S_2O_8$, Baker Instra-Analyzed reagent, A.C.S., or equivalent

12. Sodium chloride, NaCl, Fisher, Certified A.C.S., or equivalent

13. Hydroxylamine hydrochloride, NH_2OH HCl, Fisher, Certified A.C.S., or equivalent

14. Potassium permanganate solution (5%) – Weigh 50 ± 1 g of potassium permanganate ($KMnO_4$) into a 600-mL beaker. Transfer the $KMnO_4$ into a 1000-mL volumetric flask using deionized water. Dilute to approximately 950 mL with deionized water. Using a stir plate, stir until the $KMnO_4$ is dissolved. Remove spin bar and dilute to volume with deionized water.

15. Potassium persulfate solution (5%) – Weigh 25 ± 1 g of potassium persulfate ($K_2S_2O_8$) into a small beaker. Transfer the $K_2S_2O_8$ into a 500-mL volumetric flask using deionized water. Add approximately 250 mL deionized water. Swirl to dissolve the $K_2S_2O_8$. (Gentle heat may be necessary.) Dilute to volume with deionized water.

16. Sodium chloride/hydroxylamine hydrochloride solution - Weigh 120 ± 1 g of sodium chloride (NaCl) into a 400-mL beaker. Transfer using deionized

water, to a 1000-mL volumetric flask. Weigh 120 ± 1 g of hydroxylamine hydrochloride ($NH_2OH•HCl$) into a 400-mL beaker. Transfer, using deionized water, to the 1000-mL volumetric flask containing the NaCl. Add deionized water, swirl to dissolve solids, then dilute to volume with deionized water.

17. Water bath (95° ± 1°C)

18. 100-mL volumetric flasks or other appropriate Class A volumetric flasks (manual digestion)

NOTE: As long as the correct ratios are maintained, solutions may be prepared using multiples of indicated weights and volumes.

Safety Precautions:

Refer to SOP-IO-011, "Inorganic Analysis Safety Procedures."

Personnel Training and Qualifications:

Training and proof of proficiency for this procedure includes, but is not limited to, the following:

1. Review and understanding of this procedure.
2. Trainee observing trained analyst performing the procedure.
3. Trainer observing trainee performing the procedure.
4. Review of trainee's data by trainer.
5. Acceptable performance on quad studies for this or equivalent procedure.
6. Documentation of critical steps in the training process.

Procedure:

For sample preservation, storage conditions, and holding times, see SOP-IO-001, "Preservation, Storage Conditions and Holding Times for Inorganic Samples."

A. Manual digestion

Shake sample well. Using a 100-mL graduated cylinder, transfer 100 mL of well-mixed sample (or an aliquot diluted to 100 mL) into a 250-mL Erlenmeyer flask. Add 5 mL of H_2SO_4 and mix.

Add 2.5 mL of HNO_3 and mix. Add 5 mL of 5% $KMnO_4$ solution and mix. Add additional portions of 5% $KMnO_4$ solution (in 5-mL increments) up to three additions, if necessary, until the purple color persists for at least 15 minutes. (Add the same amount of $KMnO_4$ solution to entire digestion batch.) Add 2.5 mL of 5% $K_2S_2O_8$ solution and mix.

Heat for about 2 hours in a water bath at 95° ± 1°C, or until sample solution volume is 90 mL or less. Remove from heat and cool.

Prior to analysis, add 2 mL of sodium chloride/hydroxylamine hydrochloride solution to reduce excess permanganate (color changed is from purple to colorless). Add reductant in 2-mL increments until $KMnO_4$ is completely reduced. Transfer the solution to a 100-mL volumetric flask, adjust the volume to 100 mL with deionized water, and mix. Reserve for analysis.

B. Leeman Labs AP 200II Automated Mercury Preparation System

NOTE: For detailed procedures for digestion using the Leeman Labs AP200II, please refer to MC-IO-015, "Mercury Digestion Method Using the Leeman Labs AP200II."

Shake sample well. Using the 10-mL adjustable pipette set to 6.0 mL (±1%), transfer 6.0 mL of well-mixed sample (or diluted aliquot) into a 15-mL sample test tube. Load the sample into its appropriate sample location in the sample rack (see Figure 1).

Using a 25-mL graduated cylinder, transfer 18 mL of the calibration and control standards to the 50-mL standard test tubes. Load the standards and check standards into their appropriate locations on the standard rack (see Figure 1).

NOTE: Calibration standards should be digested using the same procedure. For calibration standard preparation instructions, refer to SOP-IO-007, Sections E.1 to E.3.

If insufficient sample is submitted to allow using the method stated volume or the sample contains high solids, one may use a smaller aliquot of the sample. If lower than normal limits are requested, samples may be concentrated. Make appropriate acid, reagent, and spike volume adjustments based on sample final volume. (Concentration should be avoided, if possible, due to the increased potential of loss or contamination occurring during the extended digestion.)

Quality Assurance:

Perform a method blank, sample duplicate, sample matrix spike, sample matrix spike duplicate, and laboratory control sample with every 5713 (SW-846) digestion batch (20 samples or less).

Perform a method blank, sample duplicate, sample matrix spike, and laboratory control sample with every 0821 (CLP) digestion batch (20 samples or less).

Perform a method blank, sample duplicate, sample matrix spike, and laboratory control sample with every 5714 (EPA 600) digestion batch (10 samples or less).

Revision Log:

Initiated Date: 3/86

Ver. #	Effective Date	Change
00	10/04/95	Previous issue
01	FEB 25 1999	Major changes are as follows: • Added Personnel Training and Qualifications section • Added Purpose and Reference Modifications sections • Scope revised • Apparatus and Reagents revised due to new instrumentation • Procedure revised due to new instrumentation and for clarification

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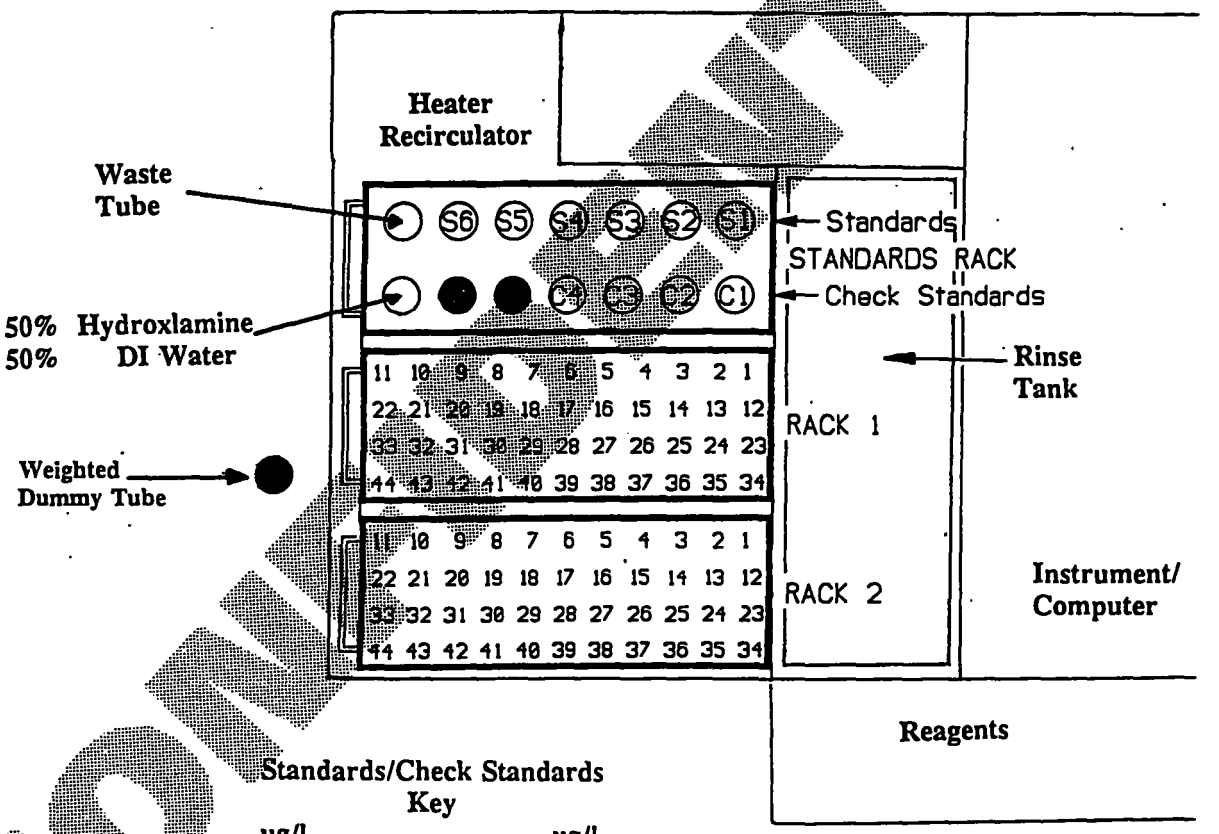
Prepared by: R. Douglas et al 11/4 Date: 2/15/99

Approved by: Robert Sturkes 8/11 Date: 2-15-99

Approved by: Dorothy M Love Date: 2/17/99

Figure 1



Standards/Check Standards Key

	ug/l			ug/l
S1	0.00	C1	CCB	(0.00)
S2	0.20	C2	ICV	(2.00)
S3	0.50	C3	CCV	(1.00)
S4	1.00	C4	CRA	(0.20)
S5	2.50			
S6	5.00			

Undigested Sample Preparation of Potable Water for Analysis of Total Recoverable Metals by Graphite Furnace Atomic Absorption and Inductively Coupled Plasma Atomic Emission Spectrometry

Reference:

1. Method 200.7, *Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry*, USEPA 600/R-94-111, Supplement I, Revision 4.4, May 1994, Office of R&D, USEPA-EMSL, Cincinnati, Ohio, May 1994.

2. Method 200.9, *Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption*, USEPA 600/R-94-111, Supplement I, Revision 2.2, May 1994, Office of R&D, USEPA-EMSL, Cincinnati, Ohio, May 1994.

3. Method 180.1 (Nephelometric), *Turbidity*, EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4/79-020.

4. *Monitek Model 21 Laboratory Nephelometer Operating Instructions Manual*, Refer to MC-IO-017, "Maintenance and Calibration of Monitek Model 21 Laboratory Nephelometer," for maintenance and calibration instructions.

Purpose:

This procedure is used for preparation of drinking water samples for "direct analysis" total recoverable determination of metals when sample turbidity is <1 NTU. The sample is made ready for analysis by mixing a measured volume with nitric acid. Samples prepared by this method can be analyzed by Method 200.7, "Determination of Metals and Trace Elements by Inductively Coupled Plasma-Atomic Emission Spectrometry," (ICP-AES) or Method 200.9, "Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption Spectrometry" (GFAA).

Reference Modifications:

Data calculation allowance is not made for addition of 1 mL HNO_3 to 100 mL of sample. 1 part in 101 is insignificant at the low metal concentrations found in potable water.

Scope:

This nondigestion procedure is used by the Metals Department of the Environmental Services Division to prepare potable water samples for analysis when silver (Ag) is not requested.

Basic Principle:

The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity. Readings are in NTUs (nephelometric turbidity units).

Sample turbidity is measured to determine if digestion is required. If turbidity is <1 NTU, the sample is batched with up to ten samples plus QC and analyzed undigested. If turbidity is ≥1 NTU, the sample requires digestion.

Apparatus and Reagents:

For reagent preparation, shelf life, and storage conditions, see SOP-IO-007, "Preparation of Standards and Solutions."

1. Monitek Model 21 Laboratory Nephelometer or equivalent
2. Sealed turbidity suspension(s)
3. Sample vial(s) – Clear, colorless glass

4. Nitric acid, HNO_3, 70.0% to 71.0%, Baker Instra-Analyzed reagent 1.428 g/mL, or equivalent

5. 100-mL graduated cylinders or other appropriate graduated cylinders

6. 100-mL volumetric flasks or other appropriate Class A volumetric flasks

7. 125-mL Nalgene bottles or other appropriate Nalgene bottles

8. Lint-free paper such as Fisher Lens Paper

NOTE: As long as the correct ratios are maintained, solutions may be prepared using multiples of indicated weights and volumes.

Safety Precautions:

Refer to SOP-IO-011, "Inorganic Analysis Safety Procedures."

Personnel Training and Qualifications:

Training and proof of proficiency for this procedure includes but is not limited to the following:

1. Review and understanding of this procedure.

2. Trainee observing trained analyst performing procedure.

3. Trainer observing trainee performing procedure.

4. Review of trainee's data by trainer.

5. Acceptable performance on quad studies for this procedure.

6. Documentation of critical steps in training process.

Procedure:

For sample preservation, storage conditions, and holding times, see SOP-IO-001, "Preservation, Storage Conditions, and Holding Times for Inorganic Analysis."

NOTE: Hold samples for 16 hours following lab preservation. Test samples with pH paper immediately prior to reading turbidity or measuring out for digestion to ensure the sample has been properly preserved (pH <2). If sample pH is verified to be >2, add more nitric acid preserving solution and hold for 16 hours until verified to be pH <2.

A. Turbidity measurement

NOTE: For accurate turbidity measurements, always keep sample vials clean. Don't handle standard or sample vial walls with fingers. Wash off all oily surfaces with good detergent (Labtone or equivalent).

1. Using the Monitek Nephelometer Model 21, turn power and lamp switches on and allow at least 30 minutes for warm-up. Set range switch to 2 (full up) position. Pour deionized water into sample vial, filling it about 80% full. Clean and dry vial by wiping with lint-free paper. Place sample vial into turbidimeter and position reference line on vial to match reference line on the meter. Zero meter by rotating zero knob until display indicates .00.

   NOTE: Deionized water passed through a 0.45-μ pore size membrane filter gives the same meter reading as deionized water that is not filtered. Therefore, unfiltered deionized water is acceptable to use as turbidity-free water for zeroing meter.

   Place the 0-1 NTU sealed reference standard (wipe clean with lint-free paper as required) into the turbidimeter with reference line on glass vial positioned to match reference line on the meter. Place light shield over vial. Recalibrate meter by rotating standardize knob until display indicates the value marked on 0-1 NTU sealed reference standard cap. Remove standard vial from meter.

Recheck zero with vial containing deionized water; adjust as required for .00 reading. Recheck 0-1 NTU sealed reference standard; making any adjustments to display value on vial cap. When both vials display proper values when placed into turbidimeter, proceed with sample measurements.

2. Empty deionized water from sample vial. Shake the sample to thoroughly disperse solids. Wait until air bubbles disappear, then pour a portion of sample into the sample vial, filling it about 80% full.

3. Clean and dry vial by wiping with lint-free paper.

4. Place sample vial into turbidimeter and position reference line on vial to match reference line on the meter. Place light shield over vial.

5. Read and record sample turbidity. If turbidity reading is <1 NTU (0.99 or lower), proceed with nondigest prep. If turbidity reading is ≥1 NTU (1.0 or higher), sample requires digestion. Notify proper personnel of sample number(s) that require digestion.

6. Rinse sample vial with deionized water between sample measurements. After reading last sample in batch, again read the 0-1 NTU sealed reference standard to verify calibration. Observed value should be within ±10% of NTU value marked on the vial cap. If outside ±10% window, recalibrate instrument and reread the samples.

7. After all turbidity measurements have been taken, turn lamp and power switches off.

NOTE: If you are unable to resolve any problems with the turbidimeter, do not attempt to use it. Place an "OUT OF ORDER-DO NOT USE" sign on it and contact your supervisor.

B. Nondigest preparation

Use the following table to prepare a batch:

5281 Nondigested Batch - Drinking Water			
Samples/QC	Initial/Final Vol (mL)	HNO_3 Vol Added (mL)	Spike Vol Added (mL)
BLANK[e]	100/100 Deionized Water	1	NONE
LCS (ICP)[a]	100/100 Deionized Water	1	2 of #1-5
LCS (GF)[b]	100/100 Deionized Water	1	1 of #8
SPIKE [R] (ICP)[c]	100/111 Sample	1	2 of #1-5
SPIKE [R] (GF)[d]	100/100 Sample	1	1 of #8
BKG [U][e]	100/100 Sample	1	NONE
DUP [D][e]	100/100 Sample	1	NONE
SAMPLES[e]	100/100 Sample	1	NONE

[a]Transfer about 75 mL of deionized water into a 100-mL volumetric flask. Add 1 mL HNO_3. Pipette 2 mL each of CLP Spikes 1-5 into the flask. Adjust to the mark with deionized water, cap, and mix well.

[b]Transfer about 75 mL of deionized water into a 100-mL volumetric flask. Add 1 mL HNO_3. Pipette 1 mL of CLP Spike 8 into the flask. Adjust to the mark with deionized water, cap, and mix well.

[c]Using a 100-mL graduated cylinder, transfer 100 mL of sample into a 125-mL Nalgene bottle. Add 1 mL HNO_3. Pipette 2 mL each of CLP Spikes 1-5 into the bottle, cap, and mix well. Document final volume as 111 mL.

[d]Using a 100-mL graduated cylinder, transfer 100 mL of sample into a 125-mL Nalgene bottle. Add 1 mL HNO_3. Pipette 1 mL of CLP Spike 8 into the bottle, cap, and mix well.

[e]Transfer approximately 100 mL of sample into a 125-mL Nalgene bottle. Add 1 mL HNO_3, cap, and mix well.

NOTE: Within a batch after mixing thoroughly, samples may be split between two Nalgene bottles for delivery to the appropriate instrument areas for analysis.

NOTE: For soluble metals analysis, filter unpreserved sample through 0.45-µ filter paper. Adjust the filtered sample to pH 2 or less with nitric acid preserving solution. Measure an appropriate volume of sample and prep as normal in an undigested batch.

Quality Assurance:

Perform a method blank, sample duplicate, sample matrix spike, and laboratory control sample with every nondigestion batch (ten samples or less).

NOTE: When samples are prepared for both inductively coupled plasma and graphite furnace on the same batch, a separate laboratory control sample and a separate spike should be prepared for ICP and GF in the batch.

Revision Log:

Initiated Date: 08/13/96

Ver. #	Effective Date	Change
00	08/13/96	Previous Issue
01	JAN 18 1999	Major changes are as follows: • Title – Revised for clarification • Reference – Revised to reflect additional references • Purpose – Added for compliance with SOP-LA-033 • Reference Modifications – Added for compliance with SOP-LA-033 • Scope – Revised for compliance with SOP-LA-033 • Basic Principle – Revised for clarification

Ver. #	Effective Date	Change
		• Apparatus and Reagents – Revised for clarification and incorporation of Amendment #1 (use of Monitek Model 21 Nephelometer) • Personnel Training and Qualifications – Added for compliance with SOP-LA-033 • Procedure – Revised for clarification and incorporation of Amendment #1 (use of Monitek Model 21 Nephelometer)

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Prepared by: Eugene K. Ahel Date: 1-14-99

Approved by: Robert Stroka Jr. Date: 1.14.99

Approved by: Dorothy M Love Date: 1/15/99

Analysis #0237, 1123, 1549, 5895, 5898, 8255, 0241, 4814
Revision 04
Supersedes Date: 08/13/98
Effective Date: MAY 13 1999
Page 1 of 27

Automated Determination of Total and Amenable Cyanide in Water, Wastewater, and Soils, Free Cyanide and Weak and Dissociable CN^- in Water and Wastewater, and Reactive Cyanide of Solids

Reference:

1. Method 9012A, *Test Methods for Evaluating Solid Waste/Physical/ Chemical*, SW-846.

2. Method 335.4, *Methods for Chemical Analysis of Water and Wastes*, EPA 600/4-79-020, March 1979.

3. Document No. 000585, *Cyanide, The Flow Solution Methodology*, Alpkem Publication, Rev. A, December 1991.

4. *Standard Methods for the Examination of Water & Wastewater*, 18th Edition, 1992.

Scope:

This method is applicable to the determination of various forms of cyanide in potable water, groundwater, and wastewater. The amenable cyanide method is applicable to any water or wastewater where the dissociable cyanide content is to be determined. All samples for total or amenable cyanide must be manually distilled (Analysis #0492, 1548, 5896, 5897, 8256, "Total and Amenable Cyanide Distillation [as Preparation for Analysis on the Alpkem Autoanalyzer"]). Analysis #0237 is cyanide in water by EPA 335.4, #8255 cyanide in water by SW-846 9012A, #1123 is reactive cyanide, #1549 is amenable cyanide in water, #5895 is cyanide in solids, and #5898 is amenable cyanide in solids. The samples for free cyanide (#0241) are analyzed without preparation on the Alpkem flow analyzer. However, samples being analyzed for weak and dissociable cyanide (#4814) must be manually prepared prior to analysis (see Analysis #7528). The limit of quantitation (LOQ) is 0.005 mg/L for water and 0.5 mg/kg for soils. The range is 0.005 to 0.350 mg/L CN^-. This range may be extended by dilution.

Modifications:

SW846 9012A specifies the pyridine-barbituric acid reagent be brought to a final volume of 250 mL with deionized water. This method utilizes pyridine-barbituric acid reagent that is brought to a final volume of 1000 mL with deionized water. This modification is performance based and meets all requirements set forth in method SW846 9012A, Section 8.0, Quality Control.

Basic Principles:

Cyanide can exist in aqueous solutions as the simple CN^- ion or it can be coordinated with various metal ions to form metallic complexes. Iron, cobalt, zinc, and copper commonly complex with CN (e.g., $Fe(CN)_6^{-3}$, $Co(CN)_6^{-3}$, $Zn(CN)_4^{-2}$, $Cu(CN)_4^{-2}$). Other species can also occupy coordination sites in conjunction with cyanide making a myriad of compounds possible. Cyanide is released from cyanide complexes by digestion and distillation. The liberated hydrogen cyanide and simple cyanides are converted to cyanogen chloride by reaction with Chloramine T. The cyanogen chloride then reacts with the pyridine-barbituric reagent to form a red-colored complex. The complex is measured at 570 nm. Samples analyzed for free cyanide show the cyanide released without preliminary distillation of the sample. Samples analyzed for weak and dissociable cyanide show the quantity of cyanide released at pH 4.5 to 6.0. (Free cyanide and weak and dissociable cyanide do not determine or quantify cyanide from tight complexes.) For the amenable cyanide determination, the sample is pretreated with chlorine to release the dissociable cyanide. Both a treated and untreated aliquot of sample are analyzed, and the difference between the two results is the cyanide amenable to chlorination.

Personnel Training and Qualifications:

Analysts are considered proficient when they have successfully completed a quad study for the analysis. A quad study consists of four laboratory control standards that are carried through all steps of the analysis and that meet the acceptance criteria for the LCS. Documentation for these studies are in each individual's training records.

Sample Handling and Preservation:

Samples may be collected in glass or plastic containers. They should be kept at an alkaline pH (12 to 12.5) to prevent the loss of hydrogen cyanide gas which can evolve at lower pHs. Oxidizing substances, such as chlorine, decomposes most cyanides. Residual chlorine in samples can be detected by using ortho-tolidine and can be eliminated by adding a few crystals of sodium thiosulfate ($Na_2S_2O_3$) or sodium sulfite (Na_2SO_3). During storage, the samples should be refrigerated at 2° to 4°C. The holding time until analysis is 14 days from sample collection for all types of cyanide except reactive cyanide, which should be analyzed as soon as possible after sample collection.

Interferences:

The method is prone to numerous interferences. These are cited below.

1. Sulfides adversely affect the colorimetric procedures. Prior to distillation, the sample is checked and treated for residual chlorine and sulfide (refer to SOP-SS-008, "Packing Bottle Orders," and Form #2264e and #2264d). If the samples are treated for sulfide, a portion of the distilled standard and blank must also be treated in the same manner. This standard and blank are then run with the corresponding treated samples.

2. Aldehydes convert cyanide to cyanohydrin which forms nitrite under the distillation conditions causing poor recoveries. Formaldehyde interference is noticeable in concentrations exceeding 0.5 mg/L; eliminate by adding silver nitrate ($AgNO_3$) to the sample. Use the spot test (Standard Methods, 18th Edition, p. 4-32) to establish the presence or absence of aldehydes.

3. Glucose and other sugars, especially at alkaline pH, lead to the formation of cyanohydrin by reaction of cyanide with aldose. Cyanohydrin can be reduced to cyanide with $AgNO_3$ (the spot test is not applicable to these sugars).

4. Carbonates can cause excessive gassing during acidification and distillation. With high carbonate samples, add hydrated lime ($Ca(OH)_2$) to raise the pH (12 to 12.5) and precipitate out the $CaCO_3$. Decant the sample from the precipitated carbonate.

5. Fatty acids and oils may interfere with the distillation and color development by forming soaps under the alkaline conditions. These fatty acids can be removed, however, by acidifying the samples to a pH 6.0 to 7.0 and extracting with hexane. Avoid multiple extractions or a long contact time at the low pH to minimize the loss of HCN. One extraction using a solvent volume 20% of the sample volume is sufficient to reduce the fatty acid below the interference level.

6. Oxidizing agents may destroy cyanides during storage and during the course of the analysis. As directed above, add sodium thiosulfate or sodium sulfite crystals to reduce these species.

7. Samples high in NO_3 or NO_2 can cause high readings in certain types of industrial waste. If this is suspected, add sulfamic acid (40 mg/10 mL sample) to the sample and shake just before analyzing.

8. Thiocyanates are a positive interference. During UV digestion, thiocyanates are decomposed to cyanide.

Apparatus:

An Alpkem automated flow analyzer (or equivalent) is required for this method. The Alpkem flow analyzer consists of the following parts:

1. Alpkem automatic sampler (Model 301) or equivalent
2. Alpkem proportioning pump (Model 502) or equivalent
3. Pump tubes and pump tube harness
4. Power distribution unit (Model 509) or equivalent
5. Cartridge heater/holder (Model 503) or equivalent
6. Monochromator and photodiode detector with 3-mm flow cell (Model 510) or equivalent
7. Alpkem cyanide super cartridge or equivalent
8. 386 PC with analog to digital (A/D) converter and Alpkem SoftPac™ software or equivalent
9. Lancaster Laboratories Wang System with Inorganics Automations group (IAG) or equivalent

Reagents:

All chemicals used must be ACS reagent grade unless otherwise noted.

Different volumes or weights may be used in the preparation of reagents or standards as long as the ratios remain equivalent.

See SOP-QA-110, "Reagents," for the appropriate labeling and documentation of reagent and standard preparation.

1. Dilution water and receptacle wash (0.25 N NaOH)

Sodium hydroxide (NaOH)	20.0 ± .2 g
Deionized water	2000 mL

Carefully dissolve 20 ± .2 g of sodium hydroxide into approximately 1800 mL deionized water in a 2-L flask. Dilute to volume with deionized water. Make fresh daily.

2. Phosphate buffer

Sodium phosphate, monobasic ($NaH_2PO_4 \cdot H_2O$)	138 ± 1 g
Brij-35	0.5 mL
Deionized water	1000 mL

Dissolve 138 ± 1 g of $NaH_2PO_4 \cdot H_2O$ in distilled water and dilute to 1 L. Add 0.5 mL of Brij-35. Store at 2° to 4°C. Prepare monthly. Indicate the date of preparation and discard date on bottle label.

3. Chloramine T reagent

Chloramine T ($C_7H_7ClNO_2SNa•3H_20$)	1.0 ± .05 g
Deionized water	250 mL

Dissolve 1.0 ± .05 g of chloramine T in 200 mL of deionized water and dilute to 250 mL. Make fresh each day.

4. Pyridine barbituric acid reagent

Barbituric acid ($C_4H_4N_2O_3$)	15 ± .2 g
Pyridine (C_5H_5N)	75 mL
Hydrochloric acid (HCl)	15 mL
Deionized water	1000 mL

Place 15 ± .2 g barbituric acid in a 1000-mL volumetric flask and add about 100 mL deionized water to wash the sides of the flask and wet down the barbituric acid. Add 75 mL of pyridine and mix. Add 15 mL of concentrated hydrochloric acid and mix. Dilute to about 800 mL with deionized water and mix until all the barbituric acid has dissolved. Dilute to volume with distilled water. This solution should be made in a hood because of the pyridine and should be discarded after 2 weeks. Store in an amber glass bottle at 2° to 4°C. Indicate the date of preparation on the bottle label.

5. Sodium hydroxide 1 N

Sodium Hydroxide (NaOH)	40 ± .4 g
Deionized water	1000 mL

Dissolve 40 ± .4 g of NaOH in deionized water and dilute to 1 L. Store at room temperature. Prepare every 6 months. Indicate the date of preparation on the bottle label.

6. Sodium hydroxide 5 N

Sodium Hydroxide (NaOH)	200 ± 2 g
Deionized water	1000 mL

Dissolve 200 ± 2 g of NaOH in deionized water and dilute to 1 L. Store at room temperature. Prepare every 6 months. Indicate the date of preparation on the bottle label.

7. Stock standard for calibration (1000 mg/L CN^-)

Potassium cyanide (KCN)	1.255 ± 0.005 g
Potassium hydroxide (KOH)	1 ± .05 g
Deionized water	500 mL

Dissolve 1.255 ± 0.005 g of potassium cyanide in 400 mL deionized water containing 1 ± .05 g of potassium hydroxide. Dilute to 500 mL with deionized water (1.00 mg CN^-/mL). Make this monthly and keep refrigerated at 2° to 4°C in an amber glass bottle. Indicate the date of preparation on the bottle label. Standardize weekly.

8. Stock standard for quality control (1000 mg/L CN^-) -

Same procedure as for the stock solution for calibration standards using potassium cyanide from a different source. Standardize weekly.

9. Standard silver nitrate solution, 0.0192 N

   Purchased - This reagent is standardized by the manufacturer against potassium chloride. A certificate of analysis is supplied with each bottle that confirms the standardized value to be 0.0192 ± 0.0001 N. Store at room temperature. See label for expiration date.

10. Rhodanine indicator

p-methyl-aminobenzalrhodanine	0.020 ± 0.001 g
Acetone	100 mL

   Dissolve 0.020 ± 0.001 g *p*-methyl-aminobenzalrhodanine in 100 mL of acetone. Prepare every 6 months. Indicate the date of preparation on the bottle label. Store at room temperature.

Standardization of Cyanide Stock Solutions: Weekly

1. Prepare three blanks by diluting 12.5 mL 5 N sodium hydroxide solution to 250 mL with deionized water and pouring each 250-mL aliquot into a separate 500-mL Erlenmeyer flask.

2. Add 20 to 25 drops of rhodanine indicator to each flask.

3. Titrate each solution with standard silver nitrate to the first change in color from yellow to brownish-pink.

4. Average the titration volumes used.

5. Measure 4 mL of the stock solution into three 500-mL Erlenmeyer flasks containing 12.5 mL 5 N sodium hydroxide and 237.5 mL deionized water.

6. Titrate each solution with standard silver nitrate to the first change in color from yellow to brownish-pink.

7. Average the titration volumes used.

8. Calculate the true value of the cyanide stock standard.

$$\frac{(A - B) \times 1000\ mg/L}{mL\ standard\ used} = CN^-\ mg/L$$

Where:

A = Volume of $AgNO_3$ from titration of standard

B = Volume of $AgNO_3$ from titration of blank

Calibration Standards:

1. Working Standard A, 10 mg/L - Pipette a volume of the calibration standard derived from the standardization procedure to equal 10 mg/L of cyanide (see calculation below) into a 100 mL volumetric flask containing 90 mL of deionized water and 5 mL of 5 N NaOH. Dilute to 100 mL with deionized water. Prepare daily.

   NOTE: Calculate the volume of stock standard required to achieve a final concentration of 10 mg/L using this formula:

$$Volume\ required\ (mL) = \frac{1000}{standardized\ value\ of\ 1000\ mg/L\ stock}$$

2. Using the Working Standard A, prepare the following calibration standards in 100-mL volumetric flasks containing 5 mL of 5 N NaOH. Dilute to volume with deionized water. Prepare daily.

Standard Name	mL Working Standard A	mg/L CN^-
S6	3.5	0.350
S5	2.0	0.200
S4	1.0	0.100
S3	0.5	0.050
S2	0.2	0.020
S1	0.05	0.005

Quality Control Standards:

1. Working Standard B, 10 mg/L - Pipette a volume of the calibration standard derived from the standardization procedure to equal 10 mg/L of cyanide (see calculation below) into a 100-mL volumetric flask containing 90 mL of deionized water and 5 mL of 5 N NaOH. Dilute to 100 mL with deionized water. Prepare daily.

   NOTE: Calculate the volume of stock standard required to achieve a final concentration of 10 mg/L using this formula:

$$\textit{Volume required (mL)} = \frac{1000}{\textit{standardized value of } 1000 \textit{ mg/L stock}}$$

2. Using the Working Standard B, prepare the following quality control standards in a 100-mL volumetric flask containing 5 mL of 5 N NaOH. Dilute to volume with deionized water. Prepare daily.

Standard	Working Standard B (mL)	mg/L CN^-
ICV, CCV	1.5	.150
LCS, for nondistilled samples	1.5	.150

Safety:

Normal laboratory safety practices should be followed. Special care should be taken in working around the heating bath and distillation head because of the danger of being burned by escaping steam or hot surfaces. Because of the toxicity of CN, special care should be taken in handling standards and samples. Breathing pyridine vapors also presents a risk. Be sure to prepare the pyridine reagent in a hood. Inspect the glassware before use. Discard or send for repair any glassware that is chipped, flawed, or broken.

Procedure:

1. Configure the Alpkem flow analyzer with the cyanide super cartridge according to Figure 1.

2. Set up the pump, the monochromator detector, the sampler, and the computer, using Table I as a guide.

   NOTE: The range may be adjusted to keep the highest standard on range if there is a change in the system.

3. Turn on the power to all units.

4. Wash the system with deionized water for at least 5 minutes.

5. Place the reagent lines in the appropriate containers and pump the system for at least 10 minutes to allow the baseline to stabilize. Autozero the detector after the baseline has stabilized.

6. Monitor the analog signal from the detector using the SoftPac™ Plus software to ensure that the baseline has stabilized.

7. Load the CN sample template which is shown in Table II. This template lists the order in which the calibration standards, blanks, and quality control standards must be run to calibrate the system and to verify that analytical system is in control. Sample names can be typed in after Position 13 and analyzed, if calibration and QC criteria have been met.

   When the CN sample template is loaded, the software will upload the channel setup field and standard table. Use the values in Table II to verify the channel setup field and standard table are correct. Save the CN template using the following numbering system:

   e.g., 93 290 CN
   ↓ ↓
   year Julian day

8. Load the sample tray according to the cyanide template list (see step 8); turn on the group number to which the output from the detector is configured using the Alpkem software.

9. Press the start button on the Alpkem sampler and begin acquiring data. The data system recognizes the "Sync" peak and activates data collection. A standard greater than half the calibration maximum is normally placed in the Sync position.

10. Following the initial calibration blank (W) in Position 13, load samples with the appropriate laboratory control standard and preparation blank. A continuing calibration verification standard (CCV) of 0.150 mg/L CN^- and continuing calibration blank (W) must be run after every **ten** injections.

11. After the calibration standards have been analyzed, check the linearity of the calibration curve. Verify that the curve-finding parameters match those in Table III. The correlation coefficient must be >0.995 for the curve to be valid and for any sample data to be reported.

12. If any sample peak is greater than full scale, dilute the sample with dilution water and analyze the dilution to obtain a peak which is within the calibration range. The dilution should be made so that the peak will fall above 10% of full scale.

13. Shutdown procedure - End the run with a calibration verification standard and calibration blank. Shut off the autosampler. Place all reagent lines and sample line in deionized water and pump for approximately 20 minutes. Remove all sample lines from the water. Turn off the system.

Calculation:

The data system automatically prepares a standard curve by plotting peak heights of standards against their concentration values and computes the concentrations of the samples (the raw result) by comparing sample peak heights with the standard curve. The blank is not used as a point on the calibration curve.

If the sample was not distilled, apply any dilution factors that may have been used against the raw result to determine the final result (calculated automatically by SoftPac™ and the IAG)

$$\textit{Final result} = \textit{raw result} \times \textit{dilution factor}$$

If the sample was distilled for total cyanide, the calculation as performed by SoftPac™ and the IAG is as follows:

Waters

$$\text{Final result} = \frac{\text{raw result} \times \text{dil. factor} \times 50\ mL}{mL\ of\ sample\ distilled}$$

Soils

$$\text{Final result} = \frac{\text{raw result} \times \text{dil. factor} \times 0.05\ L}{g\ of\ sample\ distilled} \times 1000$$

Impingers or filter extracts

$$\text{Final result} = \text{raw result} \times \text{dil. factor} \times \text{vol. of extract in L}$$

Cyanide amenable to chlorination (manual calculation)

$$\text{Final result} = \text{untreated result} - \text{treated result}$$

Reactive cyanide (manual calculations)

Samples and blank

$$\text{Final result (mg / kg)} = \frac{\text{raw result} \times DF \times .5}{wt\ (g)} \times 1000$$

$$\text{Relative percent deviation} = \frac{|\text{raw result} - \text{duplicate raw result}|}{|\text{raw result} + \text{duplicate raw result}|} \times 100\%$$

Reactivity standards (LCS)

$$\text{Final result (\% Rec)} = \left[\text{Reac std result (mg / L)} \times DF \times .5\right] + \left[\text{Reac flask (mg / L)} \times DF \times .27\right] \times 10^{\wedge}$$

Quality Control:

The following steps will be taken as part of the quality assurance program for this method:

1. A calibration curve of six standards ranging from .005 to .350 mg/L and a blank will be run at the beginning of every run. The correlation coefficient of the curve must be >0.995. If this is not met, the curve is invalid and must be rerun. The blank is not used in the correlation coefficient calculation. The blank intercept of the W cannot be greater than the LOQ or less than the negative LOQ.

2. An initial calibration verification standard (ICV) shall be run immediately after every calibration. The acceptable range is ±10% of the true value (0.150 mg/L). If the ICV does not meet this acceptance criterion, see SOP-IC-004, "Quality Control for Analyses Performed on Alpkem Flow Analyzer" for handling outliers and the corrective action which must be taken.

3. An initial calibration blank (W) shall be run after the ICV. The acceptable result is <LOQ. If the calibration blank does not meet this requirement, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

4. The method detection limit (MDL) should be determined every 6 months for Analysis #0237 by following the procedure outlined in SOP-LA-034.01, "Determining Method Detection Limits and Limits of Quantitation." For all other cyanide methods, the MDL is determined annually.

5. The linearity of the calibration must be determined every 6 months by analyzing a blank and three standards. If any verification data exceeds the initial values by ±10%, linearity must be reestablished. The values of the three standards are taken from the calculated values of the standards used for the calibration curve.

Total Cyanide

6. A batch shall contain no more than 20 samples.

7. A continuing calibration verification standard (CCV) and a continuing calibration blank (W) must be run every ten injections. The acceptable range for the CCV is ±10% of the true value (0.150 mg/L). An acceptable calibration blank result is <LOQ. If this criterion is not met, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

8. A laboratory control standard (LCS) shall be prepared and analyzed with every batch or each day samples are prepared. The acceptable range is ±10% of the true value. If the LCS does not meet this acceptance criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken. For samples of the solid matrix, the recovery should be ±10% of the concentration of the spike added.

9. Based upon client requirements, when appropriate, a laboratory control standard duplicate (LCSD) should also be prepared and analyzed under the same conditions as the LCS. The acceptable range is ±10% of the true value. The RPD between the LCS and LCSD is statistical and can be found in the WANG. If the LCSD does not meet this acceptance criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken. If the RPD between the LCS and LCSD is outside specifications, consult your coordinator or group leader to determine if reanalysis is necessary.

10. A batch blank (PB) shall be prepared and analyzed every batch or each day samples are prepared or analyzed. An acceptable result is <LOQ. If the batch blank does not meet this criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

11. A duplicate shall be prepared and analyzed for every ten samples. See Wang for current quality control acceptance criteria. If the acceptance criterion is not met, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

12. One spike shall be prepared and analyzed for every ten samples. An acceptable result is 90% to 110% recovery for samples referencing EPA 335.4 (Analysis #0237). For all other cyanide methods, see Wang for current quality control acceptance limits. If a spike recovery does not meet the acceptance criterion and the sample concentration exceeds the spike concentration by a factor of four or more, no further action is necessary.

    However, if this criterion is not met, a post-distillation spike (PDS) must be performed. If this recovery is still out of specifications, a standard additions analysis of the data for samples referencing method SW-846 9012A must be performed. No further action is required for samples referencing method EPA 335.4.

Amenable Cyanide

13. A batch shall contain no more than 20 samples.

14. An LCS shall be prepared and analyzed with every batch or each day samples are prepared. The true value is 0.20 mg/L for waters and 5 mg/kg for solids. **The expected value of these standards is 0% recovery.** See Wang for current quality control acceptance criteria. If the LCS does not meet this acceptance criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

15. Based upon client requirements, when appropriate, a laboratory control standard duplicate (LCSD) should also be prepared and analyzed under the same conditions as the LCS. If the LCSD does not meet this acceptance criterion, see SOP-IC-004 for handling outliers and corrective action which must be taken. The RPD between the LCS and LCSD should be ≤20%. If the RPD exceeds 20%, consult your coordinator or group leader to determine if reanalysis is necessary.

16. A batch blank (PB) shall be prepared and analyzed every batch or each day samples are prepared or analyzed. An acceptable result is <LOQ. If the batch blank does not meet this criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

17. A duplicate shall be prepared and analyzed for every ten samples. See Wang for current quality control acceptance criteria. If the acceptance criterion is not met, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

18. A matrix spike shall be prepared and analyzed for every ten samples. See Wang for current quality control acceptance criteria. If this criterion is not met, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

Reactive Cyanide

19. A batch shall consist of no more than 20 samples.

20. An LCS must be prepared and analyzed with every batch or each day samples are prepared. See Wang for current quality control acceptance criteria. If the LCS does not meet this acceptance criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

21. Based upon client requirements, when appropriate, a laboratory control standard duplicate (LCSD) should also be prepared and analyzed under the same conditions as the LCS. See Wang for current quality control acceptance criteria. If the LCSD does not meet this acceptance criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken. The RPD between the LCS and LCSD is statistical and can be found in the WANG. If the RPD is outside the control limit, consult your coordinator or group leader to determine if reanalysis is necessary.

22. A batch blank must be prepared and analyzed with every ten samples or each day samples are prepared. An acceptable result is <LOQ. If the batch blank does not meet this criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

23. A spike and spike duplicate must be prepared and analyzed per batch. See WANG for current quality control acceptance criteria. If the acceptance criterion is not met, see SOP-IC-004 for handling outliers and the corrective action which must be taken. The relative percent difference between the spike and spike duplicate is statistically derived and can be found in the WANG. If the RPD falls outside this criteria, see your coordinator or group leader to determine if reanalysis is necessary.

24. Based upon client requirements, when appropriate, a duplicate may also be prepared and analyzed. See WANG for current quality control acceptance criteria. If the acceptance criterion is not met, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

Free Cyanide and Weak and Dissociable Cyanide

25. A batch shall contain no more than 20 samples.

26. A laboratory control standard (LCS) shall be prepared and analyzed with every batch of samples. For samples distilled for weak and dissociable cyanide, see Wang for current quality control acceptance criteria. The acceptable range for a free cyanide LCS is ±10% of the true value. If the LCS does not meet this acceptance criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

27. Based upon client requirements, when appropriate, a laboratory control standard duplicate (LCSD) should also be prepared and analyzed under the same conditions as the LCS. The acceptable range is the same for that of the LCS. The RPD between the LCS and LCSD is statistically derived and can be found in the WANG. If the LCSD does not meet this acceptance criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken. If the RPD between the LCS and LCSD is outside the control limits, consult your coordinator or group leader to determine if reanalysis is necessary.

28. A batch blank (PB) shall be prepared and analyzed every batch or each day samples are prepared or analyzed. An acceptable result is <LOQ. If the batch blank does not meet this criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

29. A duplicate shall be prepared and analyzed for every ten samples. See Wang for current quality control acceptance criteria. If the acceptance criterion is not met, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

30. One matrix spike shall be prepared and analyzed for every batch. See Wang for current quality control acceptance criteria. If a spike recovery does not meet the acceptance criterion and the sample concentration exceeds the spike concentration by a factor of four or more, no further action is necessary. If this criterion is not met, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

Revision Log:

Initiated Date: 07/18/95

Ver. #	Effective Date	Change
00	08/25/95	Previous Issue
01	06/10/97	Major changes are as follows: • Inserted silver nitrate standardization section • Made changes to meet all state requirements across the board • Removed flask distillation head from Alpkem • Made the curve to run in descending order • New Figure 1 and new block set-up • Deleted reference to the EPA 600 method numbered 335.2

Ver. #	Effective Date	Change
02	11/21/97	Major changes are as follows: • Scope - Added new CN^- analysis number and free, weak, and dissociable analysis numbers • Method Summary - Resummarized what is occurring due to removal of distillation head on Alpkem instrument • Reagents - Deleted distillation reagent, changed phosphate buffer due to removal of distillation head • Quality Control (Total Cyanide) - Added differences in methods concerning PDS recoveries; added Free Cyanide and Weak and Dissociable Cyanide • Table II, Standards Table - Made curve to run in ascending order.
03	8/13/98	Major changes are as follows: • Interferences: deleted references to in-lab sulfide testing • Incorporated Procedural Amendment #1
04	MAY 13 1999	Major changes are as follows: • Remove standardization of $AgNO_3$ • MDL and LCR every 6 months • Delete complex standard

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Prepared by: Tonya M. Beck Date: 4/28/99

Approved by: [signature] Date: 4/28/99

Approved by: [signature] Date: 4/29/99

Table I

Cyanide Table, SFA

Range		5 - 500 µg/L
Pump		
Speed	Percent	60
Tubes	Sample	gry/gry
	Air #1	orn/wht
	Buffer	orn/orn
	Chloramine-T	orn/grn
	Pyridine-barb.	gry/gry
	Pull-off	red/red
	Sample Debubbler	orn/yel
	Sampler wash	grn/grn
Detector		
510	Wavelength	570 nm
	Rise time	10 sec.
	Range	.20 AUFS
	Heater	None
Sampler		
	Rate	51/hr.
	Sample time	35 sec.
	Wash time	45 sec.
	Pecking	OFF
	Start-up soln.	DI water/Brij
	Wash soln.	0.25 N NaOH
Computer		
	Input voltage (for 510)	0 to +1 V

NOTE: The pump tube sizes listed for the wash solution and pull-off lines are minimum requirements. Larger sized tubes may replace the minimum tube size listed. The range, wash time and sample time are merely guidelines and may be adjusted to achieve maximum instrument performance.

Table II

Table Name: CN

Cup #	Sample ID	Dil	Wt	Cup #	Sample ID	Dil	Wt
1	SYNC	1	1			1	1
2	rinse	1	1	3	B	1	1
4	S1	1	1	5	S2	1	1
6	S3	1	1	7	S4	1	1
8	S5	1	1	9	S6	1	1
10	rinse	1	1	11	B	1	1
12	ICV	1	1	13	W	1	1

Channel Setup

Channel #	=	[2]	
Channel Name	=	Cyanide	
Start Ignore Time	=	[0]	
Initial Baseline Lead Time	=	[80]	
Final Baseline Lag Time	=	[80]	
Corrections Code	Y/N	[Y]	
Cycle Time	=	[80]	
Collection Rate	=	[2]	Points/Sec.
Channel Off-Scale Warning	=	[On]	
Off-Scale Warning Limit	=	[100]	
Channel Zero Scale Warning	=	[On]	
Invert Raw Data?	Y/N	[No]	

Standards Table

Calibration Code: 1 Channel #: 2
Units: mg/L Calibration Mode: CF

S1	0.005	S11	0
S2	0.02	S12	0
S3	0.05	S13	0
S4	0.10	S14	0
S5	0.20	S15	0
S6	0.35	S16	0
S7	0	S17	0
S8	0	S18	0
S9	0	S19	0
S10	0	S20	0

Table III

Curve-Finding Parameters

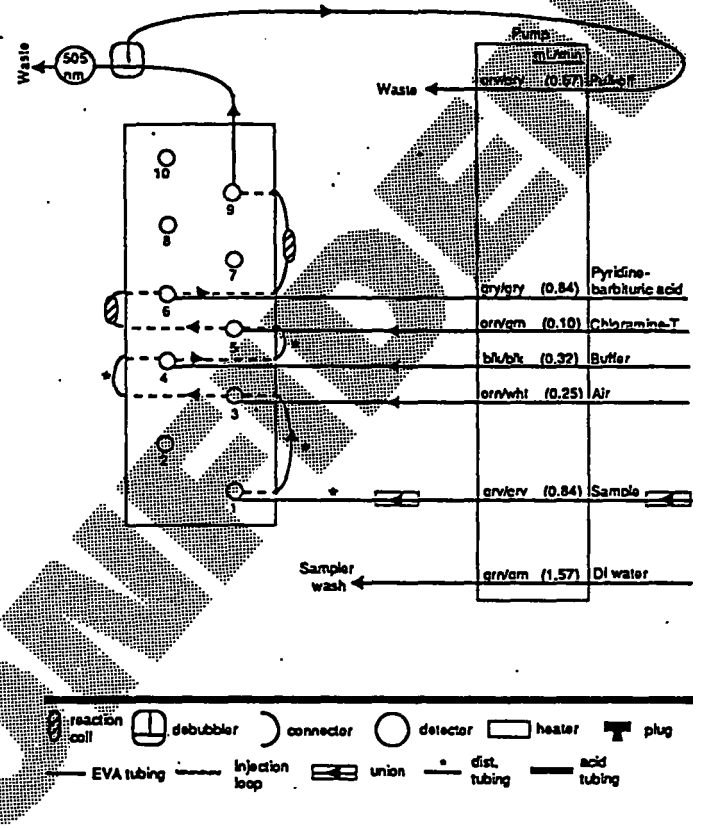
Plot curve Y/N	Y
Auto/Interactive	INTER
Decimal places 0-7	5
First sample #:	1
Peak Height/Area	HEIGHT
Threshold 1-300	10
Ascending Slope 0-100	1
Apex 1-100	10
Descending Slope 0-100	1
Plateau Points 0-100	3
Integration Points 1-100	7
Execute Y/N	Y

Figure 1

NOTE: This schematic is intended for use as a flow diagram only. Refer to the method for specific requirements.

FLOW SOLUTION FLOW DIAGRAM

Method:	*Cyanide*
Technique:	*SFA Without Distillation*
Range:	*5 - 500 µg/L CN*
Application:	*Water/Wastewater*
Date:	*May 1992*
Pump Speed:	*50*



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Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601

Procedural Amendment #1

Number: Analysis #0237, 1123, 1549, 5895, 5898, 8255, 0241, 4814

Title: Automated Determination of Total and Amenable Cyanide in Water, Wastewater, and Soils, Free Cyanide and Weak and Dissociable CN^- in Water and Wastewater, and Reactive Cyanide of Solids

Effective Date (listed on procedure): 05/13/99

Section(s) affected by change: Reference

Reason for addition(s) or change(s): To satisfy audit response for Utah state

Change will be effective from (date): JUN 2 1 1999

Samples or project affected: Utah state

List change(s) or addition(s) (specify which section):

Reference: (*add*)

NOTE: Differences between SW-846 9012 and 9012A: 9012 stipulates holding time ASAP, 9012A uses 14 days; the acceptance criteria for the LCS for 9012 is ±10%, 9012A does not set an acceptance; Method 9012A specifies the calibration curve be analyzed in ascending order, Method 9012 specifies descending order.

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Prepared by: Tonya M. Beck Date: 5/27/99

Approved by: Date: 5/27/99

Approved by: Date: 6/7/99

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Total Suspended Solids

References:

1. Method 160.2, *EPA Methods for Chemical Analysis of Water and Wastes*, EPA 600/4-79-020.

2. Method 2540D, *Standard Methods for the Examination of Water and Wastewater*, 18th Edition, 1995, p. 2-56 (background).

Scope:

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

The limit of quantitation (LOQ) for this technique varies with the sample volume. The laboratory mainframe computer may round the LOQs during the calculation.

The practical range of determination is 4 to 20,000 mg/L. Samples high in dissolved solids may yield positive interferences.

Basic Principles:

A well-mixed sample is filtered through a glass fiber filter and the residue on the filter is dried to a constant weight at 103° to 105°C. The filtrate may be used for Total Dissolved Solids. The holding time is 7 days.

Apparatus and Reagents:

1. Glass fiber filters
2. Filtration apparatus
   a. 500-mL suction flask, or equivalent
   b. Filter holder
   c. Gooch crucible
3. TSS Working Standard (150 mg/L): Dissolve .1500 ± .005 g Naphthalimide (dried approximately 1 hour at 105° ± 2°C desiccated approximately 1 hour) into deionized water. Dilute to 1000 mL. Stable 1 month at 4° ± 2°C. Naphthalimide can be weighed out and stored prior to use.

Safety Precautions:

There are no special safety precautions for this procedure. Follow routine laboratory safety steps.

Personnel Training and Qualifications:

Analysts are considered proficient when they have successfully completed a quad study for the analysis. A quad study consists of four laboratory control standards that are carried through all steps of the analysis and that meet the acceptance criteria for the LCS and the LCSD. Documentation for these studies is in each individual's training records.

Procedures:

1. Downloading a batch of samples to be performed using the LLENS system (consult SOP-WQ-014, "Instructions for Collecting Data on the LLENS System").

   a. Downloading a Sample List for Analysis #0206.

      (1) When downloading samples for Analysis #0206, all incomplete samples for analyses #0206, #0207, and #0208 will appear. Choose the appropriate samples using the INSERT key, and press PF10 to save.

      (2) A table will appear labeled Sample Table Editing.

      (3) There is no need to enter a batch standard or blank. The LLENS system adds these automatically.

      (4) If the batch chosen contains data package samples with client-submitted QC, use client QC for batch duplicate. If the batch chosen does not contain client-submitted QC, choose a sample for a duplicate. Duplicates are selected by pressing PF5 when the cursor is on the background sample.

      (5) Press PF10 to save.

   b. Hand entering a sample table for Analysis #0206.

      Using the PC WTRLEN, hand enter a sample table for incomplete #0206 samples. Select the duplicate by pressing PF5.

2. Printing a worksheet

   a. Move to the PC WTRSLD. From the Main Menu execute Examine Data.

   b. Select the batch which has the incomplete samples entered (using the INSERT key and pressing ENTER).

   c. Press PF2 to print.

   d. To choose data to print, select Tare Weights and Sample Volumes; press INSERT key.

   e. Execute PF10 to print worksheet.

   f. Press PF10 to return to Main Menu.

3. Taring crucibles

   a. Place a glass fiber filter on the bottom of a gooch crucible (wrinkled side up). Apply vacuum and wash with three 20-mL portions of deionized water. Continue suction to remove all traces of water.

   b. Dry crucible and filter in an oven for 1 hour at 103° to 105°C. If fixed or volatile suspended solids are to be performed, ignite the crucible in a muffle furnace at 550° ± 50°C for 20 minutes.

   c. Place crucible in desiccator, wait 1 hour before weighing. Check balance calibration—record the balance number in the tare weight book with crucibles and tare weight.

d. Repeat drying cycle until a constant weight is obtained, i.e., <0.5 mg drop in weight, using the lowest weight as the tare weight. Crucibles may be stored until needed.

4. Sample analysis

a. Shake sample. Remove nonrepresentative particles. Use a blender, if necessary.

b. Using a calibrated pipette or graduated cylinder, measure sample volume. Record sample volume and crucible ID on worksheet.

c. Apply vacuum and filter a small amount of deionized water. This is to seat the filter securely in the crucible. Filter the aliquot, then using three 10-mL portions of deionized water, wash residue from graduate into crucible. This step is important to ensure that dissolved solids are rinsed through, and not dried in the filter. Continue vacuum until all trace of water is gone. A final rinse may be done to remove any residue from the sides of the crucible. Vacuum until all trace of water is gone.

d. Record oven temperature on worksheet. Put samples in oven. Dry at 103° to 105°C overnight (or as little as an hour, if necessary).

Samples may remain in oven over a weekend based on a validation study performed on November 18, 1992.

5. Hand entering data (sample volumes and tare weights).

a. From Main Menu, execute Hand Enter Data.

b. Select the batch which has the incomplete samples.

c. Select the Tare Weights; press ENTER.

d. Enter crucible tare weights from databook. Press PF10 to save.

e. Select the sample volumes; press ENTER.

f. Hand enter sample volumes and crucible IDs from worksheet; press PF10 to save.

g. Record oven temperature on worksheet prior to removing samples. Desiccate for 1 hour and weigh. Check balance calibration prior to weighing samples and document on worksheet. (Check desiccant to be sure indicator crystals are still fresh.)

6. Data collection

a. Record oven temperature on worksheet prior to removing samples. Check balance calibration prior to weighing samples and document on worksheet.

b. Desiccate for 1 hour. If unable to weigh promptly after 1 hour desiccation, reheat samples for at least 1 hour in the oven. Redesiccate for 1 hour.

c. From Main Menu, execute Balance Data Collection.

d. Execute batch with incomplete samples.

e. Execute Final Dry Weights - 1.

f. Weigh samples, press space bar to transfer weight from balance to LLENS system. (An asterisk will appear when the weight stabilizes.) Press PF10 to save.

g. Repeat drying cycle (using final dry weights - 2, 3, 4 as needed) until a constant weight is obtained, i.e., <0.5 mg drop in weight, using the lowest weight for the calculation.

7. Calculating and transmitting the finished batch.

a. Move to PC WTRLEN. From LLENS Main Menu, execute Calculate Results for Analysis #0206.

b. Select incomplete batch to calculate. Execute PF10 to transmit data to the Wang system.

c. Collect all printed reports.

Calculations:

$$mg\ total\ suspended\ solids\ /\ L = \frac{(A-B)\times 1000\times 1000}{Sample\ Volume\ (mL)}$$

Where

A = Weight of filter, crucible, and residue, g

B = Weight of filter and crucible, g

Statistical Information:

For statistical information, refer to *Standard Methods for the Examination of Water and Wastewater*, 19th edition, 1995, p. 2-56.

Quality Assurance:

Quality Control needed per batch of not more than 20 samples: a blank, a 150-mg/L LCS, a 150-mg/L LCSD, and two matrix duplicates. See Wang for current quality control acceptance windows.

Comments:

The temperature range of 103° to 105°C cannot be constantly maintained due to limitations of the ovens. Acceptable results are achieved within the range of 90° to 115°C.

Revision Log:

Initiated Date: 03/06/95

Ver. #	Effective Date	Change
00	10/21/96	Previous Issue
01	DEC 1 0 1997	Major changes are as follows: • Procedure #3 and #6 - Revise taring crucible and data collection sections to incorporate weighing crucibles to constant weight.

Prepared by: Robert Heisey Date: 12/5/97

Approved by: _____ Date: 12/9/97

Approved by: _____ Date: 12/9/97

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Total Recoverable Oil and Grease (Extraction, Gravimetric)

Reference:

1. *EPA Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, p. 413.1.
2. Method 5520B, *Standard Methods for the Examination of Water and Wastewater*, 18th Edition, 1992, pp. 5-31 through 5-32.
3. Method 9070A, SW-846, 3rd Edition, November 1990.

Purpose:

The purpose of this SOP is to inform the analyst of the procedure guidelines, which enables him/her to perform oil and grease accurately.

Scope:

The range is from 8.0 to 1000 mg/L of extractable material. A Soxhlet extraction method is suggested for samples containing polar, heavy petroleum fractions, or levels of oils and greases that make solubility in trichlorotrifluoroethane impossible or very difficult. Limit of quantitation is 8.0 mg/L. The holding time is 28 days. The cited method reference specifies an evaporation bath temperature of approximately 70°C. Due to the design of the evaporation bath that is used for this method, the bath temperature must be set at approximately 85°C to induce boiling of the freon.

Basic Principles:

"Oil and grease" is any material recovered as a substance soluble in trichlorotrifluoroethane. This material is extracted from water by the intimate contact with the solvent, and then measured gravimetrically.

Trichlorotrifluoroethane may dissolve organic substances other than oils and greases resulting in a positive interference. The nature of the sample itself may cause interferences. If volatiles or heavy petroleum residuals are present, an alternative method is advisable.

Apparatus and Reagents:

1. 2-L separatory funnel with a Teflon stopcock
2. Pleated filter paper, or equivalent
3. 125-mL tared distilling flask
4. Concentrated hydrochloric acid (HCl), stable indefinitely at room temperature
5. Sodium sulfate (Na_2SO_4), anhydrous crystal, stable indefinitely at room temperature
6. Trichlorotrifluoroethane (residue checked at <2 mg residue or IR checked at <0.01 absorbance units), stable indefinitely when stored at room temperature
7. Vacuum pump or equivalent
8. Glass funnels (no stem) or equivalent
9. Analytical balance
10. Solvent capture system or equivalent
11. Desiccator

12. 100% vegetable or corn oil, stable indefinitely at room temperature

13. *N*-propanol, stable indefinitely at room temperature

14. 10,000 mg/L standard - Pipette 2.500 ± .001 g vegetable or corn oil into a 250-mL volumetric flask. Dilute to volume with *n*-propanol. Store, well stoppered at 4° ± 2°C. Hold time = 1 month.

NOTE: Alternative weights and volumes may be used for all reagents as long as final concentrations remain the same.

Safety Precautions:

Trichlorotrifluoroethane is heavier than air and reduces oxygen available for breathing. It should be used only in a well ventilated area. Freon must be disposed in a designated solvent waste can.

Personnel Training and Qualifications:

Analysts are considered proficient when they have successfully completed a quad study for the analysis. A quad study consists of four laboratory control standards that are carried through all steps of the analysis and that meet the acceptance criteria for the LCS and the LCSD. Documentation for these studies is in each individual's training records.

Procedure:

1. Collect about 1000 mL of the sample and mark sample level on bottle for later determination of sample volume.

2. Acidify the sample to a pH <2 by adding 5 mL of concentrated hydrochloric acid. (This is necessary only if 500 mL or more of deionized water is added to raise the sample volume to 1 L or if the sample has not been preserved already.)

3. Transfer the sample to a 2-L separatory funnel.

4. Rinse the graduate or sample container with approximately 30 mL of trichlorotrifluoroethane and then transfer to the separatory funnel.

5. Load the separatory funnels onto the shaker with the stopcocks open. Shake at intensity 30 for 30 seconds, then close the stopcocks and shake at intensity 45 for 90 seconds. Be sure to vent the separatory funnels before removing from the shaker! This will prevent pent-up pressure, generated while shaking, from blowing out the Teflon stopper, with resultant loss of sample. Allow the layers to separate.

6. Filter the bottom layer through solvent-moistened pleated filter paper containing approximately 1 g Na_2SO_4 into a clean tared 125-mL distilling flask. If a clear solvent layer cannot be obtained, **slowly** drain emulsified solvent onto the crystals. Add small portions of Na_2SO_4 if emulsion is excessive. Na_2SO_4 is used to selectively absorb the water from the emulsion. Samples with very heavy emulsion may require that the emulsion is centrifuged to obtain a clear freon layer.

7. Repeat steps 5 and 6 two more times with approximately 30-mL additions of solvent, but first rinse sample container with each solvent portion. Combine all three extracts in tared distilling flasks and wash filter paper with an additional 10 to 20 mL of solvent. Add more sodium sulfate if necessary.

8. Place a flask on solvent capture system that has been prewarmed (set temperature at approximately 7). The water gauge for bath water flow (in ccm) should be set at approximately 15 ccm. The gauge for condenser flow (in GPH) should be set at 4 GPH per sample (32 GPH for a full setup). The samples will take about three hours to boil down to about 1 mL of Freon.

9. Insert tubing connected to a vacuum source for at least 1 minute to remove any solvent vapor from flask. As one flask is being aspirated, remove the neck of the next flask from the condenser. This will let the last few drops of Freon escape. Wipe outside of flask to remove fingerprints and moisture.

10. Cool the flask in a desiccator for 30 minutes and weigh. Redesiccate for 15 minutes and reweigh. If second weight is more than 1 mg lower than first weight, redesiccate for 15 minutes more and reweigh. Continue in this way until constant weight is achieved.

Calculations:

$$\frac{mg\ oil\ and\ grease}{liter} = \frac{(A-B)\times 1000}{mL\ sample} \times 1000 - C$$

Where A is the weight of the flask and the residue (in grams), B is the original weight of the flask (in grams), and C is the value of the blank (in mg/L).

$$Spike\ added = \frac{(A \times B)}{C}$$

Where A is the standard (in mg/L), B is the volume of standard used (in mL), and C is the volume of sample used (in mL).

Statistical Information:

For statistical information refer to *EPA Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, p. 413.1

Quality Assurance:

Every day the analysis is performed, a blank, a 50-mg/L LCS (5 mL of the 10,000-mg/L standard to 1 L deionized water), a matrix spike (3 mL of the 10,000-mg/L standard to sample), a matrix spike duplicate, and one matrix duplicate are analyzed per batch of 20 samples. When there is not enough sample to perform the matrix spike and matrix spike duplicate, an LCSD must be prepared. See Wang for current quality control acceptance windows.

The entire batch must be repeated if the blank, LCS, or LCSD is not within specification. Control charts are kept in the quality assurance office. Refer to SOP-WQ-017, "Outlier Quality Control Data," if any of the QC samples do not meet required specifications.

Revision Log:

Initiated Date: 05/19/95

Ver. #	Effective Date	Change
00	10/07/96	Previous Issue
01	08/08/97	Major changes are as follows: • QA - Removed second duplicate
02	12/03/97	Major changes are as follows: • QA - Updated frequency of blank, LCS, LCSD • Scope - Noted discrepancy of bath temperature versus referenced method temperature

Ver. #	Effective Date	Change
03	APR 13 1999	Major changes are as follows: • Procedure – In #2 – clarity about adding acid In #9 – changed 15 seconds to 1 minute • Calculation – Added a "in grams" • Quality Assurance – Included preparations for the LCS, LCSD, matrix spike, and matrix spike duplicate • Added Purpose section

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Prepared by: Laura Saunders Date: 1-26-99

Approved by: Kenneth Bell Date: 3-29-99

Approved by: [signature] Date: 4-12-99

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APPENDIX D

LANCASTER LABORATORY SOPS - OTHER

Bottle Preparation

Purpose:

The purpose of this SOP is to provide traceability of the storage, cleaning, and preservation of sample containers.

Scope:

This SOP describes the preparation of bottles that are sent or signed out through our bottles room.

Personnel Training and Qualifications:

1. Understand bottle codes, see Form #2520
2. Shown which preservatives to use and amounts, see Form #2290
3. Review PPE and handling of acids and bases
4. Ability to use dispenser pipette
5. Familiar with the use of preservative tapes
6. Shown where to put bottles when done preserving

Procedure:

1. Containers are purchased from various distributors.

2. Bottles and vials that are purchased precleaned meet EPA specifications and guidelines.

3. The sterile plastic bottles (Code 24) are purchased precleaned and prepreserved. (See Quality Assurance Manual-Potable Water.)

4. Certain bottles are preserved at the laboratory prior to being sent to the client. (See Form #2290, "Bottle Preservation," for instructions on which bottles to preserve, what preservative to use, and the quantities used to preserve each bottle.) Once the preservative is added, a label is placed over lid. This label indicates what preservative is used. The preserved bottles are then stored on a designated shelf until they are used in a bottle order.

5. Some bottles require multiple preservatives, usually because a dechlorinating agent is required. In these cases, the dechlorinating agent is placed in the sample bottle, and the preservative is placed in a separate vial and attached to the outside of the bottle. This is also true if there are multiple preservatives that are unstable together.

6. When a new lot number is opened for each preservative, the preservative is checked for contaminants. See SOP-SS-004, "Preservation & Bottle Room Preservative Traceability."

7. We try to maintain an adequate inventory of each bottle type. Inventory is done weekly to ensure we are maintaining this supply.

8. We also supply certificates for precleaned bottles when requested prior to delivery. Because we do not stock these, 2 weeks prior notice is required. If certificates are requested, the original is filed with the client paperwork and copies are sent to the client with the bottles.

Revision Log:

Initiated Date: 02/15/94

Ver. #	Effective Date	Change
00	06/28/95	Previous Issue
01	04/24/97	Major Changes: • Updated bottle codes • Added Personnel Training and Qualifications section • Added volume and weight disclaimer • Revise form to comply with NJ regulations • Change title
02	AUG 07 1997	Major Changes: • Delete "of 5 normal NaOH" from Bottle Code 02

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Prepared by: _____ Date: 8-1-97

Approved by: _____ Date: 8-1-97

Approved by: _____ Date: 8/6/97

Figure 1

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601-5994

Bottle Preparation

Bottle Code	Preservative/Quantities/Analysis
01	Sulfuric Acid H_2SO_4 (to a pH <2) 125-mL amber glass, .25 mL of 9M H_2SO_4 Grade: ACS Certified Example Analysis: TOC
02	Sodium Hydroxide NaOH/Asc Acid (to a pH >12) 500-mL narrow-mouth plastic 30 mg Asc Acid in sample container 2 mL of 10 N NaOH attached to side of container Grade: Reagent Example Analysis: Cyanide
03	Sulfuric Acid H_2SO_4 (to a pH <2) 1000-mL clear glass, 2 mL of 9M H_2SO_4 Grade: ACS Certified Example Analysis: COD, TKN, Phenols, NH_3 Nitrogen
06	Hydrochloric Acid HCl (to a pH <2) 1000-mL glass, 5 mL of 5.8N HCl Grade: ACS Certified Example Analysis: Oil and Grease, TPH
07 09	Nitric Acid HNO_3 (to a pH <2) 500-mL wide-mouth plastic, 5 mL 2.5 N HNO_3 (168 mL Conc. HNO_3 + 832 mL deionized water) 1000-mL wide-mouth plastic, 10 mL 2.5 N HNO_3 Grade: ACS Certified Example Analysis: Metals, Radioactivity
13	Sulfuric Acid and Sodium Sulfite H_2SO_4/Na_2SO_3 (to a pH <2) 250-mL amber glass with Septa cap 30 mg of anhydrous Na_2SO_3 in sample container, .25 mL of 9M H_2SO_4 attached to side of container Grade: ACS Certified Example Analysis: TOX
14	Methanol 40-mL vial (prepared by Volatiles group) Example Analysis: Wisconsin GRO, Soil/Solid Volatiles

Note: Preservative measurements are ±10% of value given

Figure 1 - Continued

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Bottle Preparation - Continued

Bottle Code	Preservative/Quantities/Analysis
18	Hydrochloric Acid and Ascorbic Acid HCl/Asc Acid (to a pH <2) 40-mL vial, .2 mL of 30 mg of crystal Ascorbic Acid + 5.8 N HCl attached to side of container Grade: ACS Certified Example Analysis: Regulated and Unregulated VOCs (Lists 1 and 3)
24	Sodium Thiosulfate $Na_2S_2O_3$ 120-mL plastic, $Na_2S_2O_3$ Powder Grade: ACS Certified Example Analysis: Total Coliform, Standard Plate Count, Fecal Coliform
25	Sodium Thiosulfate $Na_2S_2O_3$ (to a pH >5/<9) 40-mL vial, 40 mg crystal $Na_2S_2O_3$ Grade: ACS Certified Example Analysis: Trihalomethanes
26	Hydrochloric Acid HCl (to a pH <2) 40-mL vial, .2 mL of 5.8 N HCl Grade: ACS Certified Example Analysis: BTEX, VOA in groundwater
28	Sodium Hydroxide NaOH/ZnAc (to a pH >9) 500-mL glass, 2 mL of 10 N NaOH in sample container 3 mL of ZnAc attached to side of container Grade: Reagent Example Analysis: Sulfide
29	Hydrochloric Acid HCl (to a pH <2) 1000-mL amber glass, 5 mL of 5.8N HCl Grade: ACS Certified Example Analysis: TPH-DRO
30	Sodium Thiosulfate $Na_2S_2O_3$ (to a pH >5/<9) 1000-mL amber glass, 30 mg crystal $Na_2S_2O_3$ Grade: ACS Certified Example Analysis: Pest, Herb, PCBs
37	Hydrochloric Acid and Sodium Thiosulfate HCl + $Na_2S_2O_3$ (to a pH <2) 40-mL vial, 40 mg $Na_2S_2O_3$ + .2 mL 5.8 N HCl attached to side of container Grade: ACS Certified

Figure 1 - Continued

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Bottle Preparation - Continued

Bottle Code	Preservative/Quantities/Analysis
38	See Bottle Code 26. Example Analysis: GC/MS VOA
39	See Bottle Code 18. Example Analysis: VOA in Drinking Water by GC/MS
45	See Bottle Code 30. Example Analysis: Semivolatile
46	Sulfuric Acid 9M H_2SO_4 (to pH <2) 1000-mL clear glass, 1 mL H_2SO_4 Grade: ACS Certified Example Analysis: Oil and Grease, TPH
57	See Bottle Code 26.
58	Hydrochloric Acid HCl (to pH <2) 40-mL vial, 2 mL of 1:2 N HCl Grade: ACS Certified Example Analysis: VOA by 603
64	Ammonium Chloride NH_4Cl 1mL NH_4Cl solution in a 60 mL vial Grade: ACS Certified Example Analysis: Haloacetic Acids
66	Isooctane C_8H_{18} 40-mL glass vial, 5 mL C_8H_{18} Grade: Pesticide Grade Example Analysis: PCB Wipes
68	See Bottle Code 25. Example Analysis: Unregulated VOA List 2
72	Monochloroacetic Acid MCA 40-mL vial, 1.0 mL MCA buffer solution (prepared by Dept. 24) Grade: Example Analysis: Carbamates

Figure 1 - Continued

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Bottle Preparation - Continued

Bottle Code	Preservative/Quantities/Analysis
78	Sulfuric Acid and Sodium Thiosulfate $H_2SO_4/Na_2S_2O_3$ (to a pH <2) 1000-mL Plastic Amber, 75 mg $Na_2S_2O_3$ placed in container 1 mL 9M H_2SO_4 attached to side of container Grade: ACS Certified Example Analysis: Diquat/Paraquat
79	Hydrochloric Acid and Sodium Sulfite HCl/Na_2SO_3 (to a pH <2) 1000-mL Amber glass with 30 mg anhydrous Na_2SO_3 in sample container with 5 mL 5.8 N HCl attached to side of container Grade: ACS Certified Example Analysis: EPA 525 Semivolatiles
81	Sodium Hydroxide NaOH (to a pH >12) 500 mL glass, 2 mL of 10 N NaOH Grade: Reagent Example Analysis: Dissolved Sulfide
85	Ammonium Chloride/Phosphate Buffer NH_4Cl/Buffer 60 mL glass vial with 1 gm (prepared by Dept. 32) Example Analysis: Non-Chloral Hydrate
88	Sodium Sulfite/Phosphate Buffer NA_2SO_3/Buffer 60 mL glass vial with 1 gm (prepared by Dept. 32) Example Analysis: Chloral Hydrate
92	Polyethylene Glycol PEG 40-mL vial (prepared by Dept. 30) Example Analysis: EPA 25D

NOTE: Special bottles are prepared as needed.

Figure 1 - Continued

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Abbreviation Codes

C_8H_{18}	Isooctane	Hazardous
DI H_2O	Deionized Water	Nonhazardous
HCl	Hydrochloric Acid	Hazardous
H_2SO_4	Sulfuric Acid	Hazardous
HNO_3	Nitric Acid	Hazardous
MCA Buffer	Monochloroacetic Acid	Hazardous
NaOH	Sodium Hydroxide	Hazardous
Na_2SO_3	Sodium Sulfite	Nonhazardous
$Na_2S_2O_3$	Sodium Thiosulfate	Nonhazardous
ZnAc	Zinc Acetate	Nonhazardous
ASC Acid	Ascorbic Acid	Nonhazardous
NH_4Cl	Ammonium Chloride	Nonhazardous
PEG	Polyethylene Glycol	Nonhazardous
Methanol	Methanol	Hazardous

NOTE: All Material Safety Data Sheets (MSDS) can be found in the Safety Director's office. Hazardous and nonhazardous is in reference to shipping labeling requirements.

Procedural Amendment #1

Number: SOP-SS-003.02

Title: Bottle Preparation

Effective Date (listed on procedure): 08/07/97

Section(s) affected by change: Header

Reason for addition(s) or change(s): Typo

Change will be effective from (date): 08/07/97

Samples or project affected: All

List change(s) or addition(s) (specify which section):

Header does not list correct supersedes date. Correct as shown:

SOP-SS-003.02
Supersedes Date: 04/24/97
Effective Date:
Page __ of __

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Prepared by: _____ Date: 8-18-97

Approved by: _____ Date: 8-18-97

Approved by: _____ Date: 8/19/97

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QUALITY ASSURANCE OPERATIONS MANUAL
Data Storage, Security, and Archiving

Purpose:

The analytical data generated by Lancaster Laboratories is a valuable commodity purchased by our clients. One of the objectives of our Quality Assurance Program is to provide "traceability" of reported analytical results. For all testing, we must be able to determine the analyst(s) who performed the work, the date of analysis, the raw data acquired during analysis, the condition of any instrument or equipment used, and the status of the quality control system at the time the analysis was run. Therefore, all data must be stored in an orderly fashion and access to the storage area controlled to prevent loss, deterioration, or deliberate tampering.

Scope:

This procedure applies to all business units of Lancaster Laboratories and is designed to summarize the storage and controlled access of data generated by Lancaster Laboratories' personnel. Retention of additional laboratory records are summarized by the records retention schedule in SOP-OI-012, "Archives Guidelines."

Personnel Training and Qualifications:

All personnel responsible for the archiving of data must read and understand the guidelines addressed by this procedure in addition to SOP-OI-012.

Procedure:

1. All analytical data generated in the laboratory shall be transferred to one of the archivists for storage in a locked storage area which is referred to as the archives. The archivists reside with the Office Services Department.

2. The following are types of data that are transferred to the archives:

   a. Notebooks and logbooks

   b. Chromatograms

   c. Spectra

   d. Records of instrument and equipment maintenance, calibration, and qualification

   e. Standard, reagent, and media preparation data

   f. Analysis reports

   g. Additional information summarized within the records retention schedule of SOP-OI-012

3. Each department is responsible for packing their own data in approved archive boxes. All boxes must be marked on the outside with dates, department number, and description of contents. The archivist maintains an index of the content contained in the archives.

4. It is permissible to store data in the area where it was generated for a reasonable period of time for reference purposes, but its security will be the responsibility of the department generating the information.

5. No instrumental data shall remain in the laboratory area for more than 1 year after it is generated and completed. No data notebook or logbook shall remain in the laboratory for:

   a. longer than 3 months after the book has been completed; or,

   b. longer than 3 years from the date of its creation.

If all pages in a data notebook or logbook are not used upon its archival, they shall be cancelled out prior to filing.

6. Laboratory copies of the analytical final reports shall be archived within 1 month of printing. Billing and Reporting department personnel are responsible for filing and archiving of CSMS analytical final reports.

   Copies of analytical final reports can also be archived as part of a data package, with the raw data, if a technical department chooses to do so.

7. If it is necessary to retrieve data from the archives, a records request form must be filed with the archivist. An archivist or laboratory employee can retrieve data. If laboratory personnel chooses to retrieve data, they **must** be accompanied by an archivist in the storage area at all times. The archivist notes what documents were borrowed and the date they were taken. All borrowed materials must be returned to the archivist within 30 days of retrieval. It is the responsibility of the archivist to follow-up with the individual who requested the data, if the information is not returned within this stipulated time period. Follow-up and return of materials is documented by the archivist.

8. A specific retention schedule for all archived records is summarized in SOP-OI-012. The length of retention is set by the appropriate area responsible for the data in addition to regulatory/agency requirements. Direction is given by the director of each business unit in conjunction with the company president and Quality Assurance.

9. If data is stored and maintained on magnetic, optical, or other computer readable media, procedures will need to be in place, within the responsible area, to address the maintenance, storage, and retrieval of this information.

Revision Log:

Initiated Date: 05/87

Ver. #	Effective Date	Change
00	03/15/95	Previous issue
01	08/15/97	Major changes are as follows: • Expanded upon the procedure section to summarize information from SOP-IO-012 • Added a Personnel Training and Qualifications section
02	DEC 0 1 1998	Major changes are as follows: • Removed references to individual business units from Scope • Added requirement for all notebooks and logbooks to be removed from laboratories no longer than 3 years after their creation • Removed specific references to data retention times from Procedure section 8

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Prepared by: _____ 972 Date: 11/19/98

Approved by: _____ Date: 11/21/98

Approved by: _____ Date: 11/23/98

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601

Procedural Amendment #1

Number: SOP-QA-114

Title: Data Storage, Security, and Archiving

Effective Date (listed on procedure): 12/01/98

Section(s) affected by change: Procedure

Reason for addition(s) or change(s): To allow for maintenance and calibration logbooks to remain in the laboratory as required

Change will be effective from (date): JUN 0 1 1999

Samples or project affected: N/A

List change(s) or addition(s) (specify which section):

Procedure: (*add the following to Section 5.b as follows*)

5.b. The exception to this rule is for maintenance and calibration logbooks. In certain cases, it may be beneficial to maintain these books with the instruments for extended periods of time.

To minimize the potential for loss of large amounts of data, these notebooks shall be archived or copied annually. Copies must be sent to Office Services for filing, using a copy of Form #2709 as a coversheet. A notation shall be made in the M&C logbook listing the pages copied. Previously copied pages do not need to be copied multiple times.

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052599

Prepared by: Gregory James Date: 5/26/99

Approved by: Kathleen M. Lowrie Date: 5/27/99

Approved by: [signature] Date: 5/27/99

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APPENDIX E

SOPS FOR FIELD INSTRUMENTS

STANDARD OPERATING PROCEDURES
ORGANIC VAPOR AIR MONITORING AND SCREENING PROCEDURES

1.0 PURPOSE

Exposure to airborne organic contaminants can present a significant threat to worker health and safety. Identifying and quantifying these contaminants through air monitoring is essential for reconnaissance activities. Reliable measurements of airborne contaminants are necessary for selecting appropriate levels of personal protective equipment, delineating areas where protection is needed assessing the potential health effects of exposure, and determining the need for specific medical monitoring. This SOP discusses factors to be considered when conducting air monitoring.

2.0 DISCUSSION

Strategies for assessing airborne contamination are discussed in the FSP and QAPjP. The potential instruments to be used include the Photoionization Detector (HNu®), the Organic Vapor Analyzer (OVA®), and the Organic Vapor Monitor (OVM). Specific field rounds for these instruments are attached.

The following definitions apply to this SOP:

- Flame Ionization – A process in which sample gas is ionized with a flame, allowing a count of carbon atoms.
- Photoionization Detector (HNu and/or OVM) – A portable instrument used to detect, measure, and provide a direct reading of the concentrations of a variety of trace organic gases in many industrial or hazardous atmospheres. The principle methods of detection is photoionization (see definition below).
- Ionization Potential – The amount of energy needed to strip an electron from the orbit of its resident molecule, expressed in electron volts (eV). Ionization potentials for typical compounds are provided in Table 1.
- Organic Vapor – Airborne compounds with chain or ring structures composed of carbon, hydrogen, and other elements.
- Organic Vapor Analyzer (OVA®) – A portable instrument used to detect, measure, and provide a direct reading of the concentration of a variety of race organic gases in many industrial or hazardous atmospheres. The principle method of detection is flame ionization (see definition above).
- Photoionization – A process involving the absorption of ultraviolet (UV) radiation by a gaseous molecule, leading to ionization.

The equipment used to conduct direct reading air monitoring of airborne organic compounds will be limited to the HNu® photoionization detector, the Foxboro OVA, and/or the Thermo Nutech OVM. Other equipment is available to conduct similar monitoring. Calibration gas will also be required.

3.0 PROCEDURES

The following procedures apply to direct reading air monitoring equipment.

3.1 DIRECT-READING INSTRUMENT CONSTRAINTS

All direct-reading instruments have inherent constraints in their ability to detect gaseous organic compounds. They usually detect and measure only specific classes of chemicals. Generally, they are not designed to measure and detect airborne concentrations below 1 part per million (ppm). Finally, many direct reading instruments that have been designed to detect one particular substance also detect other substances, which may cause interference and may consequently give false readings.

3.2 ACCURATE RECORDING AND INTERPRETATION

Direct-reading instruments must be operated and the data must be interpreted by individuals who understand the operating principles and limitations of the instrument. At hazardous waste sites, where unknown and multiple contaminants are frequently encountered, instrument readings should be interpreted conservatively.

The following guidelines will facilitate accurate recording and interpretation:

- Calibrate instruments according to the manufacturer's instructions, before and after every use (see operating manuals enclosed).
- The instrument's readings have limited value where contaminants are unknown. When recording readings of unknown contaminants, report them as "X" instrument units or "positive response" rather than specific concentrations in measured units such as ppm.
- Conduct additional monitoring at any location where a positive response occurs.
- Report a reading of zero as non-detectable (ND) rather than as clean. Quantities of chemicals may be present, but at concentrations that are not detectable by the instrument.
- Repeat the air monitoring survey using other detection devices. Other devices used will depend on site-specific contaminants of concern.

3.3 PHOTOIONIZATION SYSTEMS

This subsection describes the application, method, limitations, operations, and maintenance of the photoionization detector (PID).

3.3.1 Application

The photoionization detector can be used to detect total concentrations of many organic and some inorganic gases and vapors. It can sometimes be used to identify compounds if more than one probe is used.

3.3.2 Detection Method

The PID works by ionizing molecules using UV radiation. The radiation strips electrons from the molecules, producing a current that is proportional to the number of ions.

3.3.3 Limitations

The PID cannot be relied on to do the following:

1. Detect methane
2. Detect a compound if the probe used has a lower energy level than the ionization potential of the compound.
3. Respond accurately when there is a mixture of gases or vapors.
4. Respond accurately in high humidity or very cold weather.
5. Respond accurately when there is interference with other current sources.

3.3.4 Ease of Operations

To effectively use the PID, the operator must understand the operating principles and procedures and be competent in calibrating, reading, and interpreting the instrument.

3.3.5 General Care and Maintenance

The PID will need to be recharged or have its battery replaced every 10 hours. The lamp window in the probe must be cleaned regularly. The instrument and its accessories must also be regularly cleaned and maintained.

3.3.6 Typical Operating Time

The PID will typically run continuously for 10 hours on a charged battery, 5 hours with a strip chart reorder.

3.4 FLAME-IONIZATION SYSTEM

This subsection describes the application, method, limitation, operation, and maintenance of the flame-ionization system.

3.4.1 Application

When set in the survey mode, the flame-ionization detector (FID) can be used to detect the total concentration of many organic gases and vapors. In the gas chromatography (GC) mode, the OVA is used to identify and measure specific compounds.

In the survey mode, all organic compounds are ionized and detected at the same time. In the GC mode, volatile species are separated.

3.4.2 Detection Method

Organic gases and vapors are ionized in a flame. A current is produced in proportion to the number of carbon atoms present.

3.4.3 Limitations

- The FID cannot be used to detect inorganic gases and vapors and some synthetics. Sensitivity depends on the compounds.
- The FID should not be used at temperatures less than 40° F (4 ° C).
- The FID has difficulty absolutely identifying compounds.
- High concentrations of contaminants or oxygen-deficient atmospheres require system modifications.
- In the survey mode, readings can be only reported relative to the calibration standard used, such as methane equivalents.
- A high grade hydrogen supply is typically needed to operate the FID.

3.4.4 Ease of Operation

- Monitoring site conditions, especially in the GC mode, requires a certain degree of experience in using the FID.
- To identify a specific contaminant, the FID must be calibrated with the contaminant.

3.4.5 General Care and Management

- The FID battery must be recharged every 8 hours or replaced, as needed.
- The hydrogen fuel supply must be monitored during use to maintain an adequate supply.
- The FID user should perform routine maintenance as described in the operation manual.
- The FID should be routinely checked for leaks.

3.4.6 Typical operating Time

- The FID can typically run for 8 hours on a fully charged battery, 3 hours with a strip chart recorder.

4.0 INTERFERENCES AND CORRECTIVE ACTIONS

Particulates can be drawn into the probe forming deposits on the surface of the UV lamp or in the ion chamber that interfere with the operation of the PID/FID. Such interference is indicated by meter readings that are low, erratic, unstable, nonrepeatable, or drifting, or show apparent moisture sensitivity. Corrective action consists of cleaning of the lamp and/or the ion chamber, as described in the instrument specific operating manuals.

4.1 SAFETY PRECAUTIONS

None.

4.2 SAMPLE SIZE, COLLECTION, PRESERVATION, AND HANDLING

1.) Ambient Air - The air will be drawn directly into the probe; therefore, no sample will actually be collected.

2.) Soil Headspace Vapors - Collect samples as specified in the QAPjP and FSP, and place in an 8-oz. Jar until half full. Place aluminum foil over the jar mouth to achieve as tight a seal as possible. Screw the jar lid in place and allow the sample to warm to ambient temperature (approximately 75°F) by setting it out in the sun or placing it in a heated room.

4.3 ROUTINE PREVENTIVE MAINTENANCE

Consists of battery charge and cleaning of the UV lamp and/or ion chamber, if necessary.

4.4 REAGENTS AND CALIBRATION STANDARDS

PIDs are typically calibrated to isobutylene and FIDs are typically calibrated to methane.

4.5 CALIBRATION PROCEDURE

Calibration is performed at the beginning and middle of each day's use in accordance with the attached operating manuals, or as necessary during use.

In service, the calibration of the analyzer can be rapidly checked daily by the use of small disposable cylinders containing the required calibration standard with a regulator.

5.0 VARIABLES AFFECTING OUTDOOR AIR MONITORING

Complex environments containing many substances, such as those associated with hazardous waste sites, pose significant challenges to the task of accurately and safely assessing airborne contaminants. Several independent and uncontrollable variables, most notably temperature and weather conditions, can affect airborne concentrations. These factors must be considered when conducting air monitoring and interpreting data. The following environmental variables must be considered: temperature, wind speed, rainfall, moisture, vapor emissions, and work activities. Each of these variables is discussed below.

5.1 TEMPERATURE

An increase in temperature increases the vapor pressure of most chemicals. Change in temperature may affect the concentration detected.

5.2 WIND SPEED

An increase in wind speed can affect vapor concentration near a free-standing liquid surface. Dust and particulate-bound contaminants are also affected.

5.3 RAINFALL

Water from rainfall can essentially cap or plug vapor emission routes from open or closed containers, saturated soil, or lagoons, thereby reducing airborne emissions of certain substances.

5.4 MOISTURE

Dusts are highly sensitive to moisture content. This moisture content can vary significantly with respect to location and time and can also affect the accuracy of many sampling results.

5.5 VAPOR EMISSIONS

The physical displacement of saturated vapors can produce short-term, relatively high vapor concentrations. Continuing evaporation or diffusion may produce long-term vapor concentrations and may involve large areas.

5.6 WORK ACTIVITIES

Work activities often require the mechanical disturbance of contaminant materials, which may change the concentration and composition of airborne contaminants. Airborne emissions from gasoline or diesel engines used at near monitoring locations may also affect the concentration of airborne organic contaminants.

6.0 ACCURACY AND PRECISION

Accuracy will be assessed by verifying the calibration on two different lots of span gas. Accuracy should be within 10% and will be recorded to the nearest 0.1. Measurements outside of the accuracy requirements will be redone. Continued problems may require corrective action.

Precision during calibration will not be assessed due to the difficulty of splitting a span gas sample. Precision during sampling may be conducted by taking replicate measurements on a split sample.

PID and FID measurements will help to determine areas for sampling but cleanup criteria will not be established based on head space measurements.

Table 1

Ionization Potentials (eV)

Atoms and Simple Molecules		
	H_2	15.426
	N_2	15.580
	O_2	12.075
	CO	14.01
	NO	9.25
	F_2	15.7
	CI_2	11.48
	Br_2	10.55
	I_2	9.28
	HF	15.77
	HCI	12.74
	HBr	11.62
	HI	10.38
	SO_2	12.34
	CO_2	13.79
	COS	11.18
	CS_2	10.08
	N_2O	12.90
	NO_2	9.78
	H_2O	12.559
	H_2S	10.46
	NH_3	10.15
Paraffins and Cycloparaffins		
	Methane	12.98
	Ethane	11.65
	Propane	11.07
	N-Butane	10.63
	N-Pentane	10.35
	N-Hexane	1-/19
	N-Heptane	10.08
	Cyclohexane	9.88
Alykl Halides		
	Methyl Chloride	11.28
	Dichloromethane	11.35
	Trichloromethane	11.42
	Tetrachloromethane	11.47
	Ethyl Chloride	10.98
	1,2-Dichloroethane	11.12

Table 1

Ionization Potentials (eV)
(continued)

Alykl Halides (con't)	
1-Chloropropane	10.82
Methyl Bromide	10.53
Dibromomethane	10.49
CH_2BrCl	10.77
$CHBr_2Cl$	10.59
Ethyl Bromide	10.29
1,1-Dibromoethane	10.19
1-Bromopropane	10.18
Methyl Iodide	9.54
$CFCl_3$ (Freon 11)	11.77
CF_2Cl_2 (Freon 12)	12.31
CF_3Cl (Freon 13)	12.91
$CHClF_2$ (Freon 22)	12.45
$CFBr_3$	10.67
CF_2Br_2	11.07
CH_3CF_2Cl (Genetron 101)	11.98
$CFCl_2CF_2Cl$	11.99
CF_3CCl_3	11.78
Aliphatic Alcohols, Ethers, Thiols, and Sulfides	
Methyl Alcohol	10.85
Ethyl Alcohol	10.48
N-Propyl Alcohol	10.20
Dimethyl Ether	10.00
Methanethiol	9.440
Ethanethiol	9.285
1-Propanethiol	9.185
Dimethyl Sulfide	8.685
Ethyl Methyl Sulfide	8.55
Diethyl Sulfide	8.430
Alphatic Aldehydes and Ketones	
Formaldehyde	10.87
Acetaldehyde	10.21
Propionaldehyde	9.98
Acrolein	10.10
Crotonaldehyde	9.73
Benzaldehyde	9.53

Table 1

Ionization Potentials (eV)
(continued)

Alphatic Aldehydes and Ketones (con't)	
Acetone	9.69
Methyl Ethyl Ketone	9.53
Cyclohexanone	9.14
Aliphatic Acids and Esters	
Formic Acid	11.05
Acetic Acid	10.37
Propionic Acid	10.24
Ethyl Acetate	10.11
Aliphatic Amines and Amides	
Methyl Amine	8.97
Ethyl Amine	8.86
N-Propyl amine	8.78
Formamide	10.25
Acetamide	9.77
Nitromethane	11.08
Nitroethane	10.88
Other Aliphatic Molecules With an N Atom	
HCN	13.91
Acetonitrile	12.22
Propionitrile	11.84
Olefins, Cyclo-Olefins, Acetylenes	
Ethylene	10.515
Propylene	9.73
1-Butene	9.58
Trans-2-Butene	9.13
1-Hexane	9.46
1,3-Butadiene	9.07
Acetylene	11.41
1-Butyne	10.18
Some Derivatives of Olefins	
Vinyl Chloride	9.995
Trichloroethylene	9.45
Tetrachloroethylene	9.32
Vinyl Bromide	9.80

Table 1

Ionization Potentials (eV)
(continued)

Some Derivatives of Olefins (con't)		
	3-Chloropropene	10.04
	Acrolein	10.10
	Crotonaldehyde	9.73
	Vinyl Acetate	9.19
Heterocyclic Molecules		
	Pyrrole	8.20
	Pyridine	9.32
	Furan	8.89
	Tetrahydrofuran	9.54
	Thiophene	8.880
Aromatic Compounds		
	Benzene	9.245
	Toluene	8.82
	Ethylbenzene	8.76
	Biphenyl	8.27
	Phenol	8.50
	Naphthalene	8.12
	Styrene	8.47
	O-xylene	8.58
	P-Xylene	8.445
	Mestylene	8.40
	Aniline	7.70
	Fluoro-Benzene	9.195
	Chloro-Benzene	9.07
	Bromo-Benzene	8.98
	Iodo-Benzene	8.73