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Office Copy

9 September 1999

Mr. Russell D. Hart
Remedial Project Manager (HSRW-6J)
U.S. Environmental Protection Agency
Region V
77 West Jackson Boulevard
Chicago, IL 60604

RFW Work Order No. 02787.007.003
KMC Work Order No. 40-50-01-AKW-A

Re: Revised QAPP for Installation of Groundwater Remedial System
Moss-American Site
Milwaukee, Wisconsin

Dear Mr. Hart:

Enclosed are two copies of the revised QAPP. Responses to U.S. EPA and WDNR comments are also attached to this letter. Please note that, in accordance with U.S. EPA guidance, only revised pages of the QAPP or appendices are included in this document.

Should you have any questions or comments, please contact Keith Watson at (405) 270-3747 or me at (847) 918-4142.

Very truly yours

ROY F. WESTON, INC.

Thomas P. Graan

Thomas P. Graan, Ph.D.
Principal Project Manager

TPG/slr
Enclosures

cc: G. Edelstein, WDNR
R. Meserve, Covington & Burling

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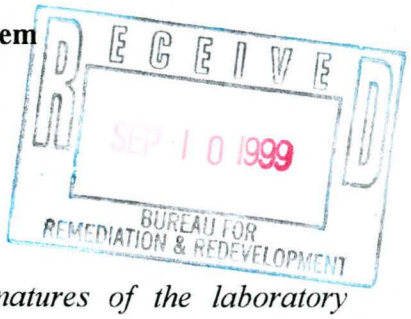
Response to Comments
Quality Assurance Project Plan for
Installation of Groundwater Remedial System
Moss-American Site
Milwaukee, Wisconsin



RESPONSE TO U.S. EPA COMMENTS

U.S. EPA Comment I: *Signature page should include the signatures of the laboratory personnel.*

KMC Response: The signature page has been revised to include the signature of Ms. Kathleen Loewen, the Quality Assurance / Quality Control Manager (QA/QC) Manager for Lancaster Laboratories.

U.S. EPA Comment II (1): *Section 2.4.2, page 2-8. The list of water discharge requirements including all environmental measurements to be performed and project required action limits for these measurements should be provided/referenced in this section.*

KMC Response: See Section 2.4.2, Page 2-9 and Table 2-1 of the QAPP.

U.S. EPA Comment II (2): *Section 2.5.2, page 2-9. The list of the parameters to characterize the excavated soils and action limits for these parameters should be provided in the QAPP.*

KMC Response: See Section 2.5.2, Page 2-11 and Table 2-2 of the QAPP.

U.S. EPA Comment II (3): *Section 2.8, step 2, page 2-12. The frequency, total number and the parameters of the interest of the soil verification samples should be identified.*

KMC Response: See Section 2.8, Step 2, Page 2-15 of the QAPP and Table 2-1 of the Field Sampling Plan (FSP).

U.S. EPA Comment II (4): *Section 2.8, step 3, page 2-13, bullet 6. The action limits for the triggering the thermal desorption of the soil samples should be identified.*

KMC Response: The action limits for triggering the Thermal Desorption of Soil are shown in Table 2-2 of the QAPP (Referenced in Section 2.8, Step 3, Page 2-17).

U.S. EPA Comment III (1): *The explanation should be provided in Section 3 whether Weston and KMC have the same responsibilities for this project.*

KMC Response: KMC has the overall responsibility for this project. WESTON has been retained by KMC to provide overall consulting services. See Section 3, Page 3-1, 1st Paragraph.

U.S. EPA Comment III (2): *The address of laboratory used for the project should be*

included in the section.

KMC Response: See Section 3.4, Page 3-6 of the QAPP.

U.S. EPA Comment III (3): *Section 3.2.7. Identify whether Weston Project Manager will be involved in the review and approval of analytical procedures.*

KMC Response: Both, KMC and WESTON project managers will review and approve analytical procedures. See Section 3.2.7, Page 3-5.

U.S. EPA Comment IV (1): *A summary table including acceptable limits required for the project (Project Quantitation limits) and laboratory method detection limits should be provided in this section. Referenced Table 2-1 of the FSP does not include that information.*

KMC Response: Table 4-1 (Section 4) lists the project quantitation limits and the laboratory method detection limits.

U.S. EPA Comment IV (2): *A summary table of acceptable control limits for all Quality Control samples for all analytes to be quantitated should be included in this section. Not all referenced SOPs include this information.*

KMC Response: Section 11 of the Laboratory QAPP (Tables 11-3 through 11-9) provides the acceptable control limits for the QC samples for each method/parameter.

U.S. EPA Comment IV (3): *Section 4.2 referenced the laboratory QAPP. Any references from the laboratory QAPP should include section and page numbers. The Laboratory QAPP should not be attached as an appendix, only sections applicable to the site specific work should be attached (see Model QAPP DOs and DON?Ts section)*

KMC Response: References to the laboratory QAPP now include section and page numbers (See Section 4.2, Page 4-3). In the future, only sections applicable to the site-specific work will be attached.

U.S. EPA Comment V (1): *Section 2.1.1, page 2-3, second paragraph. The criteria used for determining whether soils are clean or in need for further characterization should be provided in this FSP.*

KMC Response: The criteria that will be used to determine whether soils are clean or in need for further characterization have been included in Section 2.1.1, Page 2-3, Second paragraph of the FSP.

U.S. EPA Comment V (2): *Section 2.1.1, page 2-4, third paragraph. The soil cleanup standards for the site should be provided.*

KMC Response: The soil cleanup standards have been included in Table 2-2 (Section 2) of the FSP (Appendix A).

U.S. EPA Comment V (3): *Section 6.2. The examples of field Chain-of-Custody procedures should be included in this document.*

KMC Response: Field chain-of-custody procedures are discussed in Subsection 6.2 of the QAPP and further discussed in section 6 of the FSP. Example forms and tags as well as examples of completed forms have been provided.

U.S. EPA Comment V (4): *Section 8. It should be identified that the laboratory is procuring contaminant-free pre-cleaned containers that will meet EPA Specifications for Obtaining Contaminant-free Sample containers.*

KMC Response: All sample containers will be prepared according to the procedures specified in U.S. EPA's Specifications and Guidance for obtaining Contaminant-Free Sample Containers (U. S. EPA, 1992). See Section 8, Page 8-1.

U.S. EPA Comment VI (1): *Examples of Chain-of Custody should be included/referenced in the QAPP.*

KMC Response: An example of the COC form is being provided as Appendix F.

U.S. EPA Comment VI (2): *The length of time that the file will be maintained should be specified in Section 6.4. The file must be offered to U.S. EPA prior to disposal.*

KMC Response: The evidence files and its contents will be retained for six years following the sixth "five-year review" conducted for the site. All files will be offered to the U. S. EPA prior to disposal. See Section 6.4, Page 6-8.

U.S. EPA Comment VII (1): *One of the project objectives is installation of groundwater monitoring and free-product extraction wells (page 2-9). The water samples should be analyzed for pH, temperature, and conductivity to determine whether the well is stabilized. The SOPs for these field parameters should be attached and calibration procedures can be referenced to the SOPs.*

KMC Response: The Standard Operating Procedures (SOPs) for measuring the pH, temperature, and conductivity of water are being submitted with this submittal (Appendix E).

U.S. EPA Comment VIII (1): *The action limits for the project should be provided in addition to the laboratory detection limits.*

KMC Response: The action limits for the project are being provided in Table 2-1 and 2-2 of the QAPP.

U.S. EPA Comment VIII (2): *SOPs and Quality Control Samples acceptance criteria*

should be provided for all field analysis.

KMC Response: The Standard Operating Procedures (SOPs) for measuring the pH, temperature, and conductivity of water are being submitted with this submittal (Appendix E). Accuracy and precision limits for field measurements are being provided in Section 4.2.

U.S. EPA Comment VIII (3): *The following should be addressed / corrected in Table 8:*

U.S. EPA Comment VIII (3) (a): *The name of the compounds of interest should be the same (Methyl bromide, methyl chloride) as in the SOPs.*

KMC Response: Our recent conversations with personnel of the Milwaukee Metropolitan Sewerage District (MMSD) indicate that methyl bromide and methyl chloride analysis of the water samples is not required. Consequently, reference to methyl bromide and methyl chloride analysis has been removed from the QAPP and the FSP.

U.S. EPA Comment VIII (3) (b): *The detection limit for TSS is higher that detection limit in the SOP. The discrepancy should be corrected.*

KMC Response: The initial SOP submitted for TSS should be removed. An updated SOP is being submitted with this submittal. The detection limits listed in the QAPP are correct.

U.S. EPA Comment VIII (3) (c): *The detection limits (DLs) for naphthalene in soil (270 ug/kg) and lead in water (100 ug/l) are high. If these DLS are meeting the project required action limits, than it's acceptable. The explanation should be provided.*

KMC Response: Project required action limits are detailed in QAPP tables 2-1 and 2-2. The laboratory and project detection limits for naphthalene and lead are detailed in Table 4-1and 8-1.

U.S. EPA Comment VIII (3) (d): *The detection limit for pH in water sample is 10 ug/l. This should be corrected.*

KMC Response: Our recent conversations with personnel of the Milwaukee Metropolitan Sewerage District (MMSD) indicate that pH analysis of the water samples is not required. Consequently, reference to pH analysis has been removed from the QAPP and the FSP. Therefore, response to this comment is not required.

U.S. EPA Comment VIII (3) (e): *The laboratory cannot use Oil and Grease method 413.1 with Freon extraction. Method 1664 should be used instead. The laboratory should prepare SOP based on the attached method 1664.*

KMC Response: Our recent conversations with personnel of the Milwaukee Metropolitan Sewerage District (MMSD) indicate that oil and grease analysis of the water samples is not required. Consequently, reference to oil and grease analysis has been removed from the QAPP and the FSP. Therefore, response to this comment is not required.

U.S. EPA Comment VIII (4): *The Laboratory should use EPA Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality Related Documents, EPA QA/G-6, November 1995. The following are the comments on the attached SOPs:*

In future submittals, the laboratory will be requested to prepare new SOPs according to the above guidance. Conversations with the U.S. EPA QAPP reviewer indicated that current SOPs will not be required to be rewritten.

U.S. EPA Comment VIII (4) (A):

SOP for Purgeable Aromatics in High-Level Soils (BTEX compounds):

- *The detection limits for BTEX compounds are different from the detection limits in Table 8.1 of the QAPP. Correct the discrepancy.*

- *The FSP in Table 5-1 identifies that samples will be collected using the Encore. The Sample Collection Section of SOP should include the preservation and holding time.*

KMC Response: A revised SOP for BTEX is being submitted. The detection limits listed in the QAPP are the correct limits. Holding time are preservation requirements have been updated.

U.S. EPA Comment VIII (4) (B):

SOP for PAHs

- The extraction/cleanup SOP should be provided for the review.

KMC Response: The extraction SOP for PAH for water and soil by HPLC (8310) is being submitted.

U.S. EPA Comment VIII (4) (C):

SOP for Determination of Semivolatile Organic Compound By Method 8270.

- *Table 8-1 of QAPP did not referenced method 8270 for any SVOC analyzed for this site. The SOP did not provide the analytes list either. The comments will not be provided for this SOP.*

KMC Response: The SOP for Semivolatile Organic Compounds by Method 8270 was inadvertently submitted. This SOP should be removed from the original submission. This SOP is for a different phase of the project that has been in progress for several years.

U.S. EPA Comment VIII (4) (D):

SOP for The Analysis of Water for Purgeable Organics by Purge and Trap GC/MS.

- *Table 8-1 of the QAPP referenced method SW 8021 for VOC analysis, while the SOP referencing only Method 624. Please correct the discrepancy.*

- *The detection limits in Table 8.1 of the QAPP and PQL in Tables 1A-G are different. Please resolve.*

KMC Response: Water samples will be analyzed by Method 8260B as indicated in the QAPP. Detection limits listed in the QAPP are the correct limits and correspond to the detection limits listed in Section 9 of the laboratory QAPP (Appendix D).

U.S. EPA Comment VIII (4) (E):

SOP for Determination of Priority Pollutants by Method 625.

- *The preparation SOP should be provided for the review.*

- *The LOQ is 10 ug/l, the calibration standards in item D on page 5 are 5, 50, 80, 120 and 160 ppm, the calibration check standard is 80 ppm . This should be corrected.*

KMC Response: Our recent conversations with personnel of the Milwaukee Metropolitan Sewerage District (MMSD) indicate that PAH analysis of the water samples is not required. Consequently, reference to water PAH analysis has been removed from the QAPP and the FSP.

U.S. EPA Comment VIII (4) (F):

SOP for Sample Preparation of potable Water and Wastewater for Total Mercury Analysis by Cold Vapor Technique

- The Hg SOP is not included in the package, only preparation SOP provided. Use EPA QA/G-6 document for SOP preparation.

KMC Response: A Hg SOP and digestion SOP are being submitted.

U.S. EPA Comment VIII (4) (G):

SOP for Metals Analysis by Graphite Furnace AA and ICP

- *Groundwater samples for metal analysis from same site should be treated same way. All groundwater samples **should be digested** for ICP analysis. The digestion SOP should be submitted for the review.*

- *The SOP should include all metals of interest, their detection limits, interferences, calibration procedures, QC acceptance criteria and etc.. See EPA QA/G-6 document.*

KMC Response: A SOP detailing metal analysis by ICP (which explains the laboratories acceptance limits) is being submitted. Also, it should be noted that chromium is being added as a analytical parameter for water based on discussions with the Milwaukee Metropolitan Sewerage District.

U.S. EPA Comment VIII (4) (H):

SOP for Total Suspended Solids

- *The volume used for sample analysis should be specified in item 4.b.*

KMC Response: A procedural amendment to the SOP for Total Suspended Solids is being submitted that includes the volume of sample used for analysis.

U.S. EPA Comment IX (1): *It will be a time saving for the QAPP reviewer and for data validator, if the list of all laboratory Quality control samples and their acceptance requirements be included in the table format in this section.*

KMC Response: Quality Control (QC) checks for the laboratory analysis are identified in the corresponding SOPs in Appendix C. The QC acceptance limits are also summarized in the laboratory QAPP in Appendix D (Tables 11.3 through 11.9). See Section 9.3, Page 9-1.

U.S. EPA Comment X (1): *The pH, temperature and conductivity should be done in the field before collecting groundwater samples for the analysis. This should be added in Section 10.1.*

KMC Response: Groundwater samples will not be collected during the construction of the groundwater remedial system. Nevertheless, pH, temperature, and conductivity measurements will be performed during the development of the newly installed groundwater wells. These measurements will be necessary to confirm that the wells have been installed in accordance with the specifications.

U.S. EPA Comment X (2): *Section 10.2.2. The CLP methods will not be used for the analysis. Different criteria and identifiers should be used. The details of the data validation should be provided in this section.*

KMC Response: See Section 10.2.2, Page 10-2.

U.S. EPA Comment XI (1): *A list of critical spare part necessary for maintaining the field and laboratory equipment should be provided in this section in tabular format. Use Model QAPP as an example.*

KMC Response: See section 12.1 and 12.2. All group leaders maintain a list for inventory purposes.

U.S. EPA Comment XIII (1): *The characterization of potentially contaminant soils by PID should include some field measurement data. The first sentence should be changed in Section 13.1. See comment VII.1 of the memo to include additional field instruments.*

KMC Response: See Section 13.1, Page 13-1.

U.S. EPA Comment XIII (2): *The completeness of the field data should be provided.*

KMC Response: See Section 13.1, Page 13-1 of the QAPP.

U.S. EPA Comment XIII (3): *Section 4 of QAPP referenced for the accuracy of the laboratory results in Section 13.2.2. Section 4 of the QAPP does not include the referenced information. SOPs should be referenced in this section.*

KMC Response: Section 4 has been updated to include this information. Section 11 of the laboratory QAPP (Tables 11-3through 11-9) provides the acceptance control limits for the QC samples for each method/parameter.

U.S. EPA Comment XIII (4): The completeness of the laboratory data should identified in Section 13.2.3.

KMC Response: See Section 13.1, Page 13-1 of the QAPP.

U.S. EPA Comment XV (1): *The frequency of the reports to the RPM should be identified.*

KMC Response: See Section 15, Page 15-1.

RESPONSE TO WNDR COMMENTS

WDNR Comment 1: *QAPP Sections 2.4.1 and 2.5.3 and FSP Section 2.1.1, Cleanup Levels – Naphthalene contaminated soils are subject to the ROD cleanup levels based on state ARARs and your letter of May 11, 1999. We understand that letter postpones the requirement for excavation and subsequent treatment of naphthalene contaminated soils that exceed the generic groundwater RCL of 0.4 mg/kg but are below 100 mg/kg. We understand this postponement only applies to in-place soils, not soils excavated as part of the groundwater remediation construction that are to be redisposed on-site. The letter does not postpone or suspend the ROD requirement to treat such soils once they are excavated. Therefore, such soils should be stored in the lined temporary storage unit and subsequently treated.*

KMC Response: KMC understands this postponement to apply to all site soils based on U.S. EPA's 11 May 1999 letter. Excavated soils that would not require treatment at this time but that exceed the generic naphthalene groundwater RCL of 0.4 mg/kg would be stored in the unlined temporary storage until subsequent placement at the site.

WDNR Comment 2: *QAPP Section 2.4.2, Water Discharge to MMSD – Per Weston's letter and plan of June 12, 1998, we understand the discharge to the MMSD would be through a direct force main connection, not by hauling to a manhole. We understand hauling to a manhole would require hazardous waste manifesting requirements be met. Finally, confirmation that MMSD has approved of this discharge (issue a pretreatment permit) should be provided.*

KMC Response: Our response to WDNR Condition No. 4, in our letter dated 12 June, 1998, states that the MMSD will not accept trucked in waste at their treatment works. The response further states that the waste liquids are typically discharged into the closest manhole as detailed in the design report. The approved design report dated 11 March 1998 clearly states that the treated water will be pumped to tanker trucks for transportation and discharge to the sanitary sewer.

A hazardous waste manifest will be required only if the water is transported and discharged to an off-site manhole. Since the treated water will be discharged to an on-site sanitary sewer, hazardous manifesting will not be required. Please note that a sanitary sewer line has been located on the site and a permit has been obtained from the MMSD. The MMSD permit is being provided as Attachment A.

WDNR Comment 3: *QAPP Table 8-1 and appropriate sections in the FSP, Detection limit benzene in soil – The ROD RCL for benzene is 5.5 ppb, so the soil detection limit (LOQ) for benzene must be low enough to confirm this level. The current LOQ proposed is 20 ppb.*

KMC Response: Normal laboratory procedure is to set the detection limit (LOQ) 5 to 10 times the laboratory determined method detection limit. The laboratory will report all benzene detections greater than the MDL of 4 ug/L. Benzene detections between the MDL and project detection limit of 20 ug/L will be flagged with a J, indicating an estimated value. Therefore, values(hits) at or above the ROD RCL of 5 ppb will be reported.

ATTACHMENT A

MMSD PERMIT

Milwaukee Metropolitan Sewerage District
260 West Seeboth Street
P.O. Box 3049
Milwaukee, Wisconsin 53201-3049
(414) 272-5100

July 20, 1999

Mr. Deepak L. Bhojwani
Kerr McGee Chemical, LLC
c/o Roy F. Weston, Inc.
Three Hawthorn Parkway – Suite 400
Vernon Hills, IL 60061-1450

Subject: Notice of Intent 99.032
Moss American Site

Dear Mr. Bhojwani:

The Milwaukee Metropolitan Sewerage District (MMSD or District) has reviewed the Notice of Intent submitted by Roy F. Weston, Inc. for a one (1) time discharge of contaminated groundwater from the Moss American Site located at 8716 North Grandview Road, Milwaukee.

The source of the contaminated groundwater will be from groundwater located on the property during excavation.

The proposed discharge from the Moss American Site property is approved subject to the following conditions:

1. The Moss American Site must comply with the general and specific limits set forth in Chapter 11, MMSD Rules.

2. The Moss American Site must allow the District access to the site for inspection or sampling.

3. The Moss American Site must receive written approval from the City of Milwaukee for the discharge to the local sanitary sewer system and verification that their sewer system has the capacity to carry the additional flow. A copy of this letter must be sent to the District.

4. The discharge must occur to the sanitary sewer connection chosen by the City of Milwaukee.

5. Before commencement of discharge the Moss American Site must construct a facility for monitoring the volume of groundwater discharged for collecting grab samples. This facility must be inspected by a representative of the District (Mr. Chris Schultz, General Supervisor/ Monitoring at 325-5136).

6. In the groundwater discharged to the sewer system, the following pollutant levels may not be exceeded.

Cadmium	1.5 mg/l
Chromium – Total	8.0 mg/l
Copper	6.0 mg/l
Lead	2.0 mg/l
Mercury – Total	0.0026 mg/l
Nickel	4.0 mg/l
Zinc	8.0 mg/l
Total Suspended Solids	100 mg/l
Total VOC Scan	<5 mg/l

7. The Moss American Site must take one (1) grab sample per week for all the above pollutants. This sample must be taken when pump discharge is at its highest. All sample results must be faxed to the District (325-5102) as soon as they are available.

8. The District may also sample the discharged groundwater at anytime. The cost of the sample collection and analysis will be billed directly to the Moss American Site.

9. The Moss American Site must notify Mr. Chris Schultz when the discharge begins, when the discharge ends, and if any problems should occur to the monitoring facility.

10. No discharge may occur during periods of at least 0.5 inches of rain. If it does rain, the Moss American Site must cease all discharge to the sewer system. Commencement of the discharge cannot occur until 12 hours after the rain has stopped.

11. The total volume of water discharged should be reported to Mr. Chris Schultz when pumping of excavation is completed. The Moss American Site will be billed at a rate of $1.05 per 1000 gallons discharged.

The policies and procedures for these types of discharges to the sewer system are currently being studied by the District. You should be aware that this approval may be rescinded at any time or changes to your conditions may occur.

If the Moss American Site is in violation of this letter of approval at any time, the District may taken enforcement actions which may include but are not limited to issuing a Notice of Noncompliance or Notice of Violation or seeking injunctive or civil penalties up to $10,000 per day per violation.

Enclosed is your check in the amount of $344.00. We are returning the check because payment was not required for this Notice of Intent.

If you have any questions concerning this matter, please contact me at 225-2176.

Sincerely,

Peter R. Topczewski
Manager of Industrial Waste
& Conveyance Monitoring

PRT:HJM:jds

Cc: City of Milwaukee
Chris Schultz
Harvey Matyas
NOI File

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

Prepared for

Kerr-McGee Chemical, LLC
Oklahoma City, Oklahoma

Prepared by

ROY F. WESTON, INC.
Three Hawthorn Parkway
Vernon Hills, Illinois 60061

September 1999

Work Order No. 02687.007.003

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

September 1999

Prepared By: _____ Date: 9/7/99
Tonya Balla, WESTON
Project Engineer

Approved By: _____ Date: 8/23/99
Kathleen M. Loewen, Lancaster Laboratories
QA/QC Manager

Approved By: _____ Date: 9/9/99
Thomas Graan WESTON
Project Manager

Approved By: _____ Date: 9/9/99
Kurt Stimpson, WESTON
Project Director

Approved By: _____ Date: 9/9/99
A. Keith Watson, Kerr-McGee Chemical, LLC
Project Manager

Approved By: _____ Date: _____
Russell Hart, U.S. EPA
Remedial Project Manager

Approved By: _____ Date: _____
U.S. EPA Quality Assurance Reviewer

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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 would require appropriate management.

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 total volatile organic compounds (VOCs), total metals including cadmium, chromium, copper, lead, mercury, nickel, zinc, and total suspended solids (TSS). All analytical methods will be in accordance with the requirements of MMSD. Table 2-1 lists the water discharge requirements including all environmental measurements to be performed and the project required action limits for these measurements.

Table 2-1

Analytical Parameters and Discharge Standards for Water
Moss-American Site
Milwaukee, Wisconsin

Analytical Parameter	Discharge Standard [1]
Volatile Organic Compounds (VOCs)	
Total VOC Scan[2], mg/L	5
Metals	
Chromium (total), mg/L	8
Copper, mg/L	6
Lead, mg/L	2
Mercury (total), ug/L	2.6
Nickel, mg/L	4
Zinc, mg/L	8
Other Parameters	
Total Suspended Solids, mg/L	100

1 Based on Milwaukee Metropolitan Sewerage District's pretreatment standards.
2 See Table 4-1 of the QAPP for individual Organic Compounds.

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. The list of parameters to characterize the excavated soils and the action limits for these parameters are shown in Table 2-2. Characterization samples will be collected at a frequency of 1 sample per 200 cubic yards of excavated soil. A total of 100 characterization samples are expected to be collected and analyzed for the parameters indicated in Table 2-2. Table 2-1 of the Field Sampling Plan (FSP) presents the analytical parameters and frequencies associated with the characterization samples. Soil will be segregated and staged in stockpiles based on contaminant levels (e.g., soil requiring treatment and cover, soil requiring cover only, and uncontaminated soil).

The list of parameters and the action limits associated with the water samples are shown in Table 2-1. Frequency of water samples is detailed in Table 2-1 of the FSP (Appendix A).

Table 2-2

Analytical Parameters and Cleanup Levels (Action Limits) for Soil
Moss-American Site
Milwaukee, Wisconsin

Analytical Parameters	Action Limits, mg/kg	
	Soil Requiring Treatment and Cover	Soil Requiring Cover Only
Total CPAHs[1] (BAP Equivalent)	>78	1.9[2], 15[3]
Benzo(a)pyrene	>48	---
Fluorene	>100	---
Naphthalene	>100[4]	0.4
Benzene	>0.0055	---
Toluene	>1.5	---
Ethylbenzene	>2.9	---
Total xylenes	>4.1	---

-- Parameter does not determine whether soil requires cover.
[1] Total CPAHs include benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, indeno(1,2,3-cd)pyrene.
[2] Direct contact value for residential areas outside the 100-year floodplain.
[3] Direct contact value for residential areas within the 100-year floodplain.
[4] Site-specific interim standard.

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 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 allows 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. Soil that is left in-place and that exceeds the treatment action limits in Table 2-2 will be excavated and staged for treatment.

Verification samples will be collected from the excavation floor and sidewalls at a frequency of one sample per 50 linear feet. Approximately 84 verification samples will be collected and analyzed for the parameters shown in Table 2-2. Table 2-1 of the FSP presents the analytical parameters and the frequencies associated with the verification samples.

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.

Table 2-2 shows the action limits that would trigger thermal desorption of soil.

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.

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. KMC has overall operational responsibility for this project. WESTON has been retained by KMC to provide overall consulting services.

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 and WESTON Project Managers 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 Inc. Lancaster Laboratories, located at 2425 New Holland Pike (P.O. Box12425) in Lancaster, Pennsylvania, is a division of Thermo Analytical Services. 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.

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. Table 4-1 presents the project required analytical methods, project detection limits, and laboratory method detection limits. The accuracy and precision requirements are summarized in the laboratory QAPP (Appendix B) and laboratory SOPs (Appendix C). Section 11 of the laboratory QAPP (Tables 11-3 through 11-9) provides the acceptable control limits for the QC samples for each method/parameter.

Field equipment to be used for field measurements (groundwater) for pH, conductivity, and temperature, is outlined in the SOPs in Appendix E. Accuracy and precision requirements for field screening analysis are provided in the FSP (Appendix A) and in the SOPs for field instruments

Table 4-1

Project Detection Limits and Method Detection Limits
Moss-American Site
Milwaukee, Wisconsin

Parameter	Soil Method	Soil Project Detection Limit (ug/kg)	Soil Method Detection Limit (ug/kg)	Water Method	Water Project Detection Limit (ug/L)	Water Method Detection Limit (ug/L)
Volatile Organic Compounds						
Benzene	8021B	20	4	8260B	1	0.2
Bromobenzene	---	---	---	8260B	1	0.2
Bromochloromethane	---	---	---	8260B	1	0.2
Bromodichloromethane	---	---	---	8260B	1	0.2
Bromoform	---	---	---	8260B	1	0.2
Bromomethane	---	---	---	8260B	5	0.5
n-Butylbenzene	---	---	---	8260B	1	0.2
sec-Butylbenzene	---	---	---	8260B	1	0.2
tert-Butylbenzene	---	---	---	8260B	1	0.2
Carbon tetrachloride	---	---	---	8260B	1	0.2
Chlorobenzene	---	---	---	8260B	1	0.2
Chlorodibromomethane	---	---	---	8260B	1	0.2
Chloroethane	---	---	---	8260B	1	0.2
Chloroform	---	---	---	8260B	1	0.2
Chloromethane	---	---	---	8260B	5	0.5
2-Chlorotoluene	---	---	---	8260B	1	0.2
4-Chlorotoluene	---	---	---	8260B	1	0.2
1,2-Dibromo-3-Chloropropane	---	---	---	8260B	1	0.2
1,2-Dibromomethane	---	---	---	8260B	1	0.2
Dibromomethane	---	---	---	8260B	1	0.2
1,2-Dichlorobenzene	---	---	---	8260B	1	0.2
1,3-Dichlorobenzene	---	---	---	8260B	1	0.2
1,4-Dichlorobenzene	---	---	---	8260B	1	0.2
Dichlorofluoromethane	---	---	---	8260B	2	0.2
1,1-Dichloroethane	---	---	---	8260B	1	0.2
1,2-Dichloroethane	---	---	---	8260B	1	0.2
1,1-Dichloroethene	---	---	---	8260B	1	0.2
cis-1,2-Dichloroethene	---	---	---	8260B	1	0.2
trans-1,2,-Dichloroethene	---	---	---	8260B	1	0.2
1,2-Dichloropropane	---	---	---	8260B	1	0.2
1,3-Dichloropropane	---	---	---	8260B	1	0.2
2,2-Dichloropropane	---	---	---	8260B	1	0.2
1,1-Dichloropropene	---	---	---	8260B	1	0.2
cis-1,3-Dichloropropene	---	---	---	8260B	1	0.2
trans-1,3-Dichloropropene	---	---	---	8260B	1	0.2

Table 4-1

Project Detection Limits and Method Detection Limits
Moss-American Site
Milwaukee, Wisconsin
(Continued)

Parameter	Soil Method	Soil Project Detection Limit (ug/kg)	Soil Method Detection Limit (ug/kg)	Water Method	Water Project Detection Limit (ug/L)	Water Method Detection Limit (ug/L)
Ethylbenzene	8021B	20	4	8260B	1	0.2
Hexachlorobutadiene	---	---	---	8260B	1	0.2
Isopropylbenzene	---	---	---	8260B	1	0.2
p-Isopropyltoluene	---	---	---	8260B	1	0.2
Methylene Chloride	---	---	---	8260B	1	0.2
Methyl-t-butyl ether	---	---	---	8260B	1	0.2
Napthalene	---	---	---	8260B	1	0.2
n-Propylbenzene	---	---	---	8260B	1	0.2
Styrene	---	---	---	8260B	1	0.2
1,1,1,2-Tetrachloroethane	---	---	---	8260B	1	0.2
1,1,2,2-Tetrachloroethane	---	---	---	8260B	1	0.2
Tetrachloroethane	---	---	---	8260B	1	0.2
Toluene	8021B	20	4	8260B	1	0.2
1,2,3-Trichlorobenzene	---	---	---	8260B	1	0.2
1,2,4-Trichlorobenzene	---	---	---	8260B	1	0.2
1,1,1-Trichloroethane	---	---	---	8260B	1	0.2
1,1,2-Trichloroethane	---	---	---	8260B	1	0.2
Trichlorofluoromethane	---	---	---	8260B	1	0.2
1,2,3-Trichloropropane	---	---	---	8260B	1	0.2
1,2,4-Trimethylbenzene	---	---	---	8260B	1	0.2
1,3,5-Trimethylbenzene	---	---	---	8260B	1	0.2
Vinyl chloride	---	---	---	8260B	1	0.2
m,p-Xylene	---	---	---	8260B	2	0.4
O-xylene	---	---	---	8260B	1	0.2
Total Xylene	8021B	20	4	---	---	---
Semivolatile Organic Compounds						
Benzo(a)anthracene	8310	3	0.25	---	---	---
Chyrsene	8310	11	1.0	---	---	---
Dibenzo(a,h)anthracene	8310	5	0.5	---	---	---
Benzo(b)fluoranthene	8310	2	0.2	---	---	---
Benzo(k)fluoranthene	8310	2	0.2	---	---	---
Benzo(g,h,i)perylene	8310	16	1.5	---	---	---
Benzo(a)pyrene	8310	3	0.25	---	---	---
Indeno(1,2,3-cd)pyrene	8310	11	1.0	---	---	---
Fluorene	8310	27	2.5	---	---	---

Table 4-1

Project Detection Limits and Method Detection Limits
Moss-American Site
Milwaukee, Wisconsin
(Continued)

Parameter	Soil Method	Soil Project Detection Limit (ug/kg)	Soil Method Detection Limit (ug/kg)	Water Method	Water Project Detection Limit (ug/L)	Water Method Detection Limit (ug/L)
Naphthalene	8310	270	27	---	---	---
Metals						
Cadmium	---	---	---	200.7	10	2.7
Chromium (total)	---	---	---	200.7	30	5.4
Copper	---	---	---	200.7	25	3.8
Lead	---	---	---	200.7	100	2.0
Mercury	---	---	---	245.1	0.2	0.043
Nickel	---	---	---	200.7	50	5.4
Silver	---	---	---	200.7	20	3.6
Zinc	---	---	---	200.7	25	12
Other Analytical Parameters						
Total Suspended Solids(TSS)	---	---	---	160.2	900	336

Note: Specific detection limits are highly matrix dependent. The detection limits listed above are provided for guidance and may not be achievable. The laboratory can estimate down to the method detection limit. Values reported below the project detection limit are reported with a J-flag and are defined as estimated values.
Water VOCs action limits are based on Total VOCs (5 mg/L).

--- Analysis not required.

(Appendix E). A PID will be used for screening the excavated soil and 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 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. An example of the COC form is included as Appendix F. 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. The evidence file and its contents will be retained for six years following the sixth "five-year review." All files will be offered to the U.S. EPA prior to disposal.

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 detector (PID) for screening the soil and for personnel health and safety.
- pH meter.
- Conductivity meter.
- Thermometer.

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:

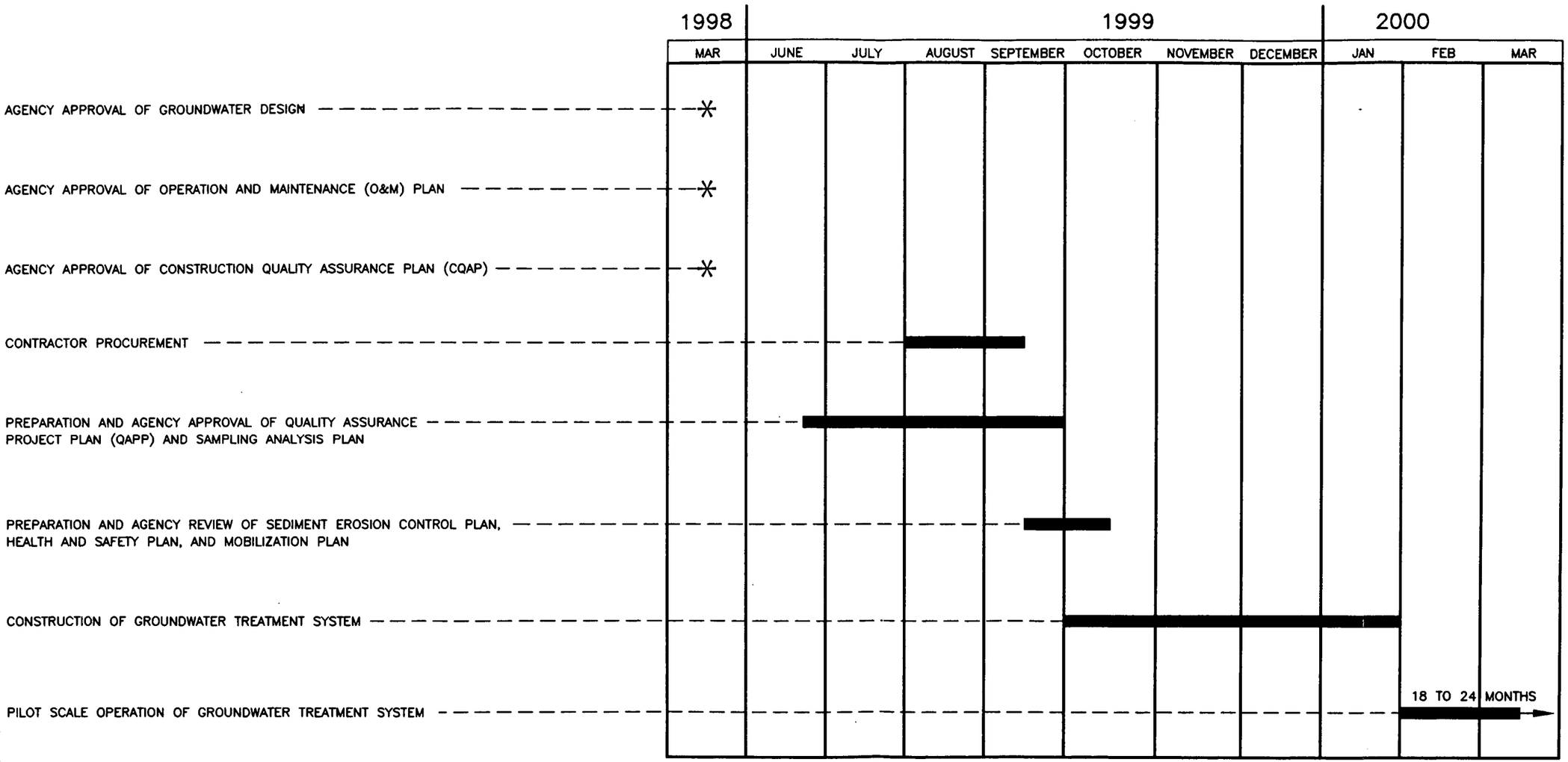
- Name of device or instrument calibrated.
- Device or instrument serial or identification (I.D.) number.
- Frequency of calibration.
- Results of calibration.
- Name of person performing the calibration.
- Identification of the calibration media (e.g., pH buffer solutions).

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.



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* – U.S. EPA APPROVAL

(REVISED ON 8 SEPTEMBER 1999)

FIGURE 2-2

WESTON MANAGERS DESIGNERS/CONSULTANTS

Three Hawthorn Parkway
Vernon Hills, Illinois
60061

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

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 water samples will be analyzed for volatile organic and semivolatile organic or PAH constituents. Water samples will also be analyzed for total Vocs, metals (cadmium, chromium, copper, lead, mercury, nickel, silver, and zinc), 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. QC acceptance criteria is presented in the laboratory QAPP (Appendix B Tables 11.3 through 11.9).

8.2 FIELD SCREENING ANALYTICAL PROTOCOLS

The procedures associated with the field measurements for groundwater(pH, temperature, and conductivity) are described in the SOPs presented in Appendix E.

Table 8-1

Project Detection Limits and Method Detection Limits
Moss-American Site
Milwaukee, Wisconsin

Parameter	Soil Method	Soil Project Detection Limit (ug/kg)	Soil Method Detection Limit (ug/kg)	Water Method	Water Project Detection Limit (ug/L)	Water Method Detection Limit (ug/L)
Volatile Organic Compounds						
Benzene	8021B	20	4	8260B	1	0.2
Bromobenzene	---	---	---	8260B	1	0.2
Bromochloromethane	---	---	---	8260B	1	0.2
Bromodichloromethane	---	---	---	8260B	1	0.2
Bromoform	---	---	---	8260B	1	0.2
Bromomethane	---	---	---	8260B	5	0.5
n-Butylbenzene	---	---	---	8260B	1	0.2
sec-Butylbenzene	---	---	---	8260B	1	0.2
tert-Butylbenzene	---	---	---	8260B	1	0.2
Carbon tetrachloride	---	---	---	8260B	1	0.2
Chlorobenzene	---	---	---	8260B	1	0.2
Chlorodibromomethane	---	---	---	8260B	1	0.2
Chloroethane	---	---	---	8260B	1	0.2
Chloroform	---	---	---	8260B	1	0.2
Chloromethane	---	---	---	8260B	5	0.5
2-Chlorotoluene	---	---	---	8260B	1	0.2
4-Chlorotoluene	---	---	---	8260B	1	0.2
1,2-Dibromo-3-Chloropropane	---	---	---	8260B	1	0.2
1,2-Dibromomethane	---	---	---	8260B	1	0.2
Dibromomethane	---	---	---	8260B	1	0.2
1,2-Dichlorobenzene	---	---	---	8260B	1	0.2
1,3-Dichlorobenzene	---	---	---	8260B	1	0.2
1,4-Dichlorobenzene	---	---	---	8260B	1	0.2
Dichlorofluoromethane	---	---	---	8260B	2	0.2
1,1-Dichloroethane	---	---	---	8260B	1	0.2
1,2-Dichloroethane	---	---	---	8260B	1	0.2
1,1-Dichloroethene	---	---	---	8260B	1	0.2
cis-1,2-Dichloroethene	---	---	---	8260B	1	0.2
trans-1,2,-Dichloroethene	---	---	---	8260B	1	0.2
1,2-Dichloropropane	---	---	---	8260B	1	0.2
1,3-Dichloropropane	---	---	---	8260B	1	0.2
2,2-Dichloropropane	---	---	---	8260B	1	0.2
1,1-Dichloropropene	---	---	---	8260B	1	0.2
cis-1,3-Dichloropropene	---	---	---	8260B	1	0.2
trans-1,3-Dichloropropene	---	---	---	8260B	1	0.2

Table 8-1

Project Detection Limits and Method Detection Limits
Moss-American Site
Milwaukee, Wisconsin
(Continued)

Parameter	Soil Method	Soil Project Detection Limit (ug/kg)	Soil Method Detection Limit (ug/kg)	Water Method	Water Project Detection Limit (ug/L)	Water Method Detection Limit (ug/L)
Ethylbenzene	8021B	20	4	8260B	1	0.2
Hexachlorobutadiene	---	---	---	8260B	1	0.2
Isopropylbenzene	---	---	---	8260B	1	0.2
p-Isopropyltoluene	---	---	---	8260B	1	0.2
Methylene Chloride	---	---	---	8260B	1	0.2
Methyl-t-butyl ether	---	---	---	8260B	**1**	**0.2**
Napthalene	---	---	---	8260B	1	0.2
n-Propylbenzene	---	---	---	8260B	1	0.2
Styrene	---	---	---	8260B	1	0.2
1,1,1,2-Tetrachloroethane	---	---	---	8260B	1	0.2
1,1,2,2-Tetrachloroethane	---	---	---	8260B	1	0.2
Tetrachloroethane	---	---	---	8260B	1	0.2
Toluene	8021B	20	4	8260B	1	0.2
1,2,3-Trichlorobenzene	---	---	---	8260B	1	0.2
1,2,4-Trichlorobenzene	---	---	---	8260B	1	0.2
1,1,1-Trichloroethane	---	---	---	8260B	1	0.2
1,1,2-Trichloroethane	---	---	---	8260B	1	0.2
Trichlorofluoromethane	---	---	---	8260B	1	0.2
1,2,3-Trichloropropane	---	---	---	8260B	1	0.2
1,2,4-Trimethylbenzene	---	---	---	8260B	1	0.2
1,3,5-Trimethylbenzene	---	---	---	8260B	1	0.2
Vinyl chloride	---	---	---	8260B	1	0.2
m,p-Xylene	---	---	---	8260B	2	0.4
O-xylene	---	---	---	8260B	1	0.2
Total Xylene	8021B	20	4	---	---	---
Semivolatile Organic Compounds						
Benzo(a)anthracene	8310	3	0.25	---	---	---
Chyrsene	8310	11	1.0	---	---	---
Dibenzo(a,h)anthracene	8310	5	0.5	---	---	---
Benzo(b)fluoranthene	8310	2	0.2	---	---	---
Benzo(k)fluoranthene	8310	2	0.2	---	---	---
Benzo(g,h,i)perylene	8310	16	1.5	---	---	---
Benzo(a)pyrene	8310	3	0.25	---	---	---
Indeno(1,2,3-cd)pyrene	8310	11	1.0	---	---	---
Fluorene	8310	27	2.5	---	---	---
Naphthalene	8310	270	27	---	---	---

Table 8-1

Project Detection Limits and Method Detection Limits
Moss-American Site
Milwaukee, Wisconsin
(Continued)

Parameter	Soil Method	Soil Project Detection Limit (ug/kg)	Soil Method Detection Limit (ug/kg)	Water Method	Water Project Detection Limit (ug/L)	Water Method Detection Limit (ug/L)
Metals						
Cadmium	---	---	---	200.7	10	2.7
Chromium (total)	---	---	---	200.7	30	5.4
Copper	---	---	---	200.7	25	3.8
Lead	---	---	---	200.7	100	2.0
Mercury	---	---	---	245.1	0.2	0.043
Nickel	---	---	---	200.7	50	5.4
Silver	---	---	---	200.7	20	3.6
Zinc	---	---	---	200.7	25	12
Other Analytical Parameters						
Total Suspended Solids(TSS)	---	---	---	160.2	900	336

Note: Specific detection limits are highly matrix dependent. The detection limits listed above are provided for guidance and may not be achievable. The laboratory can estimate down to the method detection limit. Values reported below the project detection limit are reported with a J-flag and are defined as estimated values.
Water VOCs action limits are based on Total VOCs (5 mg/L).

--- 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

QC procedures for pH, conductivity, and temperature are limited to checking the reproducibility of the measurement by obtaining multiple readings on a single or standard, and/or by calibrating the instruments (when appropriate). 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. The QC acceptance limits are also summarized in the laboratory QAPP in Appendix D (Tables 11.3 through 11.9).

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

Raw data from field measurements and sample collection activities will be appropriately recorded in the field logbook. If the data are to be used in the project reports, they will be reduced or summarized, and the method of reduction will be documented in the report.

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 each 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. In general, the overall 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.

However, QC acceptance criteria established in the corresponding laboratory method SOPs, laboratory QAPP (Appendix B - Tables 11.3 through 11.9), and this QAPP will be used to determine data useability.

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 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

A pH, temperature, and conductivity meter will be used for groundwater. Critical spare parts will be available including batteries. A spare meter will be available within one day. The pH, temperature, conductivity meter SOP is included in Appendix E. 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. The laboratory will maintain a complete inventory of replacement parts needed for preventative maintenance and spare parts that routinely need replacement (e.g. septa, gauges, sources, detectors). If an instrument fails, the problem will be diagnosed as quickly as possible, and either replacement parts will be ordered or a service call will be placed to the manufacturer. 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

Field data will be checked for compliance with the established QC criteria that are specified in the QAPP and FSP. Data from screening of excavated soil with a PID and field measurements of pH, specific conductance, and temperature will be assessed by thorough review of QC data (e.g. calibrations, standards, blanks, replicates, documentation that analytical procedures were followed, and reports from system audits).

Accuracy of field measurements will be assessed using daily instrument calibration, calibration checks, and analyses of blanks. Precision will be assessed on the basis of reproducibility by multiple readings on a single sample.

All data will be reviewed for completeness. Data completeness will be as follow:

$$\text{Completeness} = \frac{\text{Valid data obtained}}{\text{Total data planned}} \times 100\%$$

Field measurement completeness is expected to be 90 percent.

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, and acceptance criteria summarized in the laboratory QAPP (Appendix D – Tables 11.3 through 11.9) 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:

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

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:

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

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.

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 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. However, QA information will be included in the monthly reports to the U.S. EPA RPM, as appropriate. 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.

APPENDIX A

FIELD SAMPLING PLAN

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

Prepared for

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Work Order No. 02687.007.003

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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 QAPP 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:

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
	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
	TSS[5]	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 Compounds.
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.

- 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 polynuclear 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 soil will be preliminary screened with a Photoionizations Detector (PID) and using visual and olfaetony observations. The soil will be considered contaminated if any of the following conditions occur:

- The PID readings are 10 units above background
- The soil is observed to be stained
- The soil is odorous

Visual and olfactory observations will be used to screen the excavated soil for contamination. Based on the results of this screening, excavated soil will be segregated into soil that appears contaminated (i.e., soil which is stained and/or is odorous) 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. Table 2-2 shows the cleanup levels for soil. 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.

Table 2-2

Analytical Parameters and Cleanup Levels (Action Limits) for Soil
Moss-American Site
Milwaukee, Wisconsin

Analytical Parameter	Action Limits, mg/kg	
	Soil Requiring Treatment and Cover	Soil Requiring Cover Only
Total CPAHs[1] (BAP Equivalent)	>78	1.9[2], 15[3]
Benzo(a)pyrene	>48	---
Fluorene	>100	---
Naphthalene	>100[4]	0.4
Benzene	>0.0055	---
Toluene	>1.5	---
Ethylbenzene	>2.9	---
Total xylenes	>4.1	---

-- Parameter does not determine whether soil requires cover.
1 Total CPAHs include benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, indeno(1,2,3-cd)pyrene.
2 Direct contact value for residential areas outside the 100-year floodplain.
3 Direct contact value for residential areas within the 100-year floodplain.
4 Site-specific interim standard.

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 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, 4oC. Lab must add preservative within 48 hours.	14 days
Treated Water	VOCs	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
	TSS	1	1-liter HDPE	Cool, 4°C	7 days

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. Figure 6-1 presents an example chain of custody form. Figure 6-2 presents an example sample label. Appendix F presents examples of completed chain of custody documents.

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:

Analysis Request/ Environmental Services Chain of Custody

Lancaster Laboratories
A division of Thermo Analytical Inc.

For Lancaster Laboratories use only

Acct. # _____ Sample # _____

Please print. Instructions on reverse side correspond with circled numbers.

(1) Client: _____ Acct. #: _____

Project Name/#: _____ PWSID #: _____

Project Manager: _____ P.O.# _____

Sampler: _____ Quote #: _____

Name of state where samples were collected: _____

(5) **Analyses Requested**

For lab use only

FSC: _____

SCR #: _____

(2) (3) (4)

Sample Identification	Date Collected	Time Collected	Grab	Composite	Matrix: Soil	Matrix: Water (☐ Potable ☐ NPDES)	Matrix: Other	Total # of Containers	Remarks	(6) Temperature of samples upon receipt at lab (if required)

(7) **Turnaround Time Requested** (TAT) (please circle): Normal Rush
(Rush TAT is subject to Lancaster Laboratories approval and surcharge.)
Date results are needed: _____
Rush results requested by (please circle): Phone Fax
Phone #: _____ Fax #: _____

(8) **Data Package Options** (please circle if requested)
QC Summary, Type I (Tier I), Type II (Tier II), Type III (NJ Red. Del.), Type IV (CLP), Type VI (Raw Data), GLP, Other
SDG Complete? Yes No
Site-specific QC required? Yes No
(If yes, indicate QC sample and submit triplicate volume.)
Internal Chain of Custody required? Yes No

(9)

Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by:	Date	Time

2425 New Holland Pike, PO Box 12425, Lancaster, PA 17605-2425 (717) 656-2300 Copies: White and yellow should accompany samples to Lancaster Laboratories. The pink copy should be retained by the client 2102 Rev 5/26/98

FIGURE 6-1

WESTON MANAGERS DESIGNERS/CONSULTANTS
Three Hawthorn Parkway
Vernon Hills, Illinois
60061

CHAIN OF CUSTODY FORM
MOSS-AMERICAN SITE
Milwaukee, Wisconsin

CLIENT

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

SAMPLE IDENTIFICATION / 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

LL #

FIGURE 6-2

WESTON MANAGERS DESIGNERS/CONSULTANTS
Three Hawthorn Parkway
Vernon Hills, Illinois
60061

SAMPLE LABEL
MOSS-AMERICAN SITE
Milwaukee, Wisconsin

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 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.

All sample containers (bottles) will be prepared according to the procedures specified in U.S. EPA's *Specifications and Guidance for Obtaining Contaminant-Free Sample Containers* (U.S. EPA, 1992). 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.

APPENDIX B

LANCASTER LABORATORIES QAPP

LABORATORY QUALITY ASSURANCE PLAN

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

August 26, 1993
Revised August 31, 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: Kathleen M. Lowen

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 method is for the determination of *n*-hexane extractable material (HEM) in waters. A 1-L sample is acidified to a pH <2 and serially extracted 3× with *n*-hexane in a separatory funnel. The extract is dried over sodium sulfate, the solvent evaporated from the extract, and the residual HEM is weighed.

Methods for the Chemical Analysis of Water and Wastes, USEPA 600/4-79-020, Method 1664.

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 higher, it is especially useful for refractory metals. Method 6010B/Method 200.7.

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.

Volatiles by GC/MS – This method determines the concentration of volatile 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 (GC) column. The GC column is temperature programmed to separate the volatile compounds which are subsequently detected and identified using mass spectrometric techniques. Method 8260B.

PAHs by GC/MS 8270				
Compound	Waters		Soils**	
	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(g,h,i)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				
Compound	Waters		Soils**	
	LOQ* (µg/L)	J-Value (µg/L)	LOQ* (µg/kg)	J-Value (µg/kg)
Benzene	1.	0.2	20	4
Toluene	1.	0.2	20	4
Ethylbenzene	1.	0.2	20	4
m,p-Xylene***	2.	0.4	40	8
O-Xylene***	1.	0.2	20	4
Chloromethane	5.	0.5	100	10
Bromomethane	5.	0.5	100	10
Dichlorodifluoromethane	2	0.2	40	4
Vinyl Chloride	1	0.2	20	4
Chloroethane	1	0.2	20	4
Trichlorofluoromethane	1	0.2	20	4
1,1-Dichloroethene	1	0.2	20	4
Methylene Chloride	1	0.2	20	4
trans-1,2-Dichloroethene	1	0.2	20	4
1,1-Dichloroethane	1	0.2	20	4
cis-1,2-Dichloroethene	1	0.2	20	4
Bromochloromethane	1	0.2	20	4
Chloroform	1	0.2	20	4
2,2-Dichloropropane	1	0.2	20	4
1,2-Dichloroethane	1	0.2	20	4
1,1,1-Trichloroethane	1	0.2	20	4
1,1-Dichloropropene	1	0.2	20	4
Carbon Tetrachloride	1	0.2	20	4
Dibromomethane	1	0.2	20	6
1,2-Dichloropropane	1	0.2	20	4
Trichloroethene	1	0.2	20	4

Volatiles by GC				
Compound	Waters		Soils**	
	LOQ* (µg/L)	J-Value (µg/L)	LOQ* (µg/kg)	J-Value (µg/kg)
Bromodichloromethane	1	0.2	20	4
cis-1,3-Dichloropropene	1	0.2	20	4
trans-1,3-Dichloropropene	1	0.2	20	4
1,1,2-Trichloroethane	1	0.2	20	4
1,3-Dichloropropane	1	0.2	20	4
Dibromochloromethane	1	0.2	20	4
Tetrachloroethene	1	0.2	20	4
1,1,1,2-Tetrachloroethane	1	0.2	20	4
Chlorobenzene	1	0.2	20	4
Bromoform	1	0.2	20	4
1,1,2,2-Tetrachloroethane	1	0.2	20	4
1,2,3-Trichloropropane	1	0.2	20	4
Bromobenzene	1	0.2	20	4
2-Chlorotoluene (o)	1	0.2	20	4
4-Chlorotoluene (p)	1	0.2	20	4
1,3-Dichlorobenzene (m)	1	0.2	20	4
1,4-Dichlorobenzene (p)	1	0.2	20	4
1,2-Dichlorobenzene (o)	1	0.2	20	4
1,2,4-Trichlorobenzene	1	0.2	20	4
Hexachlorobutadiene	1	0.2	20	4
1,2,3-Trichlorobenzene	1	0.2	20	4
Styrene	1	0.2	20	4
Isopropylbenzene (Cumene)	1	0.2	20	4
n-Propylbenzene	1	0.2	20	4
1,3,5-Trimethylbenzene	1	0.2	20	4
tert-Butylbenzene	1	0.2	20	4

Volatiles by GC				
	Waters		Soils**	
Compound	LOQ* (µg/L)	J-Value (µg/L)	LOQ* (µg/kg)	J-Value (µg/kg)
1,2,4-Trimethylbenzene	1	0.2	20	4
sec-Butylbenzene	1	0.2	20	4
p-Isopropyltoluene	1	0.2	20	4
n-Butylbenzene	1	0.2	20	4
Naphthalene	1	0.2	20	4

*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
Cyanide	0.005	0.004	0.5	0.18
Oil and Grease	5	1.4	NA	NA

*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.

Volatiles by GC/MS		
	Waters	
Compound	LOQ* (µg/L)	J-Value (µg/L)
Dichlorodifluoromethane	5.0	2.0
Chloromethane	5.0	3.0
Vinyl Chloride	5.0	2.0
Bromomethane	5.0	3.0
Chloroethane	5.0	3.0
Trichlorofluoromethane	5.0	2.0
1,1-Dichloroethene	5.0	1.0
Methylene Chloride	5.0	2.0
trans-1,2-Dichloroethene	5.0	2.0
1,1-Dichloroethane	5.0	2.0
2,2-Dichloropropane	5.0	1.0
cis-1,2-Dichloroethene	5.0	2.0
Chloroform	5.0	1.0
Bromochloromethane	5.0	1.0
1,1,1-Trichloroethane	5.0	1.0
Carbon Tetrachloride	5.0	1.0
1,1-Dichloropropene	5.0	1.0
Benzene	5.0	1.0
1,2-Dichloroethane	5.0	2.0
Trichloroethene	5.0	1.0
1,2-Dichloropropane	5.0	1.0
Dibromomethane	5.0	1.0
Bromodichloromethane	5.0	1.0
Toluene	5.0	2.0
1,1,2-Trichloroethane	5.0	2.0

Volatiles by GC/MS		
	Waters	
Compound	LOQ* (µg/L)	J-Value (µg/L)
Tetrachloroethene	5.0	1.0
1,3-Dichloropropane	5.0	1.0
Dibromochloromethane	5.0	2.0
1,2-Dibromoethane	5.0	1.0
Chlorobenzene	5.0	1.0
1,1,1,2-Tetrachloroethane	5.0	1.0
Ethylbenzene	5.0	2.0
m+*p*-Xylene **	5.0	1.0
o-Xylene **	5.0	1.0
Styrene	5.0	1.0
Bromoform	5.0	1.0
Isopropylbenzene	5.0	2.0
1,1,2,2-Tetrachloroethane	5.0	2.0
Bromobenzene	5.0	1.0
1,2,3-Trichloropropane	5.0	1.0
n-Propylbenzene	5.0	1.0
2-Chlorotoluene	5.0	1.0
1,3,5-Trimethylbenzene	5.0	1.0
4-Chlorotoluene	5.0	1.0
tert-Butylbenzene	5.0	1.0
1,2,4-Trimethylbenzene	5.0	1.0
sec-Butylbenzene	5.0	1.0
p-Isopropyltoluene	5.0	1.0
1,3-Dichlorobenzene	5.0	2.0
1,4-Dichlorobenzene	5.0	2.0
n-Butylbenzene	5.0	1.0

Volatiles by GC/MS		
	Waters	
Compound	LOQ* (µg/L)	J-Value (µg/L)
1,2-Dichlorobenzene	5.0	2.0
1,2-Dibromo-3-chloropropane	5.0	3.0
1,2,4-Trichlorobenzene	5.0	1.0
Hexachlorobutadiene	5.0	2.0
Naphthalene	5.0	1.0
1,2,3-Trichlorobenzene	5.0	1.0
cis-1,3-Dichloropropene	5.0	1.0
trans-1,3-Dichloropropene	5.0	1.0
Methyl *t*-butyl ether	5.0	2.0

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

** 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.

Inorganic Analyte List		
	Waters	
Analyte	LOQ* (mg/L)	J-Value (mg/L)
Cadmium	0.01	0.0027
Chromium	0.03	0.0054
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.

APPENDIX C

LANCASTER LABORATORIES ANALYTICAL SOPS

Waters and Wastewaters for Volatile Target Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique

Reference:

1. Method 8260A, Revision 1, SW-846, U.S. EPA, September 1994.

2. Method 8260B, Revision 2, SW-846, U.S. EPA, December 1996.

Scope:

This method is suitable for the determination of compounds listed in Tables I-A through I-L 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.

Basic Principles:

A 5-mL sample or a dilution of a sample is placed in a specially 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 on to the head of a gas chromatographic column held at the appropriate initial temperature for the column in use. The gas chromatographic column is temperature programmed to separate the volatile compounds which are subsequently detected and identified using mass spectrometric techniques.

Apparatus:

1. Gastight micro syringes - 10 µL and larger

2. 5-mL gastight syringe

3. Analytical balance, capable of accurately weighing ±0.0001 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, Tekmar LSC 3000, or equivalent meets the requirements of this method. 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.

6. GC/MS system - The HP 5970 MSD, or equivalent GC/MS system meets the requirements for this method.

7. Autosampler - OI Analytical 4551, Archon, or equivalent meets the requirements of this method.

8. Spiker units (optional) - OI Analytical Model 4551 SIM/Spiker or equivalent. One or two automated syringe spikers can be added to the OI Analytical Model 4551 autosampler to automatically introduce 10 µL of internal standard (ISTD) and/or surrogate standard solutions to the sample as it is being transferred to the sparge vessel. The Archon has a groove that can deliver 1 µL of appropriate standards.

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 and other appropriate solvents (i.e., PEG, TGDE) - For purge and trap analysis or equivalent

Standards:

1. Stock standard solutions - Stock solutions must be prepared in methanol or an equivalent solvent. Standards are prepared from ampulated and neat compounds obtained from suppliers which indicate the purity of the compound. No correction for purity is made if the purity is listed as ≥96%. Premade solutions can be used if the concentrations of the solutions are documented by the supplier. All ampulated standards are stored at -10° to -20°C until the expiration date indicated by the vendor or for 1 year if no expiration date is provided. Secondary dilution standards in methanol are stored in teflon-lined screw-cap vials, mininert vials, and volumetric flasks at -10° to -20°C.

   a. Place about 9.8 mL methanol or an equivalent solvent 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 pipette by adding 2 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 solvent in the flask to 10.0 mL. Calculate the concentration of the standard.

c. 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.

d. Replace stock standard solutions every 6 months.

e. For most of the target compounds, the stock standard solutions are purchased from a vendor as volatile organic compound (VOC) mixes. The VOC mixes are diluted together into one volumetric flask to a known concentration and named VOCXXXZZ, where XXX designates the month and ZZ designates the day that the dilution was made.

f. Also, the target compounds which are gases at room temperature are currently being purchased from a vendor.

2. Secondary dilution standards - Using the stock standard solutions prepare secondary stock solutions in methanol or an equivalent solvent containing the desired compounds. Tables I-A through I-I list the compounds analyzed under Analysis #5382, 5383, and 6291. These standards are 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," standard is poured into 40- or 15-mL Teflon-lined screw capped vials and is stored at -10° to -20°C. A designator indicating the standard, month, and day of preparation must be on the standard vials. The designator and the calculations for the working standard preparation are to be recorded in the standards logbook. Replace secondary dilution standards every 6 months.

3. Matrix spiking solution - Prepare solutions in methanol or an equivalent solvent that contain the compounds of interest at known concentrations in a similar manner as described above for the secondary dilution standards. These solutions serve as both the matrix spiking solution and the laboratory control sample solutions. Matrix spikes also serve as duplicates, therefore, two aliquots of the same sample need to be spiked for analysis with these solutions.

4. Surrogate standard spiking solution - Prepare stock standard solutions for 1,2-dichloroethane-d4, toluene-d8, 4-bromofluorobenzene, and dibromofluoromethane in methanol or an equivalent solvent. Prepare the surrogate standard spiking solution from the stock standard solutions at a concentration of 25 or 250 µg/mL in methanol or equivalent solvent. Replace surrogate standard spiking solution every 6 months.

5. Internal standards solution - Prepare a solution in methanol or an equivalent solvent containing fluorobenzene, chlorobenzene-d5, and 1,4-dichlorobenzene-d4 at a concentration of 25 or 250 µg/mL using stock standards as described in 4 above. Replace the internal standards solution every 6 months.

6. 1,4-Bromofluorobenzene (BFB) standard - Prepare a 25 µg/mL solution of BFB in methanol. Replace this solution every 6 months.

7. 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 at approximately 100°C 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, safety glasses, and gloves.

Sample Handling:

The samples should be stored in a refrigerator between 2° and 4°C. The samples should be preserved to a pH of less than 2 in order to prevent degradation of aromatic compounds that may be present in the sample. The recommended preservative is 1+1 HCl, however, samples with other preservatives can be analyzed. In addition, all samples must be analyzed within 14 days of collection.

Waste Disposal:

Neat material and stock solutions shall be collected into a lab pack upon expiration. The lab pack is delivered to Safety personnel for appropriate disposal. Expired secondary standard solutions in methanol shall be disposed of as solvent waste. Pour expired secondary standard solutions into the appropriate solvent waste collection container. Aqueous calibration standard mixes may be disposed of as nonhazardous aqueous waste due to the low concentration of volatile organics. Samples with a pH >2 are disposed of by incineration. Samples with a pH <2 are taken to storage until disposal in an acid waste container.

Personnel Training and Qualifications:

Analysts must be trained in the proper method of volatile organic sample preparation and analysis as determined by the supervisor(s). All training and education relating to volatile organic sample preparation and analysis shall be documented by each analyst in their training record.

Procedure:

1. The purge and trap device should have the trap conditioned for at least 10 minutes 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 - 40 mL/min
Purge Temperature	ambient temp.
Purge Time	11 minutes
Desorb Temperature	220°C
Desorb Time	4 minutes
Bake Temperature	180°C
Bake Time	8 minutes

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 recommended gas chromatographic operating conditions are listed in the table below:

	non-cryogenic	cryogenic
Column liquid phase	DB-624	DB-624
Carrier gas	Helium	Helium
Carrier gas flow	9-10 mL/min	9-10 mL/min
Initial temperature	35°C	10°C
Initial hold time	5 minutes	5 minutes
Temperature ramp	10°C/min.	6°C/min.
Final temperature	180°C	160°C
Final hold time	5 minutes	7 minutes

4. The recommended mass spectrometer operating conditions are listed below:

Ions	Positive
Electron energy	70 volts
Mass range	35 - 300 amu

H-P systems Scan time:

Number A/D Samples	2^2 (4)
Integration Time/Sample	50 microsec
Total Time	0.6 sec

5. Tune the GC/MS system to meet the criteria in Table II following a 2-µL injection of BFB. The chromatographic conditions must be the same as those under which the samples will be analyzed except that the temperature ramp may be increased and the initial temperature may be different. The BFB tune must be verified every 12 hours.

6. Internal standard calibration consists of analyzing standards at least five distinct levels of analyte and surrogate and producing response factors for each compound (six levels are required for 8260B if second order regression fits are used). 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 the 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. When using an Archon or OI 4551 autosampler, blanks and standards are prepared and poured into 40-mL vials with Teflon-lined septa. For each injection, 5 mL is withdrawn from the vial and transferred to the sparge vessel along with the appropriate amount of surrogate and internal standard spiking solution.

d. Purge and desorb according to Procedure 2.

e. Continue collecting GC/MS data until the end of the GC run.

f. Empty and rinse the purging chamber twice with reagent water to minimize the possibility of carryover contamination, prior to loading another sample into the vessel.

g. Prepare the calibration standards at appropriate levels. Suggested calibration levels are 4, 20, 50, 100, and 300 µg/L (a 10 µg/L level is added for 8260B when the second order regression is used).

To prevent confusion and assure proper calibration, a Theoretical Standard Concentration (TSC) sheet is completed when multiple concentrations of target analytes are included in the calibration (Figure 2). The TSC sheet contains the theoretical concentration for each analyte in the calibration at various levels.

h. Each level is analyzed as described above by substituting 5 mL of the calibration standard as the aqueous matrix. 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

Ais = Area of the characteristic ion of the appropriate internal standard

Cis = Concentration of the internal standard

Cx = Concentration of the analyte to be measured

i. Calculate the average relative response factors for all analytes. Five analytes are checked for minimum average response factors. These compounds are system performance check compounds or SPCCs. The compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. Six additional compounds are evaluated on the basis of the relative standard deviation of the RRF values (%RSD). These compounds are known as calibration check compounds (CCCs). The CCCs are 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene, and vinyl chloride. The minimum average RRF value for SPCC compounds chloromethane, 1,1-dichloroethane, and bromoform is 0.10. SPCC compounds 1,1,2,2-tetrachloroethane and

chlorobenzene have minimum average RRF values of 0.30. The %RSD for the CCC compounds must be less than 30%. Failure to meet either of the criteria for the SPCC and CCC compounds invalidates the calibration and the calibration must be redone after proper corrective action to the system is taken.

j. The %RSD for each analyte should be ≤15%. If the %RSD is ≤15%, the average RRF value for each analyte is used for method blank and sample calculations. If the %RSD of any analyte is >15, a first or second order regression fit of the calibration points may be used for method blank and sample calculations if the following criteria are met. For a linear calibration curve (first order), the correlation coefficient (r) must be ≥0.99. For a second order regression calibration curve, the coefficient of determination (r^2) must be $r^2 \geq 0.99$. Six calibration levels are required for 8260B in order to use the second order regression. For 8260B, the average RRF may still be used providing that the average of all the %RSDs is still ≤15%. Compounds which failed the %RSD but met the average of the %RSD criteria are flagged in the data package case narrative. For every 8260B initial calibration, a standard near the method detection limit must be analyzed to ensure that all compounds of interest are detectable. For both revisions, the calibration is valid for 12 hours, at which time a new tune check and a calibration check must occur prior to the analysis of additional samples.

k. The calibration check involves analyzing the 50-µg/L standard and calculating the RRF for each compound. Minimum RRF values as described under the calibration section for the SPCC compounds must be met. Also the percent drift between the standard concentration and the measured concentration for each compound in the 50 µg/L check standard are calculated as follows:

$$\% \text{ Drift} = \frac{C_1 - C_c}{C_1} \times 100$$

Where:

C_1 = Standard concentration of analyte

C_c = Measured concentration of analyte using selected quantitation method

The percent drift for the CCC compounds must be ≤20%. The internal standard areas for the calibration check are monitored against the 50-µg/L standard of the initial calibration and must be -50% to 100% of the area counts. If any criteria listed above fails, the calibration check is considered invalid. Corrective action must be taken which may include reanalysis of the calibration check, instrument maintenance, and/or recalibration. If the criteria are met, the selected quantitation method from the initial calibration are used for blank and sample calculation for the next 12-hour period.

7. A method blank is then analyzed as described above in Step 6a through e using reagent water as the aqueous matrix. The method blank is examined for interfering peaks. Any target compound peaks are calculated as described under the Calculations section of this procedure. All compounds must be less than 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 III.

8. Sample analysis

   a. Sample analysis for water and wastewater proceeds as described in Section 6a-f above.

b. For each set of 20 field samples one sample shall be analyzed in triplicate, spiking each duplicate with the matrix spiking solutions. Results of the unspiked and spiked duplicates are compared to QC windows that are statistically derived on an annual basis. A laboratory control sample, which is reagent water spiked with the matrix spiking solutions, must be analyzed when a spike recovery fails to meet the statistically derived QC windows. For 8260B, a laboratory control sample must be analyzed with every batch of up to 20 samples. If the laboratory control sample fails to meet the criteria for target compounds, corrective action must be taken. This may include reanalysis of the laboratory control sample, instrument maintenance and/or recalibration, reanalysis of samples, or data qualification and is determined on a case-by-case basis.

c. All samples must also meet the surrogate recoveries listed in Table III. If a sample surrogate recovery falls outside the criteria, the sample shall 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 target compound (Table I-A or I-B) 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 sample 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 priority pollutant 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 standard must be reanalyzed at a dilution which will bring the concentration in solution within the calibration curve. It is desirable to have the dilution within the top half of the calibration curve but not required.

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 initial calibration

Quality Assurance:

1. The GC/MS system must be tuned to meet the criteria in Table II following BFB injection. The chromatographic conditions must be the same as those under which the samples will be analyzed except that the rate of temperature ramping may be increased and the initial temperature may be different. The BFB tune must be verified every 12 hours.

2. Internal standard calibration requires analysis of a minimum of five levels of analyte concentration (six levels required for 8260B when the second order regression fit is used). Response factors for each compound are calculated. The average response factor and the relative standard deviation of the response factors determine the suitability of the average relative response factor for calculation of the analyte concentration.

   a. Five compounds are checked for minimum average response factors. These compounds are system performance check compounds or SPCCs. The compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. The minimum average RRF value for SPCC compounds chloromethane, 1,1-dichloroethane, and bromoform is 0.10. SPCC compounds

1,1,2,2-tetrachloroethane and chlorobenzene have minimum average RRF values of 0.30. If these values cannot be attained there is a problem with the purge and trap or the chromatographic system. Corrective action needs to be taken.

b. Six additional compounds are evaluated on the basis of the percent standard deviation of the RRF values (%RSD). These compounds are known as calibration check compounds (CCCs). The CCCs are 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene, and vinyl chloride. %RSD values for these compounds on the initial calibration must be less than 30%. For continuing calibration to be valid, the percent drift between the standard concentration and the measured concentration for each CCC in the 50-µg/L check standard must be within ±20%.

c. The %RSD for each analyte should be ≤15%. If the %RSD is ≤15%, the average RRF value for each analyte is used for method blank and sample calculations. If the %RSD of any analyte is >15, a first or second order regression fit of the calibration points may be used for method blank and sample calculations if the following criteria are met. For a linear calibration curve (first order), the correlation coefficient (r) must be ≥0.99. For a second order regression calibration curve, the coefficient of determination (r^2) must be $r^2 \geq 0.99$. Six calibration levels are required for 8260B in order to use the second order regression. For 8260B, the average RRF may still be used providing that the average of all the %RSDs is still <15%. Compounds which failed the %RSD but met the average of the %RSD criteria are flagged in the data package case narrative. For every 8260B initial calibration, a standard near the method detection limit must be analyzed to ensure that all compounds of interest are detectable. For both revisions, the calibration is valid for 12 hours, at which time a new tune check and a calibration check must occur prior to the analysis of additional samples.

d. For the calibration check to be valid, the percent drift between the standard concentration and the measured concentration for each CCC in the 50-µg/L check standard must be within ±20%. The SPCC compounds must meet the criteria listed above. Additionally, the internal standard areas for the calibration check are monitored against the 50-µg/L standard of the initial calibration and must be within -50% to 100% of the area counts. If the criteria listed above are not met, the calibration check is considered invalid. Corrective action must be taken which may include reanalysis of the calibration check, instrument maintenance, and/or recalibration. If the criteria are met, the selected quantitation method from the initial calibration are used for blank and sample calculation for the next 12-hour period.

3. Abundances for the internal standards must be within +100% and -50% of the abundances of the internal standards of the current calibration check standard (50 µg/L). The retention time for each internal standard must be within 30 seconds of the value for the last calibration check. Values outside these limits indicate a change in system performance or a matrix effect. Samples with internal standard abundances outside of limits are to be reanalyzed. If the reanalysis is within limits, the data from the reanalysis is reported. If the reanalysis confirms the first analysis the sample matrix is interfering and the data from the first analysis should be reported.

4. Surrogate recoveries are calculated for each sample and blank analyzed. Surrogate recovery limits are listed in Table III. Statistical limits are generated and compared to the method limits. The tighter of the two limits sets are used for data evaluation. 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.

5. The method blank must meet all the above criteria for internal standard recoveries and surrogate recoveries. In addition, the method blank may not contain any target compound above the quantitation limit. All method blanks must meet these criteria otherwise the system is considered out of control and corrective action must be taken.

6. The matrix spike and matrix spike duplicate pair are analyzed for each set of 20 field samples. The recoveries and the percent differences of the recoveries for each spiked compound are calculated and compared to the value from the statistical results of multiple analysis of the spiked samples. The results of the matrix/matrix spike duplicate (MS/MSD) must be within the statistically derived windows. For 8260A, MS/MSD results are outside the statistical windows, analysis of a laboratory control sample is required. For 8260B samples, a laboratory control sample is required with each batch of up to 20 samples. The laboratory control sample is a method blank spiked with each of the matrix spiking solutions. The laboratory control sample must yield recoveries for all analytes within the statistically derived windows. If the laboratory control sample fails to meet the criteria for target compounds, corrective action must be taken. This may include reanalysis of the laboratory control sample, instrument maintenance and/or recalibration, reanalysis of samples, or data qualification and is determined on a case-by-case basis.

7. All data is reviewed by a data review specialist prior to release of the data from the analytical laboratory with respect to quality assurance and data interpretation. 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 can not be corrected will be referred to the group leader for further action.

Additional Analyses:

Compounds other than those listed in Tables I-A through I-L may be analyzed. Due to poor purging efficiency or poor chromatography, some nonmethod analytes require calibration at higher levels and/or higher practical quantitation limits (PQLs). Any additional compounds should be added to the theoretical standard concentration sheet. Standards containing additional analytes should be prepared as described in the Reagents section of this document. Both secondary stock solutions and matrix spike solutions should be prepared for use in analyzing additional compounds.

The analysis of MTBE by 2306 and 2309 contains some differences to the above method. The requirements for calibration and continuing calibration differ from the above method in that the CCC compounds are not required to meet any %RSD or %drift requirements. SPCC compounds must still meet the minimum RRF. The percent drift for methyl t-butyl ether, in continuing calibrations, must be ≤20%. Dibromofluoromethane is the only surrogate compound that is required to have recoveries within its acceptance window. The other three surrogate compounds may be present but are not required to fall within their acceptance windows.

Revision Log:

Initiated Date: 05/02/94

Ver. #	Effective Date	Change
00	11/06/95	Previous Issue
01	01/05/98	Major changes are as follows: • Updated entire method to comply with 8260B
02	04/14/98	Major changes are as follows: • 8260B updates • Added analysis numbers

Ver. #	Effective Date	Change
03	10/02/98	Major changes are as follows: • Added section for analysis of methyl *t*-butyl ether only • Added additional analysis numbers that had recently been taken out (2300, 2301, 2302, 2302, 2306, 2309) • Added additional approval sign off since this procedure will be shared between Departments 21 and 34
04	MAR 2 5 1999	Major changes are as follows: • Update information on correlation coefficient and coefficient of determination • Update requirement on checking areas in calibration check standards • Update LCS requirement

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Prepared by: Debbie Turner Date: 3/17/99

Approved by: Diane B. Leach Date: 3/17/99

Approved by: Thomas C [illegible] Date: 3/17/99

Approved by: Dorothy M Love Date: 3/22/99

Table I-A
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #5382, 5383

Volatiles	Practical Quantitation Limits Water/Wastewater (µg/L)
Benzene	5
Bromobenzene	5
Bromochloromethane	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	5
n-Butylbenzene	5
sec-Butylbenzene	5
tert-Butylbenzene	5
Carbon tetrachloride	5
Chlorobenzene	5
Chlorodibromomethane	5
Chloroethane	5
Chloroform	5
Chloromethane	5
2-Chlorotoluene	5
4-Chlorotoluene	5
1,2-Dibromo-3-chloropropane	5
1,2-Dibromoethane	5
Dibromomethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
Dichlorodifluoromethane	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
1,3-Dichloropropane	5
2,2-Dichloropropane	5
1,1-Dichloropropene	5
Ethylbenzene	5
Hexachlorobutadiene	5
Isopropylbenzene	5
p-Isopropyltoluene	5
Methylene chloride	5
Naphthalene	5

Volatiles	Practical Quantitation Limits Water/Wastewater (µg/L)
n-Propylbenzene	5
Styrene	5
1,1,1,2-Tetrachloroethane	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,2,3-Trichlorobenzene	5
1,2,4-Trichlorobenzene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	5
1,2,3-Trichloropropane	5
1,2,4-Trimethylbenzene	5
1,3,5-Trimethylbenzene	5
Vinyl chloride	5
m+*p*-xylene	5
o-xylene	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-B
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #6291

Volatiles	Practical Quantitation Limits Water/Wastewater (µg/L)
Chloromethane	5
Vinyl Chloride	5
Bromomethane	5
Chloroethane	5
1,1-Dichloroethene	5
Acetone	5
Carbon disulfide	5
Methylene chloride	20
1,1-Dichloroethane	5
trans-1,2-Dichloroethene	5
cis-1,2-Dichloroethene	5
2-Butanone	10
Chloroform	5
1,2-Dichloroethane	5
1,1,1-Trichloroethane	5
Carbon tetrachloride	5
Benzene	5
Trichloroethene	5
1,2-Dichloropropane	5
Bromodichloromethane	5
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5
1,1,2-Trichloroethane	5
Dibromochloromethane	5
Bromoform	5
4-Methyl-2-pentanone	10
Toluene	5
Tetrachloroethene	5
2-Hexanone	10
Chlorobenzene	5
Ethylbenzene	5
Xylene (total)	5
Styrene	5
1,1,2,2-Tetrachloroethane	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-C
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #6872

Volatiles	Practical Quantitation Limits Water/Wastewater (µg/L)
Chloromethane	5
Vinyl Chloride	5
Bromomethane	5
Chloroethane	5
Trichlorofluoromethane	5
1,1-Dichloroethene	5
Acetone	20
Methyl Iodide	5
Carbon Disulfide	5
Methylene Chloride	5
Acrylonitrile	50
trans-1,2,-Dichloroethene	5
1,1-Dichloroethane	5
Vinyl Acetate	10
cis-1,2-Dichloroethene	5
2-Butanone	10
Bromochloromethane	5
Chloroform	5
1,1,1-Trichloroethane	5
Carbon Tetrachloride	5
Benzene	5
1,2-Dichloroethane	5
Trichloroethene	5
1,2-Dichloropropane	5
Dibromomethane	5
Bromodichloromethane	5
cis-1,3-Dichloropropene	5
4-Methyl-2-pentanone	5
Toluene	5
trans-1,3-Dichloropropene	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-D
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #6873

Volatiles	Practical Quantitation Limits Water/Wastewater (µg/L)
1,1,2-Trichloroethane	5
Tetrachloroethene	5
2-Hexanone	10
Dibromochloromethane	5
1,2-Dibromoethane	5
Chlorobenzene	5
1,1,1,2-Tetrachloroethane	5
Ethylbenzene	5
Xylene (total)	5
Styrene	5
Bromoform	5
1,1,2,2-Tetrachloroethane	5
1,2,3-Trichloropropane	5
trans-1,4-Dichloro-2-butene	50
1,4-Dichlorobenzene	5
1,2-Dichlorobenzene	5
1,2-Dibromo-3-chloropropane	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-E
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #6886

Volatiles	Practical Quantitation Limits Water/Wastewater (µg/L)
Dichlorofluoromethane	5
Chloromethane	5
Vinyl Chloride	5
Bromomethane	5
Chloroethane	5
Trichlorofluoromethane	5
Acrolein	100
1,1-Dichloroethene	5
Acetone	20
Methyl Iodide	5
Carbon Disulfide	5
Acetonitrile	100
Allyl Chloride	5
Methylene Chloride	5
Acrylonitrile	50
trans-1,2-Dichloroethene	5
1,1-Dichloroethane	5
Vinyl Acetate	10
2-Chloro-1,3-butadiene	5
cis-1,2-Dichloroethene	5
2-Butanone	10
Propionitrile	100
Methacrylonitrile	50
Chloroform	5
1,1,1-Trichloroethane	5
Carbon Tetrachloride	5
Isobutyl Alcohol	250
Benzene	5
1,2-Dichloroethane	5
Trichloroethene	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-F
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #6887

Volatiles	Practical Quantitation Limits Water/Wastewater (µg/L)
1,2-Dichloropropane	5
Methyl Methacrylate	5
Dibromomethane	5
1,4-Dioxane	250
Bromodichloromethane	5
cis-1,3-Dichloropropene	5
4-Methyl-2-pentanone	10
Toluene	5
trans-1,3-Dichloropropene	5
Ethyl Methacrylate	5
1,1,2-Trichloroethane	5
Tetrachloroethene	5
2-Hexanone	10
Dibromochloromethane	5
1,2-Dibromoethane	5
Chlorobenzene	5
1,1,1,2-Tetrachloroethane	5
Ethylbenzene	5
Xylene (total)	5
Styrene	5
Bromoform	5
1,1,2,2-Tetrachloroethane	5
1,2,3-Trichloropropane	5
trans-1,4-Dichloro-2-butene	50
Pentachloroethane	10
1,2-Dibromo-3-chloropropane	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-G
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #5243

Volatiles	Practical Quantitation Limits Water/Wastewater (µg/L)
Dichlorodifluoromethane	5
Chloromethane	5
Vinyl Chloride	5
Bromomethane	5
Chloroethane	5
Trichlorofluoromethane	5
1,1-Dichloroethene	5
Acetone	20
Methyl Iodide	5
Carbon Disulfide	5
Allyl Chloride	5
Methylene Chloride	5
Acrylonitrile	50
trans-1,2,-Dichloroethene	5
1,1-Dichloroethane	5
Vinyl Acetate	10
cis-1,2-Dichloroethene	5
2-Butanone	10
Bromochloromethane	5
Chloroform	5
1,1,1-Trichloroethane	5
Carbon Tetrachloride	5
Benzene	5
1,2-Dichloroethane	5
Trichloroethene	5
1,2-Dichloropropane	5
Dibromomethane	5
Bromodichloromethane	5
cis-1,3-Dichloropropene	5
4-Methyl-2-pentanone	10
Toluene	5
trans-1,3-Dichloropropene	5
1,1,2-Trichloroethane	5
Tetrachloroethene	5
2-Hexanone	10
Dibromochloromethane	5
1,2-Dibromoethane	5
Chlorobenzene	5
1,1,1,2-Tetrachloroethane	5
Ethylbenzene	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-H
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #5244

Volatiles	Practical Quantitation Limits Water/Wastewater (µg/L)
Xylene (total)	5
Styrene	5
Bromoform	5
1,1,2,2-Tetrachloroethane	5
1,2,3-Trichloropropane	5
trans-1,4-Dichloro-2-butene	50
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
1,2-Dichlorobenzene	5
1,2-Dibromo-3-chloropropane	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-I
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #7582

Volatiles	Practical Quantitation Limits Water/Wastewater (µg/L)
Chloromethane	5
Vinyl Chloride	5
Bromomethane	5
Chloroethane	5
Trichlorofluoromethane	5
Acrolein	100
1,1-Dichloroethene	5
Methylene Chloride	5
Acrylonitrile	50
trans-1,2-Dichloroethene	5
1,1-Dichloroethane	5
cis-1,2-Dichloroethene	5
Chloroform	5
1,1,1-Trichloroethane	5
Carbon Tetrachloride	5
Benzene	5
1,2-Dichloroethane	5
Trichloroethene	5
1,2-Dichloropropane	5
Bromodichloromethane	5
2-Chloroethyl vinyl ether	10
cis-1,3-Dichloropropene	5
Toluene	5
trans-1,3-Dichloropropene	5
1,1,2-Trichloroethane	5
Tetrachloroethene	5
Dibromochloromethane	5
Chlorobenzene	5
Ethylbenzene	5
Xylene (total)	5
Bromoform	5
1,1,2,2-Tetrachloroethane	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-J
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #2306 and 2309

Volatiles	Practical Quantitation Limits Water/Wastewater (ug/L)
Methyl *t*-Butyl Ether	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-K
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #2300 and 2301

Volatiles	Practical Quantitation Limits Water/Wastewater (µg/L)
Methyl *t*-Butyl Ether	5
Benzene	5
Toluene	5
Ethylbenzene	5
Total Xylenes	5
Isopropylbenzene	5
Napthalene	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-L
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #2302 and 2303

Volatiles	Practical Quantitation Limits Water/Wastewater (µg/L)
Methyl *t*-Butyl Ether	5
Benzene	5
Toluene	5
Ethylbenzene	5
Total Xylenes	5
Isopropylbenzene	5
Napthalene	5
1,2-Dichloroethane	5
1,2-Dibromoethane	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table II
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 III
Surrogate Spike Recovery Limits for Water and Wastewater Samples

Surrogate Compound	Water/Wastewater
4-Bromofluorobenzene	86-115
Dibromofluoromethane	86-118
Toluene-d8	88-110
1,2-Dichloroethane-d4	80-120

Figure 1

Tekmar®

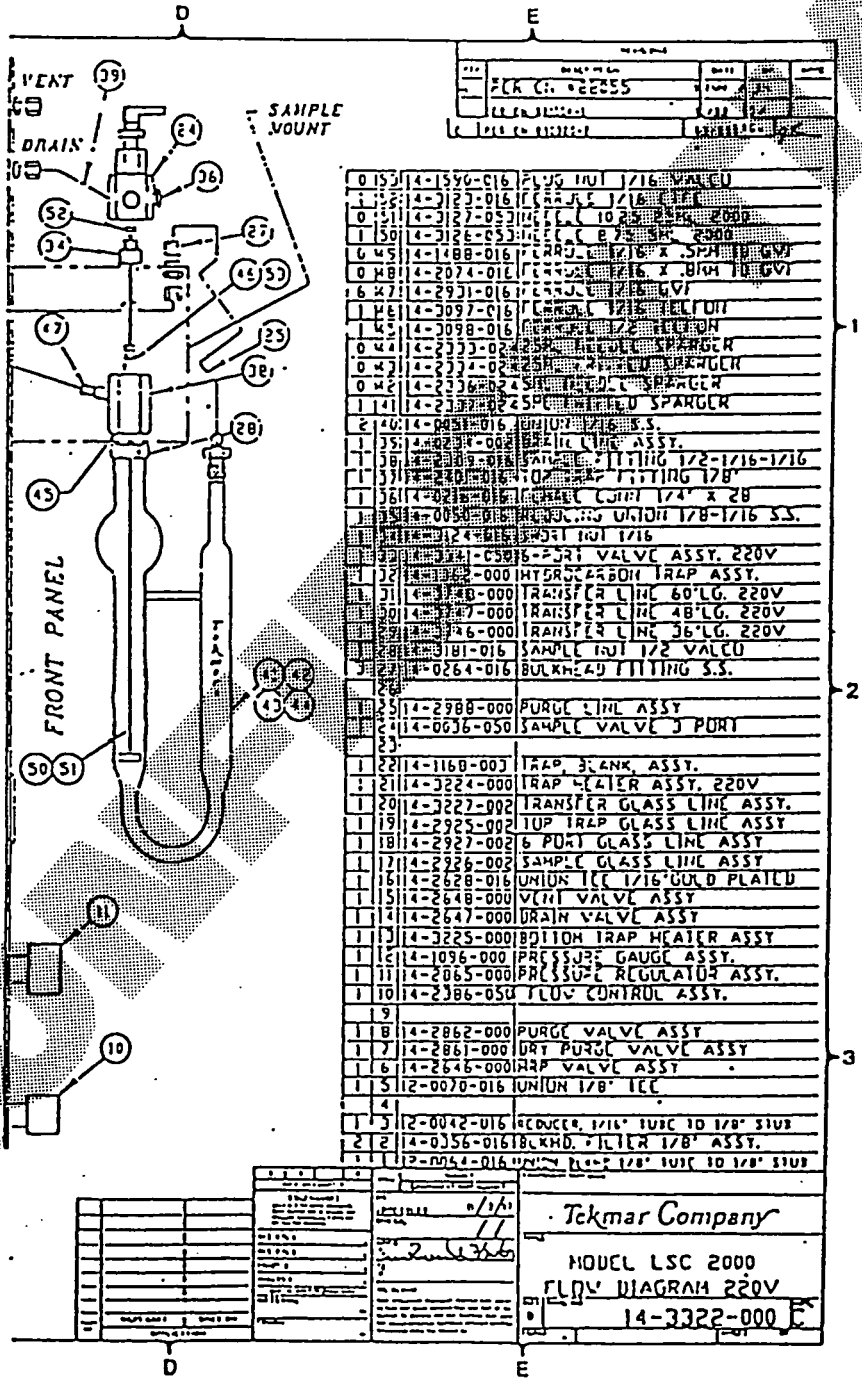

Figure 2

THEORETICAL STANDARD CONCENTRATIONS
INITIAL CALIBRATION
PURCHASED STANDARDS
HP CAPILLARY COLUMN
EPA SW846 METHOD 8260B

DATE:
INSTRUMENT:

VOA1 = 1 to 10 dilution of CS#1, CS#2 and CS#4
VOA2 = 1 to 10 dilution of CS#2
VOA3 = 1 to 10 dilution of CS#3

stock mix name	VOA1	VOA3	VOA2	VOA4 BCM	Restek GASES (2000 ppm) Lt#	FLASK ml
300 ppb std	37.5 ul	15 ul		7.5 ul	3.75 ul	25
200 ppb std	25 ul	10 ul		5 ul	2.5 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	20 ul	8 ul	20 ul	4 ul	2.0 ul	200@
10 ppb std	@Dilute 2.5 ml of 200 ml flask 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	200 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb
Benzene	CS #1	2000	300	200	100	50	20	10	4
Bromobenzene		2000	300	200	100	50	20	10	4
Bromodichloromethane		2000	300	200	100	50	20	10	4
Bromoform		2000	300	200	100	50	20	10	4
n-Butylbenzene		2000	300	200	100	50	20	10	4
sec-Butylbenzene		2000	300	200	100	50	20	10	4
tert-Butylbenzene		2000	300	200	100	50	20	10	4
Carbon tetrachloride		2000	300	200	100	50	20	10	4
Chlorobenzene		2000	300	200	100	50	20	10	4
Chloroform		2000	300	200	100	50	20	10	4
2-Chlorotoluene		2000	300	200	100	50	20	10	4
4-Chlorotoluene		2000	300	200	100	50	20	10	4
Dibromochloromethane		2000	300	200	100	50	20	10	4
1,2-Dibromo-3-chloropropane		2000	300	200	100	50	20	10	4
1,2-Dibromoethane (EDB)		2000	300	200	100	50	20	10	4
Dibromomethane		2000	300	200	100	50	20	10	4
1,2-Dichlorobenzene		2000	300	200	100	50	20	10	4
1,3-Dichlorobenzene		2000	300	200	100	50	20	10	4
1,4-Dichlorobenzene		2000	300	200	100	50	20	10	4
1,1-Dichloroethane		2000	300	200	100	50	20	10	4
1,2-Dichloroethane		2000	300	200	100	50	20	10	4
1,1-Dichloroethene		2000	300	200	100	50	20	10	4
cis-1,2-Dichloroethene		2000	300	200	100	50	20	10	4
trans,1,2-Dichloroethene		2000	300	200	100	50	20	10	4

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Figure 2 (Continued)

EPA SW846 METHOD 8260B
INITIAL CALIBRATION

compound name	std mix	stock ppm	300 ppb	200 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb
1,2-Dichloropropane	CS	2000	300	200	100	50	20	10	4
1,3-Dichloropropane	#1	2000	300	200	100	50	20	10	4
2,2-Dichloropropane		2000	300	200	100	50	20	10	4
1,1-Dichloropropene		2000	300	200	100	50	20	10	4
cis-1,3-Dichloropropene		2000	300	200	100	50	20	10	4
trans-1,3-Dichloropropene		2000	300	200	100	50	20	10	4
Ethylbenzene		2000	300	200	100	50	20	10	4
Hexachlorobutadiene		2000	300	200	100	50	20	10	4
Isopropylbenzene (Cumene)		2000	300	200	100	50	20	10	4
p-Isopropyltoluene		2000	300	200	100	50	20	10	4
Methylene Chloride		2000	300	200	100	50	20	10	4
Naphthalene		2000	300	200	100	50	20	10	4
n-Propylbenzene		2000	300	200	100	50	20	10	4
Styrene		2000	300	200	100	50	20	10	4
1,1,1,2-Tetrachloroethane		2000	300	200	100	50	20	10	4
1,1,2,2-Tetrachloroethane		2000	300	200	100	50	20	10	4
Tetrachloroethene		2000	300	200	100	50	20	10	4
Toluene		2000	300	200	100	50	20	10	4
1,2,3-Trichlorobenzene		2000	300	200	100	50	20	10	4
1,2,4-Trichlorobenzene		2000	300	200	100	50	20	10	4
1,1,1-Trichloroethane		2000	300	200	100	50	20	10	4
1,1,2-Trichloroethane		2000	300	200	100	50	20	10	4
Trichloroethene		2000	300	200	100	50	20	10	4
1,2,3-Trichloropropane		2000	300	200	100	50	20	10	4
1,2,4-Trimethylbenzene		2000	300	200	100	50	20	10	4
1,3,5-Trimethylbenzene		2000	300	200	100	50	20	10	4
m-Xylene		2000	300	200	100	50	20	10	4
o-Xylene		2000	300	200	100	50	20	10	4
p-Xylene		2000	300	200	100	50	20	10	4
Bromomethane	GAS	2000	300	200	100	50	20	10	4
Chloroethane	MIX	2000	300	200	100	50	20	10	4
Chloromethane		2000	300	200	100	50	20	10	4
Dichlorodifluoromethane		2000	300	200	100	50	20	10	4
Trichlorofluoromethane		2000	300	200	100	50	20	10	4
Vinyl Chloride		2000	300	200	100	50	20	10	4
Methacrylonitrile	CS	5000	750	500	250	125	100	50	40
Propionitrile	#2	10000	1500	1000	500	250	200	100	80
trans-1,4-Dichloro-2-Buten		5000	750	500	250	125	100	50	40
t-Butyl Alcohol		10000	1500	1000	500	250	200	100	80
2-Propanol		10000	1500	1000	500	250	200	100	80
Isobutyl Alcohol		25000	3750	2500	1250	625	500	250	200
n-Butanol		25000	3750	2500	1250	625	500	250	200
1-Propanol		25000	3750	2500	1250	625	500	250	200
1,4-Dioxane		25000	3750	2500	1250	625	500	250	200
Cyclohexanone		25000	3750	2500	1250	625	500	250	200
Monochloroacetone		25000	3750	2500	1250	625	500	250	200
2-Butanone	CS	10000	600	400	200	100	40	20	8
2-Hexanone	#3	10000	600	400	200	100	40	20	8
4-Methyl-2-Pentanone		10000	600	400	200	100	40	20	8
Acetone		10000	600	400	200	100	40	20	8
Acrolein		50000	3000	2000	1000	500	200	100	40
Acrylonitrile		50000	3000	2000	1000	500	200	100	40
2-Chloroethyl-vinyl-ether		10000	600	400	200	100	40	20	8
2-Nitropropane		10000	600	400	200	100	40	20	8

Figure 2 (Continued)

EPA SW846 METHOD 8260B
INITIAL CALIBRATION

compound name	std mix	stock ppm	300 ppb	200 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb
Methyl-t-butyl Ether	CS	2000	300	200	100	50	20	10	4
t-Butyl Formate	#4	2000	300	200	100	50	20	10	4
Ethyl Methacrylate		2000	300	200	100	50	20	10	4
Methyl Methacrylate		2000	300	200	100	50	20	10	4
Ethyl Ether		2000	300	200	100	50	20	10	4
Freon 113		2000	300	200	100	50	20	10	4
Hexane		2000	300	200	100	50	20	10	4
Heptane		2000	300	200	100	50	20	10	4
Cyclohexane		2000	300	200	100	50	20	10	4
Benzyl Chloride		2000	300	200	100	50	20	10	4
Isopropyl Acetate		2000	300	200	100	50	20	10	4
Methyl Iodide		2000	300	200	100	50	20	10	4
Carbon Disulfide		2000	300	200	100	50	20	10	4
Vinyl Acetate		2000	300	200	100	50	20	10	4
n-Propyl Acetate		2000	300	200	100	50	20	10	4
Tetrahydrofuran		2000	300	200	100	50	20	10	4
2-Chloro-1,3-butadiene		2000	300	200	100	50	20	10	4
Pentachloroethane	VOA4	1000	300	200	100	50	20	10	4
Allyl Chloride		1000	300	200	100	50	20	10	4
Bromochloromethane	BCM	1000	300	200	100	50	20	10	4

ppb of analytical standard = stock ppm x ul stock / flask ml

Calibration Standard Conc. (ppb)	300	200	100	50	20	10	4
Surrogate Spiking Volume (ul)*	6%	4%	2%	10	4	2	0.8

*Spiked directly into 5 ml syringe
% Using I826SS

ANALYST_____

DATE_____

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Low-Level Extraction Procedure for the Determination of Semivolatile PAHs in a Solid Matrix

Reference:

1. Method 3550B, SW-846.

2. *Sonicator Ultrasonic Processor and Cell Disruptor Operations Manual*, Sound Heat Systems, Inc., 1985.

Scope:

This procedure is applicable for the extraction of PAHs at low ppm levels from soils or solid wastes. Conditions such as high levels of organic compounds may interfere with normal detection limits.

Basic Principles:

A portion of sample to be analyzed is placed in a beaker. Anhydrous sodium sulfate is added to absorb any water which may be present. Surrogate standards are added to each sample to monitor recovery. (See SOP-EX-001, "Semivolatile Spiking and Calibration Standards," for preparation.) An aliquot of solvent is then added to the sample. The sample is subjected to sonic disruption to disperse the soil and force solvent contact. The organic compounds present in the soil dissolve in the solvent which is then removed. The sample is extracted two more times with fresh solvent, the solvent fractions are combined and concentrated to below 1 mL. The extract is brought to 1.0 mL and bottled in an amber autosampler vial. It is stored in the freezer until analysis.

Holding Times:

Samples should be extracted within 14 days of collection. All samples should be stored at 2° to 4°C prior to extraction.

Apparatus:

1. Sonic probe apparatus for extracting organic components from a soil matrix
2. Kuderna-Danish assembly with appropriate ampule for concentrating the solvent used during concentration
3. Water bath or S-Evap
4. Filter paper - Fisher brand glass fiber filter circles or equivalent

Reagents:

1. Methylene chloride - Pesticide grade or equivalent
2. Acetone - GC[2] or equivalent
3. Sodium sulfate - Reagent grade or equivalent. Bake at 400°C for 4 hours in a shallow pan prior to use to remove organic contaminants. Store in a glass jar for up to 1 year after baking.

Preparation of Glassware:

See SOP-OE-001, "Glassware Cleaning for Organic Extractions."

Safety Precautions:

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available such as fume hoods, lab coats, safety glasses, and gloves.

Since the extracts are concentrated on a steam bath, caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or must be disposed of in the designated containers. These will then be transferred to the lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications:

All personnel performing these techniques should have performed a solvent concentration quad study that yielded acceptable recoveries for semivolatile LCS compounds. Personnel should spend several days working with an experienced preparation technician who has demonstrated their proficiency of the extraction. Also, several batches of semivolatile samples should be performed under the direct observation of another experienced preparation technician to assure the trainee is capable of independent preparation.

Procedure:

1. Weigh out 30 ± .04 g of sample into a 250-mL stainless steel beaker. Record in the extraction log the initial weight to the nearest 0.1 g and any comments about the sample.

2. Add at least 60 g of anhydrous powdered sodium sulfate and mix well. Additional sodium sulfate may be added to obtain a free-flowing mixture.

3. Add 1.0 mL of semi PAH surrogate standard into the beaker. Also add 1.0 mL of semi PAH matrix spiking solution to the matrix spike, matrix spike duplicate, and the laboratory control sample. If the sample requires any compounds in addition to the PAH semivolatile compounds, 1.0 mL of a 100-ppm spike of this compound is added at this time (see SOP-EX-001).

4. Using a solvent pump add 100 mL of 50% acetone in methylene chloride.

5. Set up the sonic probe as described in the manual. (See MC-OE-002, "Ultrasonic Processor Maintenance and Tuning.")

6. Immerse the tip of the sonic probe approximately 1 to 2 cm below the surface of the liquid in the beaker containing the sample and above the sediment layer.

7. Disrupt the sample using a medium tip at full output of 10 and a process time/timer of 1:30.

   NOTE: This is equivalent to 3 minutes, 50% duty cycle as described in the EPA method.

8. Remove the probe from the sample and decant the liquid from the beaker. Filter through Fisher brand G 6 glass fiber filter circles using vacuum filtration.

   NOTE: Be sure to turn the vacuum off immediately after solvent is no longer observed dripping from the funnel.

9. Add 100 mL of fresh solvent to the sample and repeat steps 6 through 8.

10. Add 100 mL of fresh solvent to the sample and repeat steps 6 through 8. Pour the liquid and solids from the beaker onto the filter paper. Rinse the beaker and filter paper with approximately 30 mL of 50% acetone in methylene chloride.

    Before placing the probe into another sample, wipe the probe using a paper towel wetted with deionized water to remove any soil present from the previous sample. Rinse the probe with acetone to remove water.

11. Pour the collected extract into a Kuderna-Danish containing a Teflon boiling chip. Place a 3-ball Snyder column on the setup, wet the column with methylene chloride, and concentrate over a steam bath/S-Evap which is at 85° to 95°C to about 1 mL. Allow the sample to cool 10 minutes. Approximately 3 mL will condense into the ampule during this time.

    This steam bath temperature ensures concentration in a reasonable length of time.

    NOTE: To reduce burnout of heating elements, do not allow the water level in the steam bath/S-Evap to go below 5 cm from the top of the bath.

12. Attach the ampule of the K-D to a micro-Snyder column and concentrate the extract to below 1 mL. Allow the sample to cool.

13. Bring to a final volume of 1.0 mL with methylene chloride. The final volume is determined by placing the extract into an amber autosampler vial and comparing the level in the vial to a vial containing the targeted final volume. Methylene chloride is added to the extract until exactly the same level is in both vials. If too much solvent is added to the sample vial, remove the extract from the vial and concentrate it to slightly less than the targeted final volume and rebottle. Cap the vial and store in the freezer until analysis. Record the final volume in the extraction log.

Calculations:

See analysis method.

Statistical Information:

See analysis method.

Quality Assurance:

For each batch of samples extracted, a blank, a laboratory control sample (LCS) (sodium sulfate blank spiked with all compounds to be determined carried through the entire procedure) a matrix spike, and matrix spike duplicate must be extracted. If insufficient volume of sample is available for MS/MSD, then an LCSD must be prepared instead. A batch is defined as the samples to be extracted on any given day but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Revision Log:

Initiated Date: 10/17/96

Ver. #	Effective Date	Change
00	11/04/96	Previous Issue
01	09/15/97	Major changes as follows: • Added holding times. • Removed GPC cleanup procedure. • Added statement of following specific client and state requirements to Quality Assurance.

Ver. #	Effective Date	Change
02	01/29/98	Major changes: • Updated method reference
03	APR 0 5 1999	Major changes are as follows: • Reagents - Added length of time Na_2SO_4 can be stored after baking • Update glassware cleaning • Procedure - Clarified final volume determination • Quality Assurance - Batch per day

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Prepared by: _____ (092) Date: 3-11-99

Approved by: _____ Date: 5.12.98

Approved by: _____ Date: 5/25/99

Approved by: _____ Date: 3/30/99

Analysis #3338
Revision 02
Supersedes Date: 04/09/97
Effective Date: JAN 2 1 1998
Page 1 of 8

Extraction Procedure for the Determination of Polynuclear Aromatic Hydrocarbons (PAH) in a Solid Matrix

Reference:

1. Method 3550B, USEPA SW-846.
2. Method 8310, USEPA SW-846.
3. *Sonicator Ultrasonic Processor and Cell Disruptor Operations Manual*, Sound Heat Systems, Inc., 1985.

Scope:

This method is suitable for the extraction of PAH compounds in soils or solid wastes. Conditions such as high levels of organic compounds and/or extreme alkalinity or acidity in the sample may interfere with normal detection limits. Samples that are highly contaminated should be extracted and analyzed by USEPA SW-846, Method 8270, since this method is designed for trace level analytes.

Basic Principles:

A portion of sample to be analyzed is placed in a beaker. Anhydrous sodium sulfate is added to absorb any water which may be present. Surrogate standards are added to each sample to monitor recovery. See SOP-PP-021, "Standards Preparation, Coding, and Storage." An aliquot of solvent is then added to the sample. The sample is subjected to sonic disruption to disperse the soil and force solvent contact. The organic compounds present in the soil dissolve in the solvent which is then removed.

The sample is extracted two more times with fresh solvent, the solvent fractions are combined and concentrated to approximately 2 mL then changed over with acetonitrile. The extract is stored in a clear screw-top auto sampler vial in the freezer until analysis.

Personnel Training and Qualifications:

All personnel performing these techniques should have performed a solvent concentration quad study that yielded acceptable recoveries for PAH pesticide spike compounds. Personnel should spend several days working with an experienced preparation technician who has demonstrated their proficiency of the extraction. Also, several batches of PAH pesticide soil samples should be performed under the direct observation of another experienced preparation technician to assure the trainee is capable of independent preparation.

Holding Time:

Samples should be extracted within 14 days of collection. All samples should be stored at 2° to 4°C prior to extraction.

Apparatus:

1. Sonic probe apparatus for extracting organic components from a soil matrix
2. Kuderna-Danish assembly with jacketed ampule for concentrating the solvent used during concentration
3. Water bath or S-Evap
4. Filter paper - Whatman #3 or equivalent
5. N-Evap with nitrogen supply
6. 3-mL disposable syringe

7. Glass boiling beads

8. 0.45-µm Teflon filters

9. 2-mL volumetric flask

Reagents:

1. Methylene chloride - Pesticide grade or equivalent

2. Acetone - Pesticide grade or equivalent

3. Acetonitrile (ACN) - HPLC grade

4. Sodium sulfate - Reagent grade or equivalent. Bake at 400°C for 4 hours in a shallow pan to remove contaminants. Store in a glass jar for up to 1 year after baking.

Preparation of Glassware:

All glassware is rinsed with tap water and soaked in dilute Contrad 70 for at least 2 hours. The glassware is then rinsed with tap water followed by deionized water, and baked in a drying oven for 1 hour or until dry. See SOP-OE-001, "Glassware Cleaning for Organic Extractions."

Safety Precautions:

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Since the extracts are concentrated on a steam bath, caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or must be disposed of in the designated containers. These will then be transferred to the lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) may be disposed of in the normal solid waste collection containers.

Procedure:

1. Weigh out 30 ± .04 g of sample into a 250-mL stainless steel beaker. Record the Lancaster Laboratories number, the initial weight to the nearest 0.1 g, and any comments about the sample in the extraction log.

2. Add at least 60 g of anhydrous powdered sodium sulfate and mix well. Additional sodium sulfate may be added to obtain a free-flowing mixture.

3. Add surrogate standards and spiking solutions to the sample in the beaker.

   a. Surrogates: Add 0.5 mL PAH pest surrogate to all samples, blanks, and spikes.

   b. Spiking solutions: Add 0.5 mL of PAH pest. spiking solution to the laboratory control sample (LCS), LCSD if applicable, matrix spike, and matrix spike duplicate samples.

   NOTE: This may change to accommodate client-specific requirements.

   If a sample requires any special compounds in addition to the standard list, an appropriate spike containing the compounds is also added at this time.

4. Using a solvent pump add 100 mL of 50% acetone in methylene chloride.

5. Set up the sonic probe as described in the manual. See MC-OE-002, "Ultrasonic Processor Maintenance and Tuning."

6. Immerse the tip of the sonic probe approximately 1 to 2 cm below the surface of the liquid in the beaker containing the sample and above the sediment layer.

7. Disrupt the sample using a medium tip at full output of ten and a process time/timer of 1:30.

   NOTE: This is equivalent to a time of 3 minutes at a 50% duty cycle.

8. Remove the probe from the sample and decant the liquid from the beaker. Filter through Whatman #3 filter paper using vacuum filtration.

   NOTE: Be sure to turn the vacuum off immediately after solvent is no longer observed dripping from the funnel.

9. Add 100 mL of fresh solvent to the sample and repeat steps 6 through 8.

10. Add 100 mL of fresh solvent to the sample and repeat steps 6 through 8. Pour the liquid and solids from the beaker onto the filter paper. Rinse the beaker and filter paper with approximately 30 mL of 50% acetone in methylene chloride.

    Before placing the probe into another sample, wipe the probe using a paper towel wetted with deionized water to remove any soil present from the previous sample. Rinse the probe with acetone to remove water.

11. Pour the collected extract into a Kuderna-Danish containing a glass boiling chip. Place a 3-ball Snyder column on the set-up, wet the column with methylene chloride and concentrate to approximately 5 mL over a steam

bath/S-Evap which is at 90° to 95°C. **Do not allow the ampule to go dry.** Do not go below 2 mL since loss of volatile analytes may occur. Allow the sample to cool 10 minutes.

This steam bath temperature ensures concentration in a reasonable length of time.

NOTE: To reduce burn out of heating elements, do not allow the water level in the steam bath/S-Evap to go below 5 cm from the top of the bath.

12. Attach the ampule of the KD to a micro-snyder column, and concentrate the extract to approximately 0.5 mL; allow to cool.

13. Adjust the final volume to 2 mL with ACN. Mix thoroughly.

    EPA Method Deviation: The final volume is different from the EPA SOP since these volumes allow us to meet all required limits.

14. Filter the extract through a 0.45-µm Teflon filter using a disposable 3-mL syringe. Bottle the extract in a clear screw-cap, auto sampler vial. Label all vials with the Lancaster Labs number. All extracts are stored in a freezer until analysis.

Calculations:

See analysis method.

Statistical Information:

See analysis method.

Quality Assurance:

Each day samples are extracted, a blank and a laboratory control sample (LCS) (sodium sulfate blank spiked with compounds to be determined carried through the entire procedure) must be extracted. In addition, for every batch of samples, a matrix spike and matrix spike duplicate must be extracted. If there is limited sample which prevents the preparation of the MS/MSD, then LCSD must be prepared instead. A batch is defined as the samples to be extracted over a 14-day period but not to exceed 20 field samples. If more than 20 samples are prepared during this time, an additional batch must be prepared. If any client, agency, or state has more stringent QC or batch requirements, these must be followed instead. See the GC analysis methods for specifics on compounds in the spiking solutions.

Revision Log:

Initiated Date: 10/26/90

Ver. #	Effective Date	Change
00	03/21/95	Previous Issue
01	04/09/97	Major changes are as follows: • Split prep and analysis methods • Added Personnel Training & Qualifications • Added waste disposal to Safety
02	JAN 2 1 1998	Major changes are as follows: • Update method reference • Change final sample volume

Prepared by: _____ (092) Date: 12-19-97

Approved by: _____ Date: 1-7-98

Approved by: _____ Date: 1/8/98

Approved by: _____ Date: 1-13-98

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601

Procedural Amendment #1

Number: Analysis #3338

Title: Extraction Procedure for the Determination of Polynuclear Aromatic Hydrocarbons (PAH) in a Solid Matrix

Effective Date (listed on procedure): 01/21/98

Section(s) affected by change: Quality Assurance

Reason for addition(s) or change(s): No longer using open batches for this analysis

Change will be effective from (date): 02/05/99

Samples or project affected: All

List change(s) or addition(s) (specify which section):

Quality Assurance: *(section should read)*

For each batch of samples extracted, a blank, a laboratory control sample (LCS), (sodium sulfate blank spiked with compounds to be determined carried through the entire procedure) a matrix spike, and a matrix spike duplicate must be extracted. If there is limited sample which prevents the preparation of the MS/MSD, then an LCSD must be prepared instead. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. If any client, agency, or state has more stringent QC or bath requirements, these must be followed instead. See the GC analysis method for specifics on compounds in the spiking solution.

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Prepared by: _____ (092) Date: 2-19-99

Approved by: _____ Date: 2.19.99

Approved by: Danenys R. Clark Date: 2/24/99

Analysis #8402, 8403, 8404, 8405, 8406, 8407, 8408, 8409, 8410, 8411, 8412, 8413, 8414, 8415, 8416, 8417, 8418, 8419, 8420, 8421, 8422, 8423, 8424, 8777, 8802, 2311, 8825, 8727, 8427, 8428, 8429, 8430, 8431, 8432, 8433, 8434, 8435, 8436, 8437, 8438, 8439, 8440, 8441, 8442, 8443, 8444, 8445, 8446, 8728, 2312, 2313, 8465, 8466, 8467, 8468, 8469, 8470, 8471, 8472, 8473, 8474, 8475, 8476, 8477, 8478, 8479, 8480, 8481, 8482, 8729, 8449, 8450, 8451, 8452, 8453, 8454, 8455, 8456, 8457, 8458, 8459, 8460, 8461, 8462, 8463, 8801, 8833, 2289

Revision 01
Supersedes Date: 12/23/97
Effective Date: JAN 15 1999

Purgeable Halocarbons/Aromatics/Miscellaneous Organics in Water and Solid Samples

Reference:

1. Method 601 and Method 602, *40 CFR*, Part 136.

2. Methods 5030A, 8000A, Revision 1, SW-846, US EPA, July 1992.

3. Method 8020A, 8021A, Revision 1, SW-846, US EPA, September 1994.

4. Method 8010B, Revision 2, SW-846, US EPA, September 1994.

5. Method 5035, Revision 0, SW-846, US EPA, December 1996.

6. Method 5030B, 8021B, Revision 2, SW-846, US EPA, December 1996.

7. Method 8000B, Revision 3, SW-846, US EPA, December 1996.

8. Method 502.2, Revision 2, US EPA.

9. Method 503.1, US EPA.

Scope:

This method is suitable for analyzing water and solid samples for the purgeable halocarbon, aromatic, and miscellaneous organic compounds listed on Master Scans 1 through 4 (Form #2600, 2601, 2602, and 2603). The various Lancaster Laboratories' scan numbers which are analyzed under this method and the corresponding limits of quantitation are also listed on these forms.

The method employs gas chromatographic analysis of the compounds following preconcentration by a purge and trap concentrator and detection by a photoionization detector and an electrolytic conductivity detector (Hall detector) in series, with the Hall detector typically used as the primary detector.

Scan information for solid samples analyzed by SW-846 methods can be found on Master Form #2. These samples require preparation described in Lancaster Laboratories' Analyses #0379, "Methanolic Extraction of Soils and Solid Waste," and #8390, "Preparation of Volatile Soil Samples by EPA SW-846 Method 5035."

The methods as written in the Reference section above are very similar with only minor differences. Generally, all statements in this method will apply to the Reference section unless otherwise explicitly noted.

Summary:

The method is based on the purge and trap gas chromatography method where an inert gas is bubbled through 5 mL of the sample solution. The volatile halocarbons, aromatics, and miscellaneous organics are purged from the sample and trapped on a sorbent trap. After purging is complete, the sorbent trap is heated and backflushed with inert gas to desorb the trapped compounds onto a suitable gas chromatographic column. The gas chromatograph is then temperature programmed to separate the compounds which are then detected and quantified with a photoionization detector and an electrolytic conductivity detector in series. Typical chromatograms and printouts are shown in Figures 1 and 2.

Apparatus:

1. Purge and trap concentrator - O.I. Analytical 4560, Tekmar 3000, or equivalent device equipped with the Tenax/silica gel/charcoal trap. The trap must be at least 25 cm long and have an inside diameter of at least 0.105". If none of the CFCs (dichlorodifluoromethane, trichlorofluoromethane, or trichlorotrifluoroethane) are being analyzed for, the charcoal can be eliminated and replaced with more Tenax. If none of the gaseous compounds (chloromethane, bromomethane, vinyl chloride, or chloroethane) are being analyzed for, an all-Tenax trap can be used. A trap packed with Carbopack B and Carbosieve S-III or a trap packed with VOCARB 3000 may be used, but different desorption and bake temperatures must be used. A trap packed with VOCARB 4000 can be used if 2-chloroethyl vinyl ether is not being analyzed for. The purge-and-trap conditions are summarized in Table I.

   The purging chamber (sparge vessel) must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous headspace of the chamber must be <15 mL in volume.

The desorber should be capable of rapidly heating the trap to approximately 180°C for desorption.

2. Gas chromatograph - An HP 5890 or equivalent gas chromatograph capable of temperature programming and equipped with a Hall electrolytic conductivity detector and a photoionization detector that provide the proper sensitivity and linearity may be used. Although not necessary, if not analyzing for aromatic or miscellaneous organic compounds, the photoionization detector may be used to aid in the identification and confirmation of the multiple bonded compounds included in this method.

3. Autosampler (optional) – O.I. Analytical Model 4551, Archon, with sample cooler or equivalent. These multisamplers transfer water from standard 40-mL VOA vials to the common sparge vessel on the sample concentrators. The Archon has the capability to purge solid samples directly in the 40-mL vials.

4. Standard/surrogate spiker units (optional) – O.I. Analytical Model 4551 SIM/Spiker or equivalent. One or two automated standard/surrogate syringe spikers can be added to the O.I. Analytical Model 4551 autosampler to automatically introduce 10 µL of internal standard (ISTD), surrogate standard, and/or spiking solutions to the sample as it is being transferred to the sparge vessel. The Archon has a groove that can deliver 1 µL of surrogate or ISTD.

GC Columns:

75 m × 0.456 mm ID fused silica capillary column with bonded phase specifically designed for purgeables (e.g., J & W Scientific DB-VRX, DB-624, or equivalent). Normal operations will use the DB-VRX column; however, the DB-624 column may be used as a confirmation column. Other suitable columns may also be used as confirmation columns. The GC conditions are summarized in Table II.

Reagents:

Laboratory deionized water is used to prepare all sample dilutions and working standards. Purge and trap grade (or equivalent) methanol is also used to prepare all other calibration and QC standards. Standards can either be prepared in-house as stated in the Reference section from neat compounds obtained from suppliers which indicate the purity of the compound or prepurchased from suppliers. No correction for purity is made if the purity is listed as 96% or greater. If the purity of the neat compound standard is <96%, a calculation to correct for the standard purity is performed and documented in the standards notebook. Prepurchased standards must have the concentrations of the solutions documented by the supplier.

Materials:

1. Gastight microsyringes – 25 µL or larger

2. Glassware – Various sized volumetric flasks, Class A with ground-glass stopper; 15- and 40-mL precleaned vials

Safety Precautions:

The toxicities of all compounds used in this 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 the neat materials while preparing stock standards. These stocks must, therefore, be prepared in a hood to eliminate the risk of inhaling the vapors of the neat materials. After the neat materials are diluted with methanol or other solvents, the potential for exposure is reduced significantly. Nevertheless, care must be taken in the handling of any and all standards. Information concerning the known toxicity, properties, or special handling precautions for any compound can be found in the material safety data sheets available from the safety officer. Safety glasses and lab coats are required personal protective wear.

Waste Disposal:

The solvents utilized in this procedure are disposed of in a solvent waste container. All working solutions prepared in deionized water are flushed down the sink with tap water. Samples in 40-mL vials removed from the autosamplers are taken to storage until final disposal by incineration. Samples with a pH <2 are disposed of in an acid waste container. The decanted methanol created by the soil extraction process is disposed of in a solvent waste container. The solid waste is incinerated after the discard date.

Standards:

1. Surrogate standard – Stock surrogate standard, 1-bromo-4-chlorobenzene, is prepared in methanol from neat compound at a concentration of approximately 10,000 mg/L by adding about 100 mg of compound to methanol in a 10-mL volumetric flask.

   Internal standard – Fluorobenzene is used as an internal standard on the photoionization detector. The stock is prepared in methanol from neat compound at a concentration of approximately 10,000 mg/L by adding about 100 mg of each compound to methanol in a 10-mL volumetric flask.

   These standards are stored in 15-mL vials with screw-cap lids and Teflon-lined silicone septa at -10° to -20°C for up to 180 days.

   Secondary dilution standards in methanol at concentrations of approximately 15 mg/L are prepared monthly by diluting approximately 0.3 mL of each of the stock standards with methanol in a 200-mL volumetric flask. Secondary dilution standards in methanol are stored in 40-mL vials with screw-cap lids and Teflon-lined silicone septa at -10° to -20°C. They are labeled with the standard name, preparation date, expiration date, and storage conditions. Secondary dilution standards may also be stored in the SIM/Spiker unit at ambient temperature for no longer than 30 days.

The surrogate and the internal standards may be combined into one secondary standard for use.

Other compounds may be substituted or added as surrogates or internal standards if they do not coelute with or interfere with the quantitation of analytes of interest.

2. Calibration standards - Four different standards are used as calibration standards:

   a. Gaseous compounds - Premade solutions of dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride, chloroethane, and trichlorofluoromethane, each at concentrations of approximately 200 mg/L in methanol are purchased from a supplier and used as stock standards. These ampulized standards are stored at -10° to -20°C until the supplier's expiration date.

   The ampule of the stock standard, when ready to be used, is opened and poured into a 1.5-mL autoinjector vial with a screw-cap lid and a Teflon-lined septum and stored at -10° to -20°C. These secondary dilution standards are kept at -10° to -20°C at all times and are held for no more than 1 week before being discarded.

   Other ampulized standards containing the same six component compounds in methanol at higher concentrations may be used as stock standards. These standards must be diluted in methanol to a final concentration of approximately 200 mg/L before being used. After the ampule is opened, the diluted standards may be held for no more than 1 week before being discarded.

Care must be taken with the gaseous compound secondary dilution standards to ensure that they are kept at -10° to -20°C at all times, due to the high volatility of the compounds.

b. 2-chloroethyl vinyl ether (2-cleve) - Premade solutions of 2-cleve at a concentration of approximately 8000 mg/L in methanol are purchased from a supplier and used as stock standards. These ampulized standards are stored until the supplier's expiration date at -10° to -20°C.

Secondary dilution standards are prepared by diluting 1.0 mL of the stock standard with methanol in a 25-mL volumetric flask to give a final concentration of approximately 320 mg/L. Secondary dilution standards are stored in 1.5-mL autoinjector vials with screw-cap lids and Teflon-lined silicone septa at -10° to -20°C for 1 week. Dilution standards are held for no more than 1 day on the bench before being discarded.

2-Cleve secondary dilution standards must be prepared weekly due to the instability of the compound over longer periods of time.

c. Miscellaneous compounds – Stock standards of methyl *t*-butyl ether and trichlorotrifluoroethane are prepared in methanol from neat compounds at concentrations of approximately 10,000 mg/L by adding about 100 mg of each compound to methanol in a 10-mL volumetric flask. These standards are stored in 15-mL vials with screw-cap lids and Teflon-lined silicone septa at -10° to -20°C for up to 180 days.

Secondary dilution standards in methanol at concentrations of approximately 100 mg/L are prepared monthly by diluting 0.25 mL of the stock standard with methanol in a 25-mL volumetric flask. Secondary dilution standards are stored in 1.5-mL autoinjector vials with screw-cap lids and Teflon-lined silicone septa at -10° to -20°C for no longer than 30 days. Secondary dilution standards are held for no more than 12 hours on the bench before being discarded.

d. Primary compounds – Premade solutions containing each compound at a concentration of approximately 2000 mg/L in methanol are purchased from a supplier and used as stock standards. These ampulized standards are stored until the supplier's expiration date at -10° to -20°C.

Secondary dilution standards, prepared by diluting 1.25 mL of stock standard with methanol in a 25-mL volumetric flask to give a final concentration of approximately 100 mg/L, are prepared monthly. Secondary dilution standards are stored in 1.5-mL autoinjector vials with screw-cap lids and Teflon-lined septa at -10° to -20°C for no longer than 30 days. Secondary dilution standards are held for no more than 12 hours on the bench before being discarded.

The primary compound standards contain all compounds listed on Master Scan forms (1 through 4), except for the compounds listed in Parts a, b, and c of this section.

Generally, the primary compounds secondary dilution calibration standard and the miscellaneous compounds secondary dilution calibration standard are prepared in the same volumetric flask.

3. Quality control check standards - Four different standards are used as QC check standards:

   a. Gaseous compounds - Premade solutions of dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride, chloroethane, and trichlorofluoromethane, each at concentrations of approximately 2000 mg/L in methanol are purchased from a supplier and used as stock standards. The source for the gaseous compounds QC check standard is different from the source for the gaseous compound's calibration standard. These ampulized standards are stored until the supplier's expiration date at -10° to -20°C.

   Secondary dilution standards are prepared by diluting 1.0 mL (or 0.5 mL) of the stock standard with methanol in a 10-mL (or 5-mL) volumetric flask at -10° to -20°C to give a final concentration of approximately 200 mg/L. Secondary dilution standards are stored in 1.5-mL autoinjector vials with screw-cap lids and Teflon-lined septa at -10° to -20°C. Secondary dilution standards are kept at -10° to -20°C at all times and are held for no more than 1 week before being discarded.

   Other ampulized standards containing the same six component compounds in methanol at different concentrations may be used as stock standards. These standards must be diluted in methanol to a final concentration of approximately 200 mg/L (if their original concentration is >200 mg/L) before being used. After the ampule is opened, the diluted standards may be held for no more than 1 week before being discarded.

   Care must be taken with the gaseous compound secondary dilution standards to ensure that they are kept at -10° to -20°C at all times, due to the high volatility of the compounds.

b. 2-chloroethyl vinyl ether (2-cleve) – Premade solutions of 2-cleve at a concentration of approximately 5000 mg/L in methanol are purchased from a supplier and used as stock standards. The source for the 2-cleve QC check standard is different from the source for the 2-cleve calibration standard. These ampulized standards are stored until the supplier's expiration date at -10° to -20°C.

Secondary dilution standards are prepared by diluting 1.0 mL of the stock standard with methanol in a 25-mL volumetric flask to give a final concentration of approximately 200 mg/L. Secondary dilution standards are stored in 1.5-mL autoinjector vials with screw-cap lids and Teflon-lined silicone septa at -10° to -20°C for 1 week. Dilution standards are held for no more than 1 day on the bench before being discarded.

2-Cleve secondary dilution standards must be prepared weekly due to the instability of the compound over longer periods of time.

c. Miscellaneous compounds – Stock standards of methyl *t*-butyl ether and trichlorotrifluoroethane are prepared in methanol from neat compounds at concentrations of approximately 10,000 mg/L by adding about 100 mg of each compound to methanol in a 10-mL volumetric flask. These standards are stored in 16-mL vials with screw-cap lids and Teflon-lined silicone septa at -10° to -20°C for up to 180 days.

Secondary dilution standards in methanol at concentrations of approximately 200 mg/L are prepared monthly by diluting 0.2 mL of the stock standard with methanol in a 10-mL volumetric flask. Secondary dilution standards are stored in 1.5-mL autoinjector vials with screw-cap lids and Teflon-lined silicone septa at -10° to -20°C for no longer than 30 days. Secondary dilution standards are held for no more than 12 hours on the bench before being discarded.

d. Primary compounds - Premade solutions containing each compound at a concentration of approximately 2000 mg/L in methanol are purchased from a supplier and used as stock standards. The source for the primary compound's QC check standard is different from the source for the primary compound's calibration standard. These ampulized standards are stored until the supplier's expiration date at -10° to -20°C.

Secondary dilution standards, prepared by diluting 1.0 mL of stock standard with methanol in a 10-mL volumetric flask to give a final concentration of approximately 200 mg/L, are prepared monthly. Secondary dilution standards are stored in 1.5-mL autoinjector vials with screw-cap lids and Teflon-lined septa at -10° to -20°C for no longer than 30 days. Secondary dilution standards are held for no more than 12 hours on the bench before being discarded.

The primary compound standards contain all compounds listed on Master Scan forms (1 through 4), except for the compounds listed in Parts a, b, and c of this section.

Generally, the primary compound's secondary dilution QC check standard and the miscellaneous compound's secondary dilution QC check standard are prepared in the same volumetric flask.

4. Spiking standards – Four different standards are used as spiking standards:

a. Gaseous compounds – Gaseous compound's spiking standards are prepared by diluting 0.5 mL (or 1 mL) of the gaseous compound's secondary dilution QC check standard with methanol in a 5-mL (or 10-mL) volumetric flask at -10° to - 20°C to give a final concentration of approximately 20 mg/L. Spiking standards are stored in 1.5-mL

autoinjector vials with screw-cap lids and Teflon-lined silicone septa at -10° to -20°C. Secondary dilution standards are kept at -10° to -20°C at all times and are held for no more than 1 week before being discarded.

Care must be taken with the gaseous compound spiking standards to ensure that they are kept at -10° to -20°C at all times, due to the high volatility of the compounds.

b. 2-Chloroethyl vinyl ether (2-cleve) – Premade solutions of 2-cleve at a concentration of approximately 2000 mg/L in methanol are purchased from a supplier and used as stock standards. These ampulized standards are stored until the supplier's expiration date at -10° to -20°C.

2-Cleve secondary dilution spiking standards are prepared by diluting 0.25 mL of the stock standard with methanol in a 50-mL volumetric flask to give a final concentration of approximately 10 mg/L. Secondary dilution standards are stored in 1.5-mL autoinjector vials with screw-cap lids and Teflon-lined silicone septa at -10° to -20°C for 1 week. Secondary dilution standards are held for no more than 12 hours on the bench before being discarded.

2-Cleve secondary dilution standards must be prepared weekly due to the instability of the compound over longer periods of time.

c. Miscellaneous compounds – The same standard used as the miscellaneous compound's stock QC check standard is used as the stock spiking standard (containing methyl *t*-butyl ether and trichlorotrifluoroethane each at concentrations of approximately 10,000 mg/L).

Secondary dilution standards in methanol at concentrations of approximately 10 mg/L are prepared monthly by diluting 0.1 mL (or 0.05 mL) of stock standard with methanol in a 100-mL (or 50-mL) volumetric flask. Secondary dilution standards are stored in 1.5-mL autoinjector vials with screw-cap lids and Teflon-lined silicone septa at -10° to -20°C for no longer than 30 days. Secondary dilution standards are held for no more than 12 hours on the bench before being discarded.

d. Primary compounds – Premade solutions containing each compound at a concentration of approximately 2000 mg/L in methanol are purchased from a supplier and used as stock standards. These ampulized standards are stored until the supplier's expiration date at -10° to -20°C.

Secondary dilution standards are prepared monthly by diluting 0.5 mL (or 0.25 mL) of stock standard with methanol in a 100-mL (or 50-mL) volumetric flask to give a final concentration of approximately 10 mg/L. Secondary dilution standards are stored in 1.5-mL autoinjector vials with screw-cap lids and Teflon-lined silicone septa at -10° to -20°C for no longer than 30 days. Secondary dilution standards are held for no more than 12 hours on the bench before being discarded.

The primary compound standards contain all compounds listed on Master Scan forms (1 through 4), except for the compounds listed in Parts a, b, and c of this section.

Generally, the primary compound's secondary dilution spiking standard and the miscellaneous compound's secondary dilution spiking standard are prepared in the same volumetric flask.

5. Low-level vinyl chloride standard – Premade solutions of vinyl chloride at a concentration of approximately 200 mg/L in methanol are purchased from a supplier and used as stock standards. These ampulized standards are stored until the supplier's expiration date at -10° to -20°C.

   Secondary dilution standards are prepared by diluting 0.5 mL (or 1 mL) of the stock standard with methanol in a 5-mL (or 10-mL) volumetric flask at -10° to -20°C to give a final concentration of approximately 20 mg/L. Secondary dilution standards are stored in 1.5-mL autoinjector vials with screw-cap lids and Teflon-lined silicone septa at -10° to -20°C. Secondary dilution standards are kept at -10° to -20°C at all times and are held for no more than 30 days before being discarded.

   Care must be taken with the low-level vinyl chloride standard to ensure that it is kept at -10° to -20°C at all times, due to the high volatility of the compound.

See Table III at the end of this method for a summary of concentrations, storage conditions, and shelf life for standards used with this method.

Calibration:

At least five levels of calibration are required for all target analytes and the surrogate when calibrating according to the SW-846 methods. At least three levels are required when using the 600 series methods. Methods 503.1 and 502.2 give the option of single-point calibration or using a calibration curve. Method 503.1 does not specify the number of levels required for a calibration curve. For Method 502.2, the number of calibration levels is dependent on the calibration range desired. A minimum of three levels is required to calibrate a range of a factor of 20 in concentration. For a factor of 50, at least four levels are used and for a factor of 100, at least five levels are used.

For each method, the calibration range should be near but above the method detection limit through a working range that mimics the normal concentration found in samples. The calibration range used is from approximately 0.5 to 140 µg/L (approximately 10 to 60 µg/L for the surrogate standard, approximately 1 to 120 µg/L for gaseous compounds, and approximately 1 to 140 µg/L for 2-chloroethyl vinyl ether). For every initial calibration that is performed, an MDL standard must be analyzed to ensure that all compounds of interest are detectable.

Working calibration standards are prepared by diluting the appropriate volumes (1.5 to 470 µL) of the gaseous compound's secondary dilution standard, the 2-chloroethyl vinyl ether secondary dilution standard, the miscellaneous organic compound's secondary dilution standard (mix A or B), the surrogate secondary dilution standard, and the primary compound's secondary dilution standard (also containing the miscellaneous compounds) with reagent water into 25-, 50-, 100-, 200-, or 500-mL volumetric flasks. For the high-level soil calibration, add 5% methanol by volume to mimic the solid sample extract analysis conditions. All secondary dilution standards, except the gaseous compounds, are allowed to come to ambient temperature before an aliquot is withdrawn. (Due to the high volatility of the compounds, the gaseous compound standards should be added to the flask last.) The working standards are mixed by inverting the volumetric 3x. The working standards are transferred into 40-mL vials with Teflon-lined silicone septa if the autosampler is being used. 5 mL of this vial is transferred via automation to the sparge vessel along with 10 µL of the secondary dilution/internal standard solution (via the SIM/Spiker unit).

Calibration can be performed using either the external or internal standard calibration. 1-Bromo-4-chlorobenzene is normally used as a surrogate. For the internal standard calibration, fluorobenzene is normally used as the internal standard. The calibration factor (CF; as defined in SOP-OR-020, "Manual Calculation of the Analyte Response/Calibration Factors, the Relative Standard Deviation for Analyte Response/Calibration Factors, and the Correlation Coefficient") is calculated for each analyte in each calibration level. If the relative standard deviation (RSD) of the CFs for any analyte is <20% for SW-846 methods or 10% for 500 and 600 series methods, the

average CF may be used for the quantitation. If the RSD exceeds 20% for SW-846 methods or 10% for 500 and 600 series methods, a calibration curve must be utilized. If the %RSD fails criteria, the calibration curve must be reviewed by comparing the individual calibration factors for each level. Any suspect levels are to be repeated to see if a lower %RSD can be achieved. For 8021B, if compounds fail the % RSD criteria, the average calibration factor may still be used providing that the average of all of the % RSDs is <20%. Alternately, a quadratic fit or linear least squares fit to the calibration data may be used. For Method 8021B, a quadratic curve fit may be used if six calibration standards are analyzed.

For Method 502.2 and 503.1, a single-point calibration may be utilized if the response of the standard used for the single point is ±20% of the response of the compound in the sample that the single-point calibration is being used for. Information on single-point calibration can be found in SOP-OR-004, "Manual Calculations of Analyte Concentrations for Volatiles by GC."

Once the system is calibrated, the working calibration curve is verified by analyzing a working QC check standard. This standard is prepared by diluting 20 µL of the primary compounds secondary dilution standard (also containing the miscellaneous compounds), 20 µL of the 2-chloroethyl vinyl ether secondary dilution standard, and 20 µL of the gaseous compound's secondary dilution standard with reagent water into a 200-mL volumetric flask to give a final concentration of approximately 20 µg/L of each compound. For the high-level soil QC check standard, add 5% methanol by volume to mimic the solid sample extract analysis conditions. All secondary dilution standards, except the gaseous compounds, are allowed to come to ambient temperature before an aliquot is withdrawn. The working QC check standard is mixed by inverting the volumetric flask 3×. (Due to the high volatility of the compounds, the gaseous compounds standard should be added to the flask last.)

The working standard is transferred into a 40-mL vial with a Teflon-lined silicone septum if the autosampler is being used. A 5-mL aliquot of this vial is transferred via automation to the sparge vessel along with the appropriate amount of secondary dilution surrogate/internal standard solution (via the automatic spiking unit).

If the recovery (% drift or difference for 8021B) of any analyte is outside the acceptable range (see Appendix A), the QC check standard is repeated. (For 8021B, if compounds fail the % drift (difference) criteria, the average calibration factor may still be used providing that the average of all of the % drifts (differences) is <15%.) The compounds originally outside the acceptable range are rechecked. If these compounds are within the acceptable range on the repeat, the QC check standard is acceptable. If these compounds are still outside the acceptable range on the repeat, the system is recalibrated.

For 8021B external standard calibrations, bracketing QC check standards must be performed every 10 samples or 12 hours, whichever comes first, and at the end of each batch of 20 samples. If the recovery of any analyte is outside the acceptable range (see Appendix D), the samples run between the QC check standards are checked for the presence of those analytes outside the acceptable range. If analytes are present, the samples are rerun under the QC check standards that are acceptable. The calibration curves are verified in this manner at least every 10 samples.

Sample Collection, Preservation, and Preparation:

Samples should be collected in triplicate in 40-mL vials with Teflon-lined silicone septa. All samples must be cooled to 2° to 6°C (36° to 43°F) at the time of collection until analysis.

Samples being analyzed for aromatic compounds should be adjusted to pH <2 with approximately 0.2 mL of 1:1 hydrochloric acid (HCl). Samples to be analyzed for trihalomethanes should be preserved with sodium thiosulfate (approximately 10 to 40 mL of sample) or ascorbic acid (approximately 25 to 40 mL of sample).

If 2-chloroethyl vinyl ether is to be analyzed for, the sample should not be acidified. Samples referencing Method 502.2 should be preserved with both the HCl solution and the ascorbic acid (sodium thiosulfate may be substituted for the ascorbic acid).

For solid samples, two preparation procedures are described in Lancaster Laboratories' Analyses #0379, #8390. For all samples, no further preparation is required except for the addition of the surrogate compound and possibly for dilutions which are described below in the Procedure section.

All acid preserved 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 analysis. A quad study consists of four laboratory control standards that are carried through all steps of the analysis and that meet the defined acceptance criteria. Documentation for these studies are in each individual's training records.

Procedure:

Prior to analyzing samples on the O.I. Analytical Model 4551 autosampler or Archon, the following must be completed:

1. The SIM/Spiker units are filled with secondary dilution surrogate/internal standard solution.

2. The sequence function on the O.I. Analytical Model 4560 or Tekmar 3000 concentrator is properly programmed. (A sequence of up to 51 injections can be analyzed at one time.)

3. The autosampler waste container is empty and the wash vial is approximately half full.

Set the purge and trap and GC conditions as described in Tables I and II for the particular trap and column being used. Calibrate the system as described above and perform the necessary QC analyses as described below.

When not using the autosampler, 5 mL of the sample is manually prepared in a 5-mL syringe. The sample vial is allowed to come to room temperature before analysis. The sample may be prepared by either pouring it directly into the syringe (dilution of 1), diluting it into the syringe (low dilution), or diluting it into a volumetric flask and pouring 5 mL of the flask contents into the syringe (high dilution). 10 µL of the secondary dilution surrogate/internal standard solution is manually added to the 5-mL syringe before loading onto the concentrator.

When using the autosampler, all samples, blanks, and standards are prepared and poured into 40-mL vials with Teflon-lined silicone septa. For each injection, 5 mL of the sample is withdrawn from the vial and transferred to the sparge vessel along with 1 µL for the Archon or 10 µL for the 4551 of the secondary dilution surrogate/internal standard solution.

1. Identification of analytes - Comparison of sample peak retention times to standard peak retention times is used to tentatively identify compounds. A window of ±3 standard deviations from the average retention time based on three previously analyzed QC check standards can also be used as the basis for identification. Further considerations should also include normal vs. abnormal peak shape, comparison of the chromatograms obtained from each detector (when a PID is being used) and each different column (if multiple columns are being used), and the shift in retention times of the surrogates and internal standards. Ultimately, the experience and discretion of the analyst should weigh heavily in the interpretation of the chromatogram.

2. Higher dilutions - Samples which contain levels of analytes above the dynamic range of the method (the highest-level calibration standard) at the dilution factor of analysis must be reanalyzed. Before continuing with the analysis of the further diluted sample, the analyst must be assured that the high level of the analyte present will not carry over into the next injection. This can be accomplished by analyzing a reagent water blank (cleanup blank). If the analytes are all below the limit of quantitation (LOQ), then the analysis of the further diluted sample can begin. If not, the cleanup blank is repeated until analyte levels are below the quantitation limit. Stricter cleaning may be required if lower reporting limits are required.

   To dilute a sample, the sample is pulled into a 25-, 100-, 250-, 500-, or 1000-µL gas-tight syringe. The exact volume is then added to enough reagent water in a volumetric flask (or possibly a 5-mL syringe, if not using the autosampler) to achieve the correct dilution factor. The contents of the flask are mixed by inverting the flask 3× and then poured into the 40-mL vial (or 5-mL syringe).

   Care should be taken to avoid carryover of high levels. The syringes and flasks used in diluting samples and the sparge vessel should be cleaned by rinsing with methanol and reagent water before analyzing further samples.

   The dilution factor (DF) is calculated as follows:

   When the sample is diluted directly into the 5-mL syringe:

$$DF = \frac{5}{(mL\ of\ sample\ added\ to\ the\ syringe)}.$$

   When an intermediate dilution into a volumetric flask is performed (always performed if using the autosampler):

$$DF = \left(\frac{TV}{Vs}\right) \times \left(\frac{5}{Vds}\right)$$

Where:

TV = Total volume (in mL) of the intermediate dilution (i.e., the volume of the volumetric flask)

Vs = Volume (in mL) of sample which is diluted in the intermediate dilution

Vds = Volume (in mL) of the diluted sample which is added to the 5-mL syringe

If more than one intermediate dilution is performed, the factor (TV/Vs) is calculated for each intermediate dilution. The factor (5/Vds) is omitted when the sample dilution is being poured into a 40-mL vial for use on the autosampler.

Calculation:

Procedures and the necessary equations for manual and automatic (computer data reduction) calculations are found in SOP-OR-004.

Quality Control:

In order to monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix, each blank, standard, sample, and spiked sample is spiked with 1 µL for the Archon or 10 µL for the 4551 of secondary dilution surrogate/internal standard solution. Surrogate recoveries should be within the statistically generated range (±3 standard deviations from the average recovery based on at least 25 previously analyzed samples) or within the 70% to 130% range until windows have been generated for a particular matrix. If the internal standard method is

used, the height of the internal standard for each injection is recorded. The acceptable range for the height is 80% to 120% of the average of those obtained during calibration or 80% to 120% of the height obtained in a recently analyzed acceptable QC check standard. If the recoveries fall outside of these ranges, the injection should be repeated.

For Lancaster Laboratories Analysis #8390, surrogates are spiked into the extract at the appropriate concentration for the volume of methanol used.

As stated above in the Calibration section, the calibration curves are verified at least every 10 samples by analyzing a QC check standard which contains every analyte of interest. If the recovery of any analyte is outside of the acceptable range, the QC check standard is repeated. Acceptable ranges are defined as 80% to 120% for 500 series methods and 85% to 115% for method 8021A. The acceptance criteria for method 8021B is ±15% drift (difference). All other reference methods specify differing windows for each individual compound. The compounds with recoveries originally outside the acceptable range are rechecked. If these compounds are within the acceptable range on the repeat, the QC check standard is acceptable. If these compounds are still outside the acceptable range on the repeat, the system is recalibrated.

For 8021B, if compounds fail the % drift (difference) criteria, the average calibration factor may still be used providing that the average of all of the % drifts (differences) is <15%.

For 8021B external standard calibrations, bracketing QC check standards must be performed every 10 samples or 12 hours, whichever comes first, and at the end of each batch of 20 samples. If the recovery of any analyte is outside the acceptable range (see Appendix A), the samples run between the QC check standards are checked for the presence of those analytes outside the acceptable range. If analytes are present, the samples are rerun under QC check standards that are acceptable. The calibration curves are verified in this manner at least every 10 samples.

A matrix spike (MS) and matrix spike duplicate (MSD) are performed on one sample in each batch of up to 20 samples. For the MS and MSD injections, each of the relevant spiking solutions (see Parts 4.a., 4.b., 4.c., and 4.d. of the Standards section) is added to the sample dilution so that a final spiking concentration of approximately 20 µg/L is achieved for all compounds. When using the autosampler, the primary compound's secondary dilution spiking standard (also containing the miscellaneous compounds) and the 2-chloroethyl vinyl ether secondary dilution spiking standard must be diluted by a factor of 500 into the volumetric flask which contains the intermediate sample dilution. In the same flask, the gaseous compounds spiking standard must be diluted by a factor of 1000.

The sample injection using a 5-mL syringe, 10 µL of the primary compound's secondary dilution spiking standard (also containing the miscellaneous compounds), 10 µL of the 2-chloroethyl vinyl ether secondary dilution spiking standard, and 5 µL of the gaseous compound's spiking standard must be added directly to the 5-mL syringe containing the sample dilution.

For Lancaster Laboratories Analysis #8390, an MS and MSD are performed by spiking the compounds of interest into the extract at the appropriate concentration for the volume of methanol used.

Maximum allowable spike recovery windows are generated by Department 25 on an annual basis. The window is calculated using + or - 3 standard deviations from the average recovery based on at least 30 data points.

If the recovery for any analyte falls outside the allowable spike recovery window, the Batch QC Protocol Flowchart in Figure 3 at the end of this method is followed. The laboratory control sample (LCS) referenced in this flowchart is prepared identically to the MS and MSD injections described above, except that reagent water is used as the sample matrix. The analysis of the LCS is required in each batch of up to 20 samples for Method 8021B.

A laboratory fortified blank (LFB) for 502.2 is spiked at 2 µg/L for vinyl chloride and 5 µg/L for all other compounds and is analyzed once per 20-sample batch.

Method blanks (reagent water with 1 µL for the Archon or 10 µL for the 4551 of secondary dilution surrogate/internal standard solution) are analyzed at the rate of at least one per 24 hours each day the instrument is analyzing samples. For the high-level soil method blank, add 5% methanol by volume to mimic the solid sample extract analysis conditions. Additional method blanks may be analyzed if contamination is suspected or if the analyst is trying to assess the background levels for compounds of interest. Method blanks are considered in specifications if all compounds of interest are below the method detection limit. If a compound of interest exceeds the method detection limit, additional method blanks must be analyzed until the analyte level is below the method detection limit.

The results from the unspiked (BKGD), MS, MSD, and LCS (if necessary) samples are recorded in the Lancaster Laboratories' sample management/QA database, referencing each appropriate batch of up to 20 samples in which it was performed. Surrogate standard recoveries and blank results are also entered into the database.

Statistical limits are maintained and distributed to the department by the department's QC coordinator.

Revision Log:

Initiated Date: 12/18/87

Ver. #	Effective Date	Change
00	12/27/96	Previous Issue
01	12/23/97	Major changes are as follows: • Incorporated EPA Update III method changes

Ver. #	Effective Date	Change
01	JAN 15 1999	Major changes are as follows:

- Changed Analysis #0180, 0181 ,0182, 0296, 0297, 0300, 0418, 0419, 0420, 0463, 0515, 0537, 0538, 0539, 0912, 0913, 1032, 1033, 1035, 1165, 1170, 1176, 1227, 1228, 1334, 1379, 1380, 1382, 1462, 1564, 4146, 4267, 4268, 4269, 4717, 5022, 5023, 5264, 5531, 5559, 5643, 5646, 6314, 6975, 7040, 7110, 7111, 7282, 7287, 7288, 7289, 7310, 7331, 7531, 7729, 7730, 7730, 7844, 7860 to Analysis #8402, 8403, 8404, 8405, 8406, 8407, 8408, 8409, 8410, 8411, 8412, 8413, 8414, 8415, 8416, 8417, 8418, 8419, 8420, 8421, 8422, 8423, 8424, 8777, 8802, 2311, 8825, 8727, 8427, 8428, 8429, 8430, 8431, 8432, 8433, 8434, 8435, 8436, 8437, 8438, 8439, 8440, 8441, 8442, 8443, 8444, 8445, 8446, 8728, 2312, 2313, 8465, 8466, 8467, 8468, 8469, 8470, 8471, 8472, 8473, 8474, 8475, 8476, 8477, 8478, 8479, 8480, 8481, 8482, 8729, 8449, 8450, 8451, 8452, 8453, 8454, 8455, 8456, 8457, 8458, 8459, 8460, 8461, 8462, 8463, 8801, 8833, 2289
- Removed Appendix A, B, & C; changed Appendix D to Appendix A
- Revised Quality Control section
- Revised Scope section

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Table I
Purge-and-Trap Conditions

Trap Type*	T/SG/C	CPB/CSS	V3 or V4
Purge Ready Temp (°C)	20	20	20
Purge Flow (mL/min)**	40	40	40
Purge Time (min) - required	12	12	12
Dry Purge Time (min)	0+	13	0
Desorb Preheat Temp (°C)***	0	0	0
Desorb Temp (°C)	180	250	245
Desorb Time (min) - required	4	4	4
Bake Temp (°C)	180	260	260
Bake Time (min)	10 to 20	10 to 20	10 to 20
Heated Valve and Line Temps (°C)	100 to 130	100 to 130	100 to 130
Purge Pressure (psi)****	20 to 25	20 to 25	20 to 25
Bake Gas Bypass*****	OFF	OFF	OFF
Water Management Settings (for O.I. Analytical Model 4560 concentrator)			
Purge Temp (°C)	100	100	100
Desorb Temp (°C)	0	0	0
Bake Temp (°C)	240	240	240

Table I (Continued)
Purge-and-Trap Conditions

*T/SG/C = Tenax/Silica Gel/Charcoal
CPB/CSS = Carbopack B/Carbosieve S-III
V3 = VOCARB 3000
V4 = VOCARB 4000
**Can be set lower for optimum gases response (32 to 40 mL/min)
***Not activated if O.I. Analytical Model 4560 concentrator is being used
****Will drop to 3-10 psi during purge cycle on O.I. Analytical Model 4560 concentrator
*****Not applicable if O.I. Analytical Model 4560 concentrator is being used
+If the silica gel is eliminated, a dry purge time may be used.

Higher bake temperatures and times may be used to remove analytes which may carry over after the analysis of samples containing high levels of volatiles.

Unless listed above as required, the purge-and-trap conditions may be modified to achieve optimum instrument performance based on the manufacturer's specifications without adversely affecting the method performance.

Table II
GC Conditions

Detector Temp - Hall (°C)	250
Detector Temp - PID (°C)	250
Injector Temp (°C)	220
Carrier Flow (mL/min)	6 to 12
Detector Makeup Flow (mL/min).	20 to 25

Temperature Program (for the DB-VRX column listed in the GC columns section)

Initial Temp (°C)	30
Initial Hold Time (min)	8
First Ramp Rate (°C/min)	5
First Hold Temp (°C)	60
First Hold Time (min)	1
Second Ramp Rate (°C/min)	19
Final Temp (°C)	200
Final Hold Time (min)	4.7

O-I Electrolytic Conductivity Detector

Mode	Halogen
Reactor Tube	Nickel 1/16-inch OD
Reactor Temp (°C)	800 to 900
Electrolyte	1-Propanol
Electrolyte Flow (mL/min)	0.03 to 0.05
Reaction Gas	Hydrogen, 90 to 110 mL/min
Sensitivity	Low

Table II (Continued)
GC Conditions

The ELCD sensitivity should be set so that 0.5 µg/L of chloroform gives an S/N ratio of at least 10:1. If the sensitivity of the ELCD is not sufficient to reach this level, the electrolyte, the conductivity cell, the reactor tube, and other components should be cleaned or replaced.

The PID sensitivity should be set so that 1 µg/L of benzene gives an S/N ratio of at least 10:1. If the sensitivity of the PID is not sufficient to reach this level, the lamp should be cleaned or replaced.

Alternatively, the purge-and-trap concentrator should be checked for leaks and/or poor trap performance.

The GC conditions may be modified to achieve optimum instrument performance based on the manufacturer's specifications without adversely affecting the method performance.

Table III
Standards Used in the Purgeable Halocarbons/ Aromatics/Miscellaneous Organics Method

Standard	Approximate Concentration	Storage	Shelf Life
Surrogate/ Internal (stock)	10,000 mg/L in methanol	-10° to -20°C, vial	180 days
Surrogate/Internal (sec. dil.)	15 mg/L in methanol	-10° to -20°C, vial	30 days
		-10° to -20°C, volumetric flask	30 days
		ambient, SIM/Spiker	30 days
		ambient, vial	1 day
Gas. Cmpds. Cal. (stock)	200 mg/L in methanol	-10° to -20°C, ampule	Until exp. date
Gas. Cmpds. Cal. (sec. dil.)	200 mg/L in methanol	-10° to -20°C, vial	1 week
Gas. Cmpds. Cal. (working)	1 to 120 µg/L in water	ambient, flask	5 minutes
2-Cleve Cal. (stock)	8,000 mg/L in methanol	-10° to -20°C, ampule	Until exp. date
2-Cleve Cal. (sec. dil.)	320 mg/L in methanol	-10° to -20°C, vial	1 week
		ambient, vial	1 day
2-Cleve Cal. (working)	1 to 140 µg/L in water	ambient, flask	5 minutes
Misc. Cmpds. Cal. (stock)	10,000 mg/L in methanol	-10° to -20°C, vial	180 days

Table III (Continued)
Standards Used in the Purgeable Halocarbons/ Aromatics/Miscellaneous Organics Method

Standard	Approximate Concentration	Storage	Shelf Life
Misc. Cmpds. Cal. (sec. dil.)	100 mg/L in methanol	-10° to -20°C, vial ambient, vial	30 days 1 day
Misc. Cmpds. Cal. (working)	0.5 to 140 µg/L in water	ambient, flask	5 minutes
Primary Cmpds. Cal. (stock)	2,000 mg/L in methanol	-10° to -20°C, ampule	Until exp. date
Primary Cmpds. Cal. (sec. dil.)	100 mg/L in methanol	-10° to -20°C, vial ambient, vial	30 days 1 day
Primary Cmpds. Cal. (working)	0.5 to 140 µg/L in water	ambient, flask	5 minutes
Gas. Cmpds. QC Check (stock)	2,000 mg/L in methanol	-10° to -20°C, ampule	Until exp. date
Gas. Cmpds. QC Check (sec. dil.)	200 mg/L in methanol	-10° to -20°C, vial	1 week
Gas. Cmpds. QC Check (working)	20 µg/L in water	ambient, flask	5 minutes
2-Cleve QC Check (stock)	5,000 mg/L in methanol	-10° to -20°C, ampule	Until exp. date
2-Cleve QC Check (sec. dil.)	200 mg/L in methanol	-10° to -20°C, vial ambient, vial	1 week 1 day
2-Cleve QC Check (working)	20 µg/L in water	ambient, flask	5 minutes

Table III (Continued)
Standards Used in the Purgeable Halocarbons/ Aromatics/Miscellaneous Organics Method

Standard	Approximate Concentration	Storage	Shelf Life
Misc. Cmpds. QC Check (stock)	10,000 mg/L in methanol	-10° to -20°C, vial	180 days
Misc. Cmpds. QC Check (sec. dil.)	200 mg/L in methanol	-10° to -20°C, vial ambient, vial	30 days 1 day
Misc. Cmpds. QC Check (working)	20 µg/L in water	ambient, flask	5 minutes
Primary Cmpds. QC Check (stock)	2,000 mg/L in methanol	-10° to -20°C, ampule	Until exp. date
Primary Cmpds. QC Check (sec. dil.)	200 mg/L in methanol	-10° to -20°C, vial ambient, vial	30 days
Primary Cmpds. QC Check (working)	20 µg/L in water	ambient, flask	5 minutes
Gas. Cmpds. Spike	20 mg/L in methanol	-10° to -20°C, vial	1 week
2-Cleve Spike (stock)	2000 mg/L in methanol	-10° to -20°C, ampule	Until exp. date
2-Cleve Spike (sec. dil.)	10 mg/L in methanol	-10° to -20°C, vial ambient, vial	7 days 1 day
2-Cleve Spike (working)	20 µg/L in water	ambient, flask	5 minutes

Table III (Continued)
Standards Used in the Purgeable Halocarbons/ Aromatics/Miscellaneous Organics Method

Standard	Approximate Concentration	Storage	Shelf Life
Misc. Cmpds. Spike (stock)	10,000 mg/L in methanol	-10° to -20°C, vial	180 days
Misc. Cmpds. Spike (sec. dil.)	10 mg/L in methanol	-10° to -20°C, vial ambient, vial	30 days 1 day
Primary Cmpds. Spike (stock)	2000 mg/L in methanol	-10° to -20°C, ampule	Until exp. date
Primary Cmpds. Spike (sec. dil.)	10 mg/L in methanol	-10° to -20°C, vial ambient, vial	30 days 1 day
Low-Level V.C. (stock)	200 mg/L in methanol	-10° to -20°C, ampule	Until exp. date
Low-Level V.C. (sec. dil.)	20 mg/L in methanol	-10° to -20°C, vial	30 days
Low-Level V.C. (working)	1 µg/L in water	ambient, flask	5 minutes

*Contains the primary compounds, the miscellaneous compounds, the gaseous compounds, and 2-chloroethyl vinyl ether

Appendix A

Maximum Allowable QC Check Standard Recovery Range in the Halocarbons/Aromatics/Miscellaneous Organics Method

Compounds	8010B	601
Dichlorodifluoromethane	82.0-174.0	82.0-174.0
Chloromethane	59.5-140.5	59.5-140.5
Vinyl chloride	68.5-131.5	68.5-131.5
Bromomethane	58.5-141.5	58.5-141.5
Chloroethane	77.0-123.0	77.0-123.0
Trichlorofluoromethane	66.5-133.5	66.5-133.5
1,1-Dichloroethene	63.0-137.0	63.0-137.0
Methylene Chloride	77.5-122.5	77.5-122.5
Trichlorotrifluoroethane	85.0-115.0	85.0-115.0
trans-1,2-Dichloroethene	64.0-136.0	64.0-136.0
1,1-Dichloroethane	84.0-116.0	84.0-116.0
cis-1,2-Dichloroethene	64.0-136.0	64.0-136.0
Bromochloromethane	85.0-115.0	85.0-115.0
Chloroform	75.0-125.0	75.0-125.0
2,2-Dichloropropane	85.0-115.0	85.0-115.0
1,2-Dichloroethane	71.5-128.5	71.5-128.5
1,1,1-Trichloroethane	71.0-129.0	71.0-129.0
1,1-Dichloropropene	85.0-115.0	85.0-115.0
Carbon Tetrachloride	68.5-131.5	68.5-131.5
Dibromethane	85.0-115.0	85.0-115.0
1,2-Dichloropropane	74.0-126.0	74.0-126.0
Trichloroethene	77.0-123.0	77.0-123.0
Bromodichloromethane	76.0-124.0	76.0-124.0
2-Chloroethyl vinyl ether	60.0-140.0	60.0-140.0
cis-1,3-Dichloropropene	64.0-136.0	64.0-136.0
1,1,2-Trichloroethane	64.0-136.0	64.0-136.0
1,3-Dichloropropane	78.5-121.5	78.5-121.5
Dibromochloromethane	85.0-115.0	85.0-115.0

Appendix A (Continued)

Maximum Allowable QC Check Standard Recovery Range in the Halocarbons/Aromatics/Miscellaneous Organics Method

Compounds	8010B	601
Ethylene dibromide	85.0-115.0	65.5-134.5
Tetrachloroethene	70.0-130.0	85.0-115.0
1,1,1,2-Tetrachloroethane	85.0-115.0	70.0-130.0
Chlorobenzene	72.0-128.0	85.0-115.0
Bromoform	73.5-126.5	72.0-128.0
1,1,2,2-Tetrachloroethane	49.0-151.0	73.5-126.5
1,2,3-Trichloropropane	85.0-115.0	49.0-151.0
Bromobenzene	85.0-115.0	85.0-115.0
o-Chlorotoluene	85.0-115.0	85.0-115.0
p-Chlorotoluene	85.0-115.0	85.0-115.0
m-Dichlorobenzene	49.5-150.5	85.0-115.0
p-Dichlorobenzene	69.5-130.5	49.5-150.5
o-Dichlorobenzene	70.0-130.0	69.5-130.5
1,2-Dibromo-3-chloropropane	85.0-115.0	70.0-130.5
1,2,4-Trichlorobenzene	85.0-115.0	85.0-115.0
Hexachlorobutadiene	85.0-115.0	85.0-115.0
1,2,3-Trichlorobenzene	85.0-115.0	85.0-115.0
1,2-Dichloroethene	64.0-136.0	64.0-136.0
Compounds	**8020A**	**602**
Vinyl chloride	68.5-131.5	68.5-131.5
1,1-Dichloroethene	63.0-137.0	63.0-137.0
trans-1,2-dichloroethene	64.0-136.0	64.0-136.0
Methyl *t*-butyl ether	85.0-115.0	85.0-115.0
cis-1,2-Dichloroethene	64.0-136.0	64.0-136.0
1,1-Dichloropropene	85.0-115.0	85.0-115.0
Benzene	77.0-123.0	77.0-123.0
Trichloroethene	77.0-123.0	77.0-123.0
2-Chloroethyl vinyl ether	60.0-140.0	60.0-140.0

Appendix A (Continued)

Maximum Allowable QC Check Standard Recovery Range in the Halocarbons/Aromatics/Miscellaneous Organics Method

Compounds	8020A	602
cis-1,3-Dichloropropene	64.0-136.0	64.0-136.0
trans-1,3-Dichloropropene	64.0-136.0	64.0-136.0
Toluene	77.5-122.5	77.5-122.5
Tetrachloroethene	70.0-130.0	70.0-130.0
Chlorobenzene	72.0-128.0	72.0-128.0
Ethylbenzene	63.0-137.0	63.0-137.0
m, p-Xylene	85.0-115.0	85.0-115.0
Styrene	85.0-115.0	85.0-115.0
o-Xylene	85.0-115.0	85.0-115.0
Isopropylbenzene	85.0-115.0	85.0-115.0
Bromobenzene	85.0-115.0	85.0-115.0
n-Propylbenzene	85.0-115.0	85.0-115.0
o-Chlorotoluene	85.0-115.0	85.0-115.0
p-Chlorotoluene	85.0-115.0	85.0-115.0
1,3,5-Trimethylbenzene	85.0-115.0	85.0-115.0
tert-Butylbenzene	85.0-115.0	85.0-115.0
1,2,4-Trimethylbenzene	85.0-115.0	85.0-115.0
sec-Butylbenzene	85.0-115.0	85.0-115.0
m-Dichlorobenzene	72.5-127.5	72.5-127.5
p-Dichlorobenzene	69.5-130.5	69.5-130.5
p-Isopropyltoluene	85.0-115.0	85.0-115.0
o-Dichlorobenzene	68.0-132.0	68.0-132.0
n-Butylbenzene	85.0-115.0	85.0-115.0
1,2,4-Trichlorobenzene	85.0-115.0	85.0-115.0
Napthalene	63.3-132.0	63.3-132.0
Hexachlorobutadiene	85.0-115.0	85.0-115.0
1,2,3-Trichlorobenzene	85.0-115.0	85.0-115.0
Total Xylenes	85.0-115.0	85.0-115.0

Figure 1

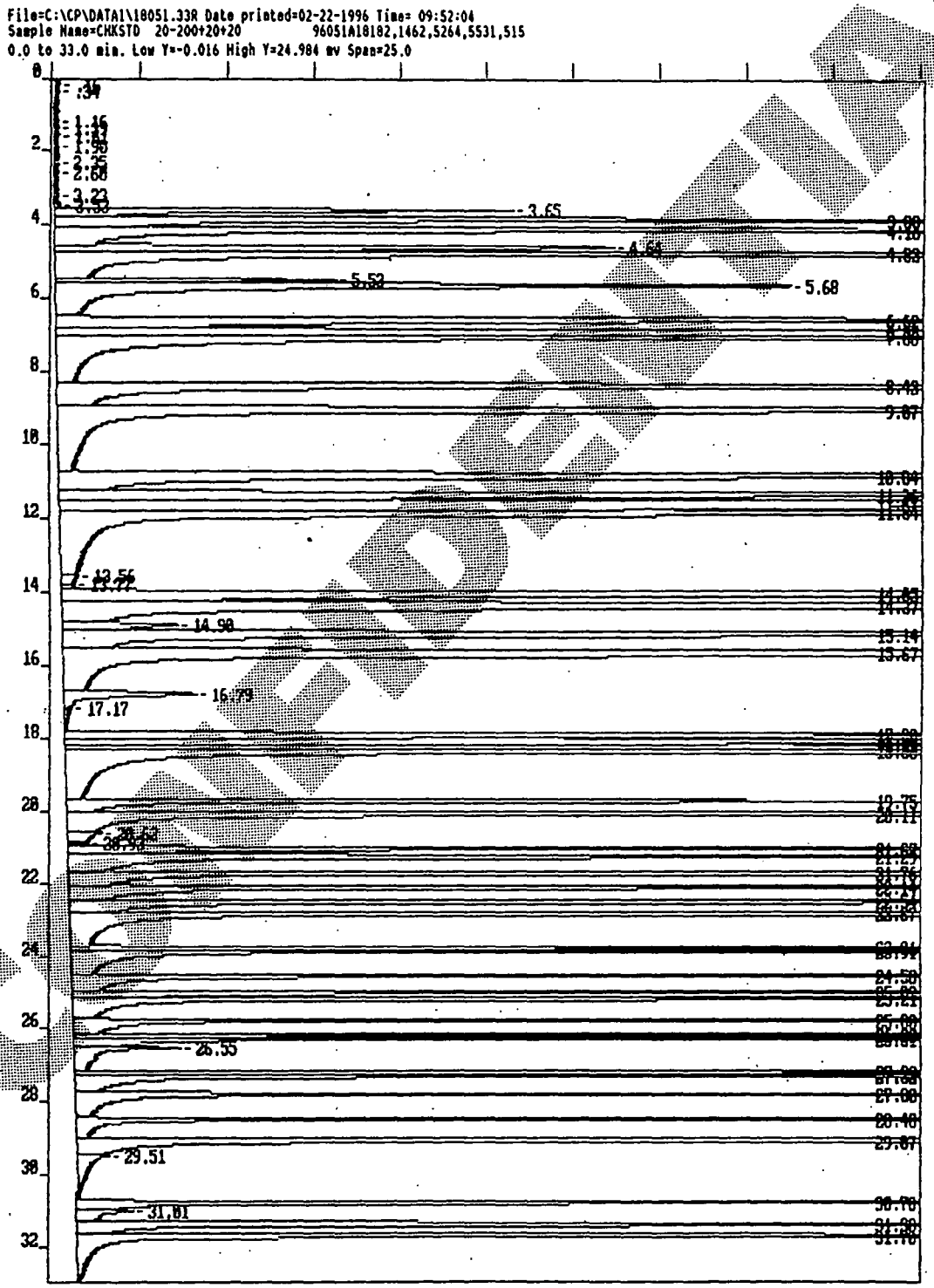


Figure 1 (Continued)

Sample Name: CHKSTD 20-200+20+20
Batch: 96051A18 Date Taken: Feb 21, 1996 16:14:00
Raw File: C:\CP\DATA1\18051.33R
Area File: C:\CP\DATA1\18051.33A
Method File: C:\CP\DATA1\18051.MET(version 2)
EPA Methods 502.2/601/8010B/8021A Run Time: 33
75m x 0.45mm ID J&W Scientific DB-VRX column
T/SG/C/ trap Inst. ID#05843 - 5890#18HALL
Peak Width: .05 Dil. Factor: 1 Analyst: 115
Threshold: -3 Calculation: EXTERNALStd using peak heights
Peak table using calibration C:\CP\DATA1\H18043.CAL(version 20):

Ret Time (min)	Expected RT (min)	Peak Name	Amount * (ug/L)	Peak Area	Peak Height
3.647	3.649	DICHLORODIFLUOROMETH	33.0186	71926	13172
3.883	3.896	CHLOROMETHANE	20.3467	196722	28905
4.156	4.166	VINYL CHLORIDE	20.8369	255933	32585
4.640	4.640	BROMOMETHANE	16.8485	131115	16050
4.817	4.825	CHLOROETHANE	19.3910	293473	44673
5.678	5.693	TRICHLOROFLUOROMETHA	18.9266	228774	21117
6.617	6.627	1,1-DICHLOROETHENE	19.1041	305991	43489
6.929	6.933	METHYLENE CHLORIDE	19.5839	351139	58621
7.084	7.103	TRICHLOROTRIFLUOROET	21.6748	401385	37090
8.423	8.433	TRANS-1,2-DICHLOROET	20.4455	453244	71688
9.069	9.077	1,1-DICHLOROETHANE	21.8257	547211	64549
10.844	10.862	CIS-1,2-DICHLOROETHE	22.3995	513253	65789
11.359	11.376	BROMOCHLOROMETHANE	21.9687	376582	49393
11.610	11.633	CHLOROFORM	22.3515	656474	75856
11.844	11.873	2,2-DICHLOROPROPANE	21.8241	403220	30139
14.047	14.073	1,2-DICHLOROETHANE	21.1954	461492	61567
14.367	14.394	1,1,1-TRICHLOROETHAN	20.0041	547908	52100
15.143	15.166	1,1-DICHLOROPROPENE	21.2320	433177	52120
15.667	15.687	CARBON TETRACHLORIDE	21.6729	652416	67643
17.903	17.850	DIBROMOMETHANE	21.7983	234337	43726
18.080	18.030	1,2-DICHLOROPROPANE	21.9046	440335	72612
18.247	18.270	TRICHLOROETHENE	22.0390	565864	100787
18.360	18.390	BROMODICHLOROMETHANE	22.0086	550357	85908
19.754	19.770	2-CHLOROETHYL VINYL	21.4884	147698	32844
20.107	20.050	CIS-1,3-DICHLOROPROP	24.8653	477757	112289
21.067	21.086	TRANS-1,3-DICHLOROPR	20.7022	349073	100033
21.292	21.313	1,1,2-TRICHLOROETHAN	21.6918	518824	132420
21.755	21.774	1,3-DICHLOROPROPANE	21.6452	413593	113119
22.139	22.159	DIBROMOCHLOROMETHANE	22.1321	355381	94240
22.541	22.560	ETHYLENE DIBROMIDE	23.0542	244158	67905
22.868	22.888	TETRACHLOROETHENE	22.1793	579528	161089
23.806	23.820	1,1,1,2-TETRACHLOROE	22.1836	519729	165948
23.905	23.930	CHLOROBENZENE	22.2063	275066	79178
24.582	24.604	BROMOFORM	22.6138	244086	74980
25.032	25.055	1,1,2,2-TETRACHLOROE	21.3067	351620	122652
25.214	25.236	1,2,3-TRICHLOROPROPA	22.1446	326690	104550
25.798	25.824	BROMOBENZENE	21.5443	170006	51596
26.202	26.220	O-CHLOROTOLUENE	22.1303	191218	67380
26.308	26.330	P-CHLOROTOLUENE	22.6166	209755	69527
27.221	27.250	M-DICHLOROBENZENE	22.0010	291523	105205
27.316	27.340	P-DICHLOROBENZENE	22.6948	349680	112837
27.804	27.830	O-DICHLOROBENZENE	21.8317	331278	101570
28.484	28.513	1,2-DIBROMO-3-CHLORO	21.6065	106068	27258
29.074	29.100	SURR-1BR4CLBENZENE-H	95.1187	507500	143319
30.777	30.806	1,2,4-TRICHLOROBENZE	22.3075	305176	80526
31.383	31.408	HEXACHLOROBUTADIENE	23.0655	397434	89741
31.703	31.731	1,2,3-TRICHLOROBENZE	21.8832	327031	71961
		TOTAL 1,2-DICHLOROETHENE:	42.8450		

* Amounts in ug/L for water samples, ug/kg for solid samples.
Surrogate Recovery: BrClBenzene 95.1187%

Figure 2

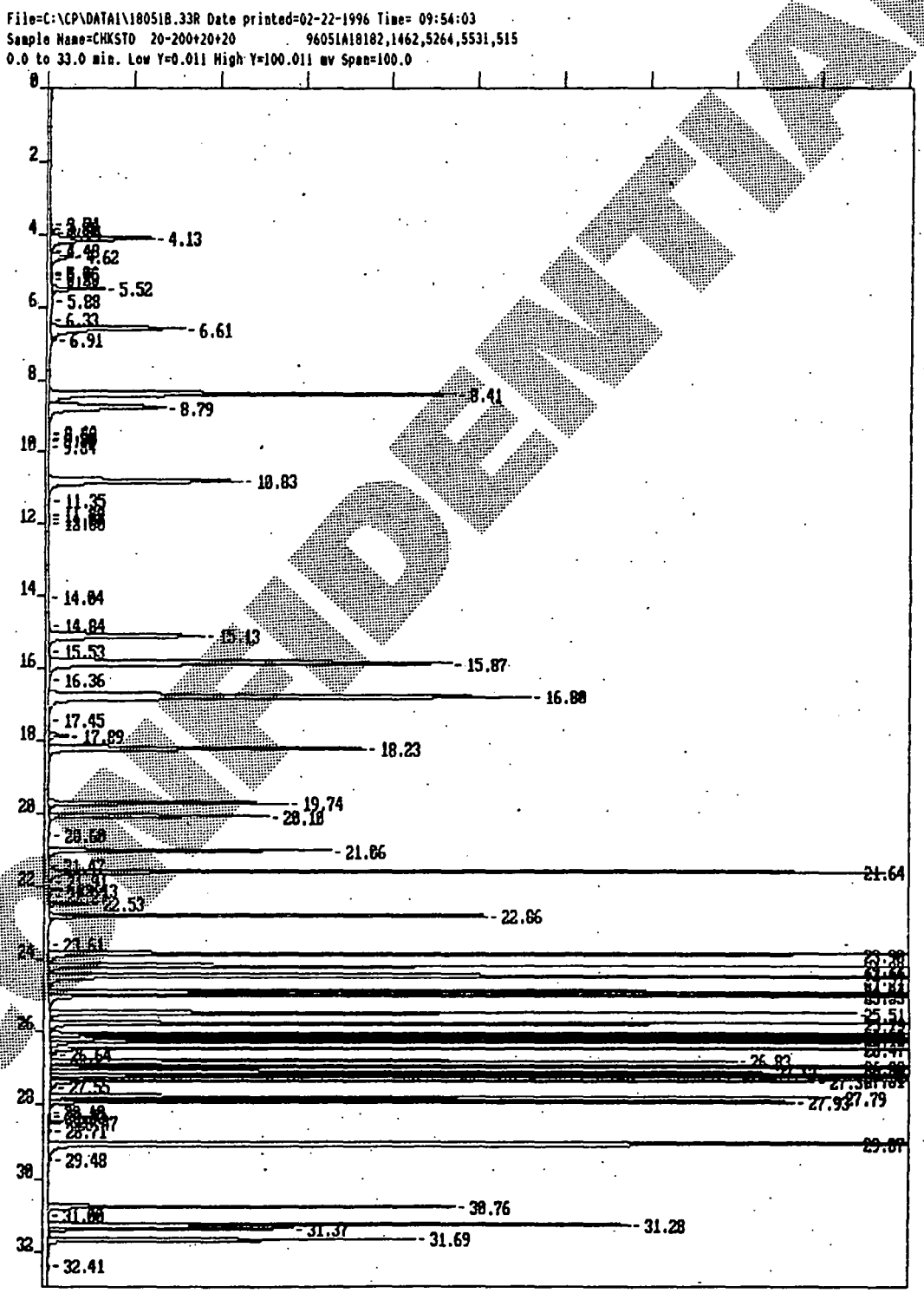


Figure 2 (Continued)

Batch: 96051A18 Date Taken: Feb 21, 1996 16:14:00
Raw File: C:\CP\DATA1\18051B.33R
Area File: C:\CP\DATA1\18051B.33A
Method File: C:\CP\DATA1\18051B.MET(version 2)
EPA Methods 502.2/601/8010B/8021A Run Time: 33
75m x 0.45mm ID J&W Scientific DB-VRX column
T/SG/C trap Inst. ID#05843 - 5890#18PID
Peak width: .03 Dil. factor: 1 Analyst: 115
Threshold: 0 Calculation: INTERNALStd using peak heights

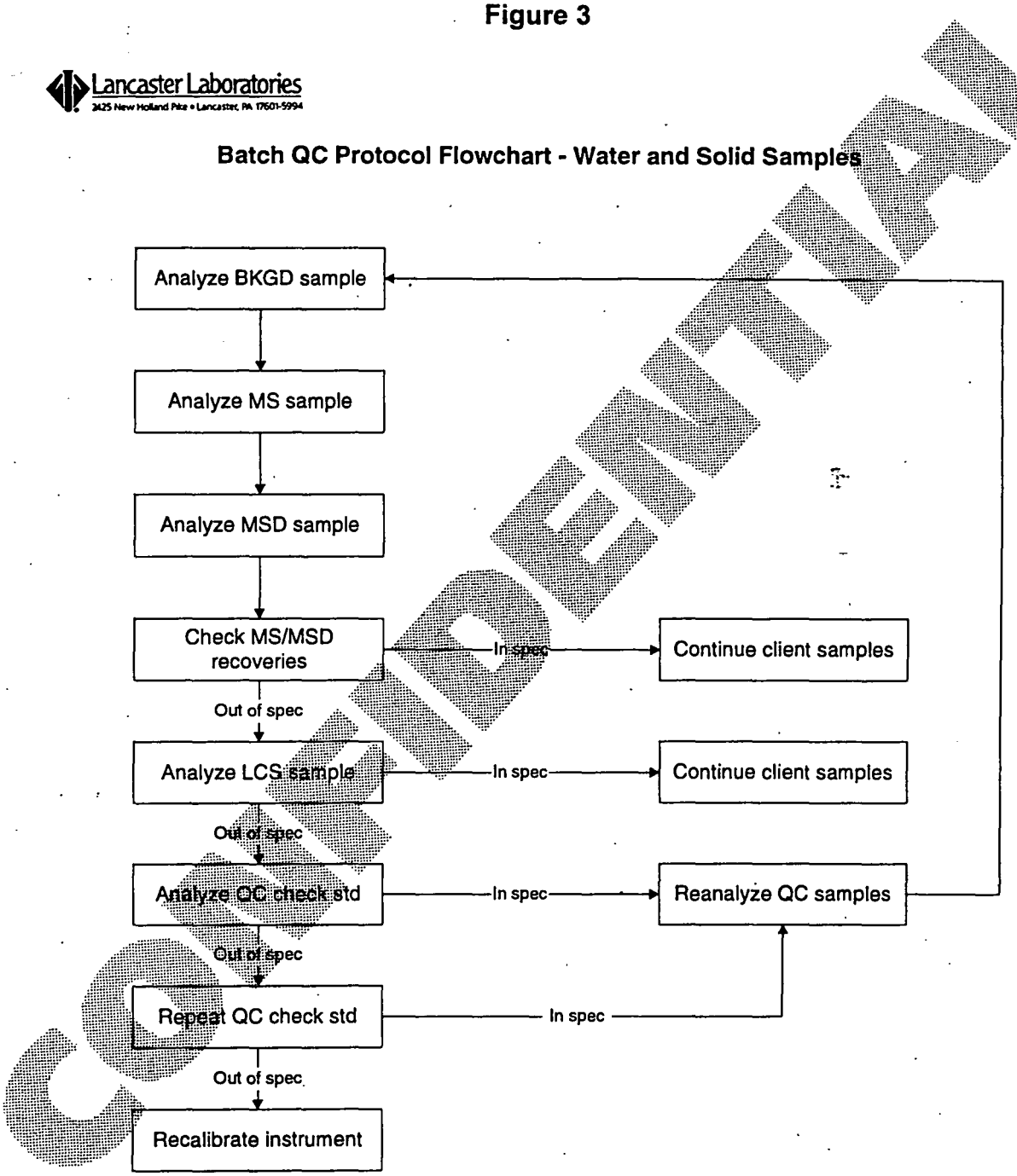
Peak table using calibration C:\CP\DATA1\P18043.CAL(version 13):

Ret Time (min)	Expected RT (min)	Peak Name	Amount * (ug/L)	Peak Area	Peak Height
4.133	4.139	VINYL CHLORIDE	25.5354	80133	11867
5.876	5.892	ACETONE	0.9452	810	155
6.608	6.616	1,1-DICHLOROETHENE	18.4527	102272	15380
8.410	8.422	TRANS-1,2-DICHLOROET	19.9931	266975	47052
8.787	8.801	METHYL T-BUTYL ETHER	19.7488	120354	13428
10.526	10.526	METHYL ETHYL KETONE	0.0000	0	0
10.830	10.847	CIS-1,2-DICHLOROETHE	21.9136	162631	22205
15.131	15.153	1,1-DICHLOROPROPENE	21.7548	133008	17955
15.870	15.894	BENZENE	21.0923	334443	46684
16.802	16.824	ISTD-FBENZENE	1.0000	416051	55919
18.234	18.256	TRICHLOROETHENE	21.5289	197592	36759
19.745	19.763	2-CHLOROETHYL VINYL	20.6628	112153	27774
20.097	20.116	CIS-1,3-DICHLOROPROP	23.5638	94995	25357
21.056	21.076	TRANS-1,3-DICHLOROPR	19.9150	107502	32705
21.639	21.660	TOLUENE	21.1771	324040	97143
22.857	22.878	TETRACHLOROETHENE	21.1500	152616	50774
23.896	23.918	CHLOROBENZENE	21.3528	352025	129617
24.224	24.247	ETHYLBENZENE	21.5614	298121	110819
24.506	24.515	M-XYLENE	42.9952	699796	229346
24.935	24.950	STYRENE	21.1980	377643	154334
25.027	25.040	O-XYLENE	21.3117	316487	120488
25.507	25.510	CUMENE	24.2332	250561	93519
25.788	25.812	BROMOBENZENE	21.5138	366288	140342
26.083	26.100	N-PROPYLBENZENE	21.4052	272187	103794
26.190	26.210	O-CHLOROTOLUENE	21.8761	312369	117551
26.295	26.310	P-CHLOROTOLUENE	21.4113	331034	126830
26.473	26.430	1,3,5-TRIMETHYLBENZE	21.5867	407947	154407
26.631	26.790	TERT-BUTYLBENZENE	21.7918	228092	79521
26.983	26.940	1,2,4-TRIMETHYLBENZE	21.5864	312067	117784
27.121	27.140	SEC-BUTYLBENZENE	21.8210	235707	82491
27.211	27.230	M-DICHLOROBENZENE	21.6057	314411	117245
27.306	27.320	P-DICHLOROBENZENE	21.7273	319906	118206
27.368	27.390	P-ISOPROPYLTOLUENE	22.6503	239231	88813
27.794	27.810	O-DICHLOROBENZENE	21.5759	251232	90569
27.928	27.880	N-BUTYLBENZENE	22.0436	248228	86415
29.065	29.080	SURR-1BR4CLBENZENE-P	105.4191	790756	259683
30.765	30.794	1,2,4-TRICHLOROBENZE	21.3783	169632	47296
31.276	31.300	NAPHTHALENE	21.2064	250784	67348
31.370	31.390	HEXACHLOROBUTADIENE	21.7979	112760	28227
31.691	31.650	1,2,3-TRICHLOROBENZE	21.2734	167459	42602
	TOTAL XYLENES:	64.3069			

* Amounts in ug/L for water samples, ug/kg for solid samples.
Surrogate Recovery: BrClBenzene 105.4191%
Chrom Perfect version 6.07 Reported on 02-22-1996at 09:56:48

Internal Standard Calculations From ISTDCHK v. 950609.1130
IStd height for ISTD-FBENZENE is within range.
IStd range: 44,602 - 66,903 (80% - 120% of midpoint 55,753)
Used check standard file C:\CP\DATA1\18051B.20A to determine range.

Figure 3



2527 02/23/96

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601

Procedural Amendment #1

Number: Analysis #8402, 8403, 8404, 8405, 8406, 8407, 8408, 8409, 8410, 8411, 8412, 8413, 8414, 8415, 8416, 8417, 8418, 8419, 8420, 8421, 8422, 8423, 8424, 8777, 8802, 2311, 8825, 8727, 8427, 8428, 8429, 8430, 8431, 8432, 8433, 8434, 8435, 8436, 8437, 8438, 8439, 8440, 8441, 8442, 8443, 8444, 8445, 8446, 8728, 2312, 2313, 8465, 8466, 8467, 8468, 8469, 8470, 8471, 8472, 8473, 8474, 8475, 8476, 8477, 8478, 8479, 8480, 8481, 8482, 8729, 8449, 8450, 8451, 8452, 8453, 8454, 8455, 8456, 8457, 8458, 8459, 8460, 8461, 8462, 8463, 8801, 8833, 2289

Title: Purgeable Halocarbons/Aromatics/Miscellaneous Organics in Water and Solid Samples

Effective Date (listed on procedure): 01/15/99

Section(s) affected by change: Quality Control

Reason for addition(s) or change(s): Clarify QC requirement

Change will be effective from (date): 02/11/99

Samples or project affected: All

List change(s) or addition(s) (specify which section):

Quality Control: *(add as paragraph #11)*

If the LCS does not meet criteria, the nonconformance is documented on the "Nonconformance Form" (Form #2586). Corrective action may include instrument maintenance and/or recalibration, re-extraction and/or reanalysis of the samples, or data qualification and is determined on a case-by-case basis.

84028403.DOC
021199

Prepared by: Kah M Snyder Date: 2-11-99

Approved by: Michele M. Turner Date: 2/11/99

Approved by: Dorothy M Love Date: 2/11/99

Allow to cool. Transfer the solution to a 100-mL volumetric flask. Adjust volume to the mark with deionized water and mix. Transfer the solution to a Nalgene bottle. The sample is now ready for analysis.

0820, 1848, 6360 (SW-846) - Shake sample well. Using a 50-mL graduated cylinder, transfer 50 mL of well-mixed sample into a 250-mL beaker. Add 1 mL of (1:1) HNO_3 and 5 mL of (1:1) HCl. Heat the solution on a hot plate at about 95°C for approximately 2 hours or until sample volume is reduced to between 15 and 20 mL, making certain the sample does not boil.

Allow to cool. Transfer the solution to a 50-mL volumetric flask. Adjust volume to the mark with deionized water and mix. Transfer the solution to a Nalgene bottle. The sample is now ready for analysis.

NOTE: When insoluble matter is present in the digestate, allow it to settle by gravity or filter prior to introduction to the instrument.

Quality Assurance:

Perform a method blank, sample duplicate, sample matrix spike, sample matrix spike duplicate, and laboratory control sample with every #0820 AA (SW-846), #6360 GF for Sb (SW-846), and #1848 ICP (SW-846) digestion batch (20 samples or less).

Perform a method blank, sample duplicate, sample matrix spike, and laboratory control sample with every #5720 ICP (CLP) and #6360 GF for Sb (CLP) digestion batch (20 samples or less).

Revision Log:

Initiated Date: 03/06/91

Ver. #	Effective Date	Change
00	02/22/96	Previous issue

2. Because SW-846 and CLP protocols are combined into one method for efficiency, acid volumes are based only on the CLP total recoverable method.

   For SW-846 protocol, 1 mL (1:1) HNO_3 and 5 mL (1:1) HCl is used instead of 2 mL HNO_3 and 5 mL HCl. Final acid concentration in Method 3005A is 2% HNO_3/5% HCl; final acid concentration in this method is 1% HNO_3/5% HCl. No effect on data has been observed; performance evaluation standard samples have been acceptable.

3. Ribbed watch glasses are not used during evaporation; samples are heated without watch glasses in non-metallic hoods to speed evaporation. No contamination trends have been observed in prep blanks evaporated without using watch glasses.

4. Samples are heated at about 95°C on hotplates, not 92° to 95°C as stated in ILMO4.0; hotplates cannot be maintained within 3°C range.

Scope:

This digestion procedure is used by the Metals Department of the Environmental Sciences Division. This method is used whenever SW-846 Method 3010 is not requested or required for total metals.

Basic Principle:

Samples are heated with nitric and hydrochloric acids with a substantial reduction in volume during digestion to dissolve metals.

Analysis #0820, 1848, 5720, 6360
Revision 01
Supersedes Date: 02/22/96
Effective Date: DEC 3 1 1998

Sample Preparation of Wastewater for Analysis of Total Recoverable Metals by Flame Atomic Absorption (0820) or Inductively Coupled Plasma Atomic Emission Spectrometry (1848 & 5720) and for Analysis of Antimony (6360) by Graphite Furnace Atomic Absorption Spectrometry

Reference:

1. USEPA CLP SOW No. ILM02.1, Exhibit D, Section III, Part A.1., Page D-5.
2. USEPA CLP SOW No. ILM04.0, Exhibit D, Section III, Part A., Page D-5.
3. Method 3005A, *Test Methods for Evaluating Solid Waste*, USEPA SW-846, Third Edition, Revision 1, Part I, Chapter 3, Office of Solid Waste and Emergency Response, Washington, DC 20460, July 1992 (**Modified**).

Purpose:

This acid digestion procedure is used to prepare wastewater samples for measurement of total recoverable metals by flame atomic absorption (FLAA) or inductively coupled plasma atomic emission spectroscopy (ICP-AES) and for analysis of antimony (Sb) by graphite furnace atomic absorption spectrometry (GFAA) following SW-846 and CLP protocols.

Reference Modifications:

1. For SW-846 protocol, a 50-mL sample aliquot and final volume is used instead of 100 mL to improve digestion throughput, conserve sample usage, and limit waste generation. Because all reagents are also adjusted so that concentrations are equivalent to a 100-mL aliquot, there is no impact on the data.

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 and Holding Times for Inorganic Analyses."

5720, 6360 (CLP) - Shake sample well. Using a 100-mL graduated cylinder, transfer 100 mL of well-mixed sample into a 250-mL beaker. Add 2 mL of (1:1) HNO_3 and 10 mL of (1:1) HCl. Heat the solution on a hot plate at about 95°C for approximately 2 hours or until the sample volume is reduced to between 25 and 50 mL, making certain the sample does not boil.

Apparatus and Reagents:

For reagent preparation, shelf life, and storage conditions, see SOP-IO-007, "Preparation of Standards and Solutions."

1. 250-mL beakers, 400-mL beakers, or other appropriate beakers
2. Watch glasses
3. Nitric acid, HNO_3, 70.0% to 71.0%, Baker Instra-Analyzed reagent, 1.428 g/mL, or equivalent
4. Hydrochloric acid, HCl, 36.5% to 38.0%, Baker Instra-Analyzed reagent, 1.194 g/mL, or equivalent
5. Nitric acid (1:1) - Add 500 mL of HNO_3 to 500 mL of deionized water
6. Hydrochloric acid (1:1) - Add 500 mL of HCl to 500 mL of deionized water
7. 100-mL graduated cylinders or other appropriate graduated cylinders
8. 100-mL volumetric flasks or other appropriate Class A volumetric flasks
9. 125-mL Nalgene bottles or other appropriate Nalgene bottles
10. Hot plates, adjustable and capable of maintaining a temperature of 90° to 95°C

NOTE: If boron (B) or silicon (Si) is requested on a sample, use Teflon vessels.

NOTE: As long as the correct ratios are maintained, solutions may be prepared using multiples of indicated weights and volumes.

Allow to cool. Transfer the solution to a 100-mL volumetric flask. Adjust volume to the mark with deionized water and mix. Transfer the solution to a Nalgene bottle. The sample is now ready for analysis.

0820, 1848, 6360 (SW-846) - Shake sample well. Using a 50-mL graduated cylinder, transfer 50 mL of well-mixed sample into a 250-mL beaker. Add 1 mL of (1:1) HNO_3 and 5 mL of (1:1) HCl. Heat the solution on a hot plate at about 95°C for approximately 2 hours or until sample volume is reduced to between 15 and 20 mL, making certain the sample does not boil.

Allow to cool. Transfer the solution to a 50-mL volumetric flask. Adjust volume to the mark with deionized water and mix. Transfer the solution to a Nalgene bottle. The sample is now ready for analysis.

NOTE: When insoluble matter is present in the digestate, allow it to settle by gravity or filter prior to introduction to the instrument.

Quality Assurance:

Perform a method blank, sample duplicate, sample matrix spike, sample matrix spike duplicate, and laboratory control sample with every #0820 AA (SW-846), #6360 GF for Sb (SW-846), and #1848 ICP (SW-846) digestion batch (20 samples or less).

Perform a method blank, sample duplicate, sample matrix spike, and laboratory control sample with every #5720 ICP (CLP) and #6360 GF for Sb (CLP) digestion batch (20 samples or less).

Revision Log:

Initiated Date: 03/06/91

Ver. #	Effective Date	Change
00	02/22/96	Previous issue

Ver. #	Effective Date	Change
01	DEC 3 1 1998	Major changes are as follows: • Title revised for clarification • 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 • Personnel Training and Qualifications added for compliance with SOP-LA-033

08201848.DOC
121798

Prepared by: Eugene K. Abel Date: 12-23-98

Approved by: Tamara Moss Date: 12/28/98

Approved by: Dorothy M Love Date: 12/29/98

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 1 8 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. Ahof Date: 1-14-99

Approved by: Robert Strocko Jr. Date: 1.14.99

Approved by: Dorothy M Love Date: 1/15/99

The Setup and Pouring of an ICP Run

Purpose:

The purpose of this SOP is to outline the proper procedure used to set up and pour an ICP run.

Scope:

This SOP will cover the apparatus and quality assurance procedures needed to set up and pour an ICP run. The procedure is the same regardless of method and/or protocol.

Personnel Training and Qualifications:

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.

Apparatus:

The following is a list of the apparatus necessary to the setup and pouring of an ICP run:

1. ICP preprinted run sheets (pages 1-4)

   Page 1: Includes standards and initial QC preprinted according to method and instrument.

   See Figures 1-6 for examples of run sheets.

   Figure 1 - Trace/CLP & EPA 600

   Figure 2 - Trace/SW-846

   Figure 3 - Trace/Multipoint Calibration (pages 2 and 3 can be used for any trace ICP run)

   Figure 4 - I61/CLP & EPA 600

   Figure 5 - I61/SW-846 (pages 2 and 3 can be used for any I61 run)

   Figure 6 - I61/Multipoint Calibration

2. Test tube racks
3. 13- x 100-mm Polystyrene Tubes (61)
4. 17- x 100-mm Polystyrene Tubes (Trace ICPs)
5. Filter paper - Whatman No. 40, 90 mm ashless
6. 1 x 100 10-mL sterile disposable syringes

7. 13-mm syringe filters, PTFE, 0.45 µm

8. 30-mL polypropylene medicine cups

9. Eppendorf fixed-volume hand-held pipettes; 50, 100, 200, 250, 500, and 1000 µL

   NOTE: For routine operation, calibration, and maintenance of Eppendorf fixed-volume hand-held pipettes, see MC-IO-003, "Fixed Volume Hand Held Pipettes."

10. Eppendorf pipette tips (or equivalent)

    a. 1 to 100 µL yellow plastic

    b. 200 to 1000 µL blue plastic

Solutions:

See SOP-IO-007, "Preparation of Standards and Solutions," Section C.

Safety:

Refer to SOP-IO-011, "Inorganic Analysis Safety Procedures," for laboratory safety procedures.

Procedure:

A. Setting up an ICP run

1. Obtain appropriate preprinted ICP run sheets (based on method and instrument) and record the initials, employee number, and date the run was set up.

2. Determine the batches to be analyzed.

   NOTE: An ICP run typically contains less than 70 tubes.

3. The runsheets should then be filled out with the sample name, class, initial volume (IV), final volume (FV), dilution factor (DF), batch number, and comments. The information can all be found on the Prep Batch Sheet.

   a. Sample names include:

      (1) PBW – Prep blank (water)

      (2) LCSW – Laboratory control sample (water)

      (3) LCSDW – Laboratory control sample duplicate (water)

      (4) PBS – Prep blank (solid)

      (5) LCSS – Laboratory control sample (solid)

      (6) LCSDS – Laboratory control sample duplicate (solid)

      (7) Lancaster Laboratories' sample number

      (8) CCV – Continuing calibration verification

      (9) CCB – Continuing calibration blank

      (10) CRI – Contract-required detection limit standard

      (11) ICSA – Interelement correction standard – A

      (12) ICSAB – Interelement correction standard - AB

(13) HS1, HS2, HS3, HS4 – High standard 1, 2, 3, or 4

NOTE: For definition of above, see SOP-IO-014, "Reviewing ICP Data for Acceptance," pages 2 through 4.

b. Initial volume (IV) – The sample aliquot digested.

c. Final volume (FV) – The final sample volume after digestion.

d. Dilution factor (DF) – The dilution factor of the sample prepared at the time of analysis. Needed to bring the sample into the linear range of the instrument, to negate a matrix effect, or for serial dilutions.

e. Batch No. – The batch number of the sample. By convention, the batch number is recorded opposite the first tube listed for the batch as well as the first line of each additional page.

f. Protocol – The protocol used to review the data for specific method requirements in the IAG database.

g. SDG – Sample delivery group number for data package samples.

h. Comments – Any description of the sample (from prep logs), status of the sample (i.e., RUSH, Promised), and chain-of-custody documentation needed, if any, should be recorded here. Reread elements are also recorded here. Lot numbers for standards must also be recorded here.

4. When setting up a run, Batch QC (i.e., PB, LCS, background, duplicate, spike, matrix spike duplicate, post-digest spike, and serial dilution) should be placed in the same block of ten or fewer samples. If there are two LCSs, they should be placed one after the other.

a. Following the regular standards on the run, high standards must be run with any SW-846 batch.

b. CCV/CCB must be run after every ten samples.

c. CRI, ICSA, ICSAB, CCV, CCB follow the standards (or high standards for SW-846) and must conclude each run. For CLP4.0, there must be a CRI, ICSA, ICSAB following every 20 samples.

d. Any deviations from protocol should be noted in the Comments Section of the cover page.

e. Any unused portion of the run must be "Z'd" out.

B. Pouring an ICP run

It is important to minimize any chance of contamination, both of yourself and the samples. Keep your hands and the area clean at all times.

1. Choose the appropriate run sheet for the instrument and protocol. Record on each page of the ICP runsheet: initials, employee number, and the date the run was poured.

2. Prepare and record any standards, check standards, and interference check standards needed to complete the pouring of the run.

   NOTE: See SOP-IO-007, Section C.

3. Determine the size of tubes needed for the run.

4. Obtain the appropriate number of tubes, number each tube, and place them in test tube racks.

5. Pour appropriate standards and initial run QC as labeled on the run sheet (approximately 4 mL for regular ICPs; approximately 5-mL for Trace ICPs).

   Any samples that require chain of custody may be taken out of the locked storage cabinet. The transfer should be documented on the chain-of-custody form with first initial, full last name, employee number, date and time (military). Samples must be in the analyst's possession until they are signed back into custody of locked storage. See SOP-QA-104, "Internal Chain-of-Custody Documentation."

6. Prepare and label the PDS required for each batch (sample volume permitting). A PDS is prepared using 0.1 mL of a custom ordered PDS solution into 4.9 mL of background sample. Record the lot number of the solution in the comments column of the ICP run sheet.

7. Prepare a serial dilution by diluting the background sample approximately 5×. If the background sample chosen for serial dilution has been diluted due to matrix interference or to bring the analyte concentration into the linear range of the instrument, the diluted sample must be diluted 5× (i.e., if Bkg = DF5, S.D. must = DF25).

8. Using Whatman No. 40 filter paper, filter those samples that are cloudy or contain particulate. If the filtrate remains cloudy, filter again. Samples with limited sample volume may be filtered using a 10-mL sterile disposable syringe fitted with a 0.45 µm PTFE syringe filter.

9. For any TCLP or SPLP sample that requires method of standard additions, obtain four 5-mL volumetric flasks and determine if you need LL#1 or LL#2 to spike with. Add 0, 0.05, 0.1, 0.15 mL of spiking solution respectively and fill to volume with sample. Mix well.

10. Pour each sample or sample filtrate into the appropriate tube. Usually, the order of the batch QC is PB, LCS, (LCSD), Bkg, PDS, DUP, MS, (MSD), SD.

11. Cover the run with plastic wrap to prevent the contamination of the samples.

12. Return samples to sample storage. If samples require chain of custody, return samples to locked storage and sign the COC.

NOTES:

1. A post-digest spike and a serial dilution will be performed on one sample in each digestion batch. Typically, the background sample is chosen. If the batch QC is split between two samples, the post-digest spike will be performed on the background sample accompanied by a matrix spike; the serial dilution will be performed on the background sample accompanied by a matrix duplicate. (For definition of batch QC, see SOP-IO-014, pages 1 and 2). If sample volume is limited, the duplicate may be used for the PDS and SD.

2. Air filter batches need only a serial dilution on one sample in the batch (a post-digest spike is not required).

3. "As Received" samples should be run with a blank and LCS (prepared by the analyst). These "batches" are recorded in the Dilute & Run Batch Book located in the ICP lab.

4. See MC-IO-002, "Operation of the Thermo Jarrell Ash ICAP™ 61," MC-IO-018, "Operation of the Thermo Jarrell Ash ICAP™ 61E Trace Analyzer Spectrometer," and INDBMS training notes for operation of the ICPs.

5. After the run has started, note the run number on the batch sheet, copy at 65% reduced size, file original in the batchbook, and write the batch number on the top of the copy in Sharpie to keep with the samples. Write the run number on any reread sheets and file in the reread book.

6. Documentation is of utmost importance. Double check all entries.

Revision Log:

Initiated Date: 11/09/93

Ver. #	Effective Date	Change
00	10/09/96	Previous issue
01	MAY 0 4 1999	Major changes are as follows: • Added Personnel Training and Qualifications section • Removed all references to ICAP™ 1100 • Figures updated • Clarifications made throughout this SOP

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040699

Prepared by: Jane B. Fellwock Date: 4-6-99

Approved by: Robert Stroetz Jr. Date: 4.8.99

Approved by: Dorothy McClave Date: 4/20/99

Figure 1

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601

Elemental Analysis by 61E Trace
Inductively Coupled Argon Plasma Spectrometry
EPA600/CLP

Run:_____T61/T62/T63/T64 Setup By:_____ Date:_____
Rinse Time: _____ sec. Poured By: _____ Date:_____
Analyst: _____ Date:_____

Tube	Sample	IV	FV	DF	Batch	Prot	SDG	Comments
1	S0							Lot #
2	S1							
3	S2							
4	S3							
5	ICV							
6	ICB							
7	CRI							
8	ICSA							
9	ICSAB							
10	CCV							
11	CCB							
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Figure 2

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Elemental Analysis by 61E Trace
Inductively Coupled Argon Plasma Spectrometry
SW-846

Run:_____T61/T62/T63/T64 Setup By:_____ Date:_____
Rinse Time: _____ sec. Poured By: _____ Date:_____
Analyst: _____ Date:_____

Tube	Sample	IV	FV	DF	Batch	Prot	SDG	Comments
1	S0							Lot #
2	S1							
3	S2							
4	S3							
5	HS1							
6	HS2							
7	HS3							
8	ICV							
9	ICB							
10	CRI							
11	ICSA							
12	ICSAB							
13	CCV							
14	CCB							
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Figure 3

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Elemental Analysis by 61E Trace
Inductively Coupled Argon Plasma Spectrometry
Multi-Point Calibration

Run:_____T61/T62/T63/T64 Setup By:_____ Date:_____
Rinse Time: _____ sec. Poured By: _____ Date:_____
Analyst: _____ Date:_____

Tube	Sample	IV	FV	DF	Batch	Prot	SDG	Comments
1	M0							Lot #
2	M1							
3	M2							
4	M3							
5	M4							
6	M5							
7	M6							
8	HS1							
9	HS2							
10	HS3							
11	ICV							
12	ICB							
13	CRI							
14	ICSA							
15	ICSAB							
16	CCV							
17	CCB							
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Figure 3 – Continued

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Elemental Analysis by 61E Trace
Inductively Coupled Argon Plasma Spectrometry

Run:_____T61/T62/T63/T64 Setup By:_____ Date:_____
Rinse Time: _____ sec. Poured By: _____ Date:_____
Analyst: _____ Date:_____

Tube	Sample	IV	FV	DF	Batch	Prot	SDG	Comments
32								
33								
34								
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Figure 3 - Continued

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Elemental Analysis by 61E Trace
Inductively Coupled Argon Plasma Spectrometry

Run:_____T61/T62/T63/T64 Setup By:_____ Date:_____
Rinse Time: _____ sec. Poured By: _____ Date:_____
Analyst: _____ Date:_____

Tube	Sample	IV	FV	DF	Batch	Prot	SDG	Comments
63								
64								
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Figure 4

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2425 New Holland Pike • Lancaster, PA 17601

Elemental Analysis by Inductively Coupled Argon Plasma Spectrometry

EPA600/CLP

Run: _____ I61 Setup By: _____ Date: _____
Rinse Time: _____ sec. Poured By: _____ Date: _____
Analyst: _____ Date: _____

Tube	Sample	IV	FV	DF	Batch	Prot	SDG	Comments
1	S0							Lot #
2	S1							
3	S2							
4	S3							
5	S4							
6	ICV							
7	ICB							
8	CRI							
9	ICSA							
10	ICSAB							
11	CCV							
12	CCB							
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Figure 5

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2425 New Holland Pike • Lancaster, PA 17601

Elemental Analysis by Inductively Coupled Argon Plasma Spectrometry

SW-846

Run: _____ I61 Setup By: _____ Date: _____

Rinse Time: _____ sec. Poured By: _____ Date: _____

Analyst: _____ Date: _____

Tube	Sample	IV	FV	DF	Batch	Prot	SDG	Comments
1	S0							Lot #
2	S1							
3	S2							
4	S3							
5	S4							
6	HS1							
7	HS2							
8	HS3							
9	HS4							
10	ICV							
11	ICB							
12	CRI							
13	ICSA							
14	ICSAB							
15	CCV							
16	CCB							
17								
18								
19								
20								
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23								
24								
25								
26								
27								
28								
29								
30								
31								

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Figure 5 - Continued

Lancaster Laboratories
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Elemental Analysis by Inductively Coupled Argon Plasma Spectrometry

Run: _____ I61 Setup By: _____ Date: _____
Rinse Time: _____ sec. Poured By: _____ Date: _____
Analyst: _____ Date: _____

Tube	Sample	IV	FV	DF	Batch	Prot	SDG	Comments
32								
33								
34								
35								
36								
37								
38								
39								
40								
41								
42								
43								
44								
45								
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60								
61								
62								

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Figure 5 – Continued

Lancaster Laboratories
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Elemental Analysis by Inductively Coupled Argon Plasma Spectrometry

Run: _____ I61 Setup By: _____ Date: _____
Rinse Time: _____ sec. Poured By: _____ Date: _____
Analyst: _____ Date: _____

Tube	Sample	IV	FV	DF	Batch	Prot	SDG	Comments
63								
64								
65								
66								
67								
68								
69								
70								
71								
72								
73								
74								
75								
76								
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90								
91								
92								
93								

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Figure 6

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Elemental Analysis by Inductively Coupled Argon Plasma Spectrometry

Multi Point Calibration

Run: _____ I61 Setup By: _____ Date: _____

Rinse Time: _____ sec. Poured By: _____ Date: _____

Analyst: _____ Date: _____

Tube	Sample	IV	FV	DF	Batch	Prot	SDG	Comments
1	M0							Lot #
2	M1							
3	M2							
4	M3							
5	M4							
6	M5							
7	M6							
8	M7							
9	HS1							
10	HS2							
11	HS3							
12	ICV							
13	ICB							
14	CRI							
15	ICSA							
16	ICSAB							
17	CCV							
18	CCB							
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								
31								

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Reviewing ICP Data for Acceptance

Reference:

1. ILM02.1, Section E., USEPA CLP Statement of Work, March 1990.
2. ILM04.0, Section E., USEPA CLP Statement of Work.
3. Method 6010A, USEPA SW-846, 7/92.
4. Method 6010B, USEPA SW-846, 12/96.
5. Method 200.7, USEPA 600/4-91/010.
6. Method 200.7, USEPA 600/R-94-111.

Purpose:

This SOP is designed to provide consistent guidelines for the evaluation of ICP data.

Scope:

This procedure applies to analyses performed in Environmental Sciences using ICP for identification and quantitation of metallic constituents.

Personnel Training and Qualifications:

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 the trainee's data by trainer.

5. Documentation of critical steps in the training process.

6. Demonstration of proficiency by being able to independently review ICP data.

Definition:

A. Batch QC samples

The following samples may be part of every digestion batch prepared depending on method requirements. When there is insufficient sample volume to perform any QC determinations, a note indicating this should be made on the run sheet.

1. Background Sample (U) - The original sample from which the batch QC is derived.

2. Duplicate Sample (D) - A replicate of the original sample, processed in parallel. This sample is used to provide a measure of the in-lab repeatability (precision) of the analytical process.

3. Matrix Spike Sample (R) - A replicate of the original sample spiked with a known amount of analyte. This sample is used to determine if there are any matrix effects that could influence analyte recovery during the digestion procedure.

4. Matrix Spike Duplicate (MSD) - A duplicate of the Matrix Spike Sample (R) which is a replicate of the original sample spiked with a known amount of analyte. This sample is used to determine if there are any matrix effects that could influence analyte recovery during the digestion procedure. It is also used as a measure of the precision of the analytical process.

5. Post Digestion Spike (PDS) - This sample is a spike of the Background Sample prepared after digestion, at the time of analysis. It is used to determine if low spike recoveries are due to problems in the digestion or are matrix related.

6. Laboratory Control Sample (LCS) - This is a matrix-matched synthetic sample of known composition. It is used to judge efficiency of the digestion procedure, as measured by the % recovery of the analytes.

7. Laboratory Control Sample Duplicate (LCSD) - This is a duplicate of the matrix-matched synthetic sample of known composition. It is used to judge efficiency of the digestion procedure, as measured by the % recovery of the analytes. It is also used as a measure of the precision of the analytical process.

8. Preparation Blank (PB) - This is a reagent blank carried through the entire digestion procedure. It is used to determine if contamination has occurred during the digestion procedure.

9. Serial Dilution (SD) - This sample is a 1:4 (5×) dilution of the Background Sample, prepared after the digestion. It is used to indicate the presence of any matrix effects that could cause a nonlinear response at the instrument.

B. Instrument run-time QC

The following samples are used to monitor the instrument stability during an analytical run:

1. Initial Calibration Verification (ICV) - This is a standard near the middle of the calibration range, prepared from a different source than the calibration standards. It is used to prove that the instrument is calibrated correctly at the start of the run.

2. Initial Calibration Blank (ICB) - This is a standard reagent blank, used to prove that the low end of the calibration is acceptable. It must be run immediately after the ICV.

3. Continuing Calibration Verification (CCV) - A mid-range standard run at a frequency of 10% (every ten samples) throughout the run. This is used to monitor instrument drift.

4. Continuing Calibration Blank (CCB) - A reagent blank run immediately after every CCV. This is used to monitor the stability of the low end of the calibration.

5. Interelement Correction Standard-A (ICSA) - A standard containing high concentrations of commonly interfering elements. It is used to assess the spectral interferences due to matrix elements that can normally be expected to be found in a sample. This standard, and its companion below, must be run at the beginning and end of every run or once every 8 hours.

6. Interelement Correction Standard-AB (ICSAB) - A standard containing both interfering elements and target analytes, run immediately after the ICSA. It is used to demonstrate the effectiveness of the interelement correction factors in use.

7. Contract Required Detection Limit Standard (CRI) - A low-level standard used to monitor the performance of the instrument near the detection limit. This standard must be run at the beginning and end of every run or every 8 hours.

8. High Standards (HS1, HS2, etc.) – Run on SW-846 runs only, these are the same standards used to calibrate the instrument. They are reanalyzed immediately following calibration.

Procedure:

A. Rounding

For all calculations, carry all digits until the final result is obtained. To round off a number to a specific number of digits, truncate the number to the desired number of digits and treat the excess as a fraction. Then:

1. If the fraction to be dropped is <½, round the last retained digit down.

2. If the fraction to be dropped is >½, round the last digit up.

3. If the fraction to be dropped is =½, round the last retained digit up only if it is odd, otherwise retain the last digit as it is.

B. Raw data quality checks

For each QC check, if an analyte is out of specification, indicate the error in the reread or redigest column next to the affected analyte on the raw data (see Table I for the proper codes).

1. Make sure that the run is correctly labeled, dated, and signed and that the corresponding cover sheet is attached to the front of the run.

2. For SW-846 runs, check the High Standards percent recovery. The acceptance range for High Standards is ±5%. If an analyte is not within specifications, no data from this run may be used.

3. Check the ICV and ICB.

   a. For EPA 600, Method 200.7 (NPDES and PW samples), the acceptance range for the ICV is ±5% of the true value. For all other work, the range is ±10%. Apply this test after the result has been rounded to the correct number of significant figures.

   If an analyte is not within specifications, no data from this run may be used.

   b. The absolute value of the ICB result must be <LOQ (SW-846 6010A, EPA 600), <CRDL (CLP), <client-specified LOQ, or <3× the IDL (SW-846 6010B) for the ICB to be acceptable.

   If an analyte is not within specifications, no data from this run may be used (except for SW-846 6010B where the following block of QC is evaluated per 6010B rules – see B.4.b.(2) below).

4. Check the CCV/CCB pairs.

   a. For EPA 600, Method 200.7 (NPDES), the acceptance range for the CCV is ±5%. For PW samples, the acceptance range for the CCV is ±10%. For all other work, the acceptance range is ±10%. Apply this test after the result has been rounded to the correct number of significant figures. For SW-846, Method 6010B, the relative standard deviation (RSD) on the CCV must be <5%.

      If an analyte is not within specifications, no data from the block immediately prior to or following the CCV may be used (except for CLP — no data from the entire run may be used).

   b. The absolute value of the CCB result must be <LOQ (SW-846 6010A, EPA 600), <CRDL (CLP), <client-specified LOQ, or <3× the IDL (SW-846 6010B) to be acceptable.

      If an analyte is not within specifications, apply the following corrective action:

      (1) SW-846 6010A/EPA 600 samples - No data from the block of samples immediately prior to or following the CCB may be used.

      (2) For SW-846 6010B, if the CCB is >3× IDL, then any sample <10× CCB reading and >LOQ would need to be reanalyzed.

      (3) CLP samples - No data from the run may be used.

5. Check the ICSA and ICSAB.

   a. Examine only the interferents in the ICSA. Concentrations must fall within ±20% of the true values. For CLP 4.0, the target analytes whose CRDLs are ≤10 ppb must read ≤2× CRDL (Ag, As, Be, Cd, Cr, Pb, Se, Tl).

   b. Examine the target analytes in the ICSAB. All concentrations must fall within ±20% of the true values.

   c. If any interferent is out of spec in the ICSAB, then any element that it interferes with will also need to be reanalyzed.

   d. The surrounding CCVs and CCBs must also be within specification. SW-846 6010B rules apply if a CCB (or ICB) is >3× IDL.

   If an analyte is not in-spec, no data from this run may be used.

6. Check the CRI. The results should fall within ±50% of the true value. Certain clients require compliance to ±20% of the true value. Other than specific client quality assurance plans, no action is required if the requirements are not met. However, continued failure to meet the criteria should be investigated and corrected.

7. Check the LCS, LCSD, and Preparation Blank (PB).

   a. LCS and LCSD criteria - All analytes must recover within ±20.0% of the theoretical value (or the statistical window for the solid LCS). For EPA 600, the LCS and LCSD must recover within ±15% of the theoretical value. Also check the duplicate precision between the LCS and LCSD using Section 8.a.

b. Prep Blank criteria - The absolute value of the concentration of the analyte must be less than the applicable detection limit. For all sample results, correct the raw concentration for sample weight and dilution before performing this test as follows:

Solids:

$$\text{Final Conc } (mg/kg) = \frac{\text{ICP Reading } (mg/L) \times \text{Final Vol } (mL)}{\text{Sample Weight } (g)} \times \text{Instrument Dilution Factor}$$

Waters:

$$\text{Final Conc } (mg/L) = \frac{\text{ICP Reading } (mg/L) \times \text{Final Prep Vol } (mL)}{\text{Initial Prep Vol } (mL)} \times \text{Instrument Dilution Factor}$$

For CLP groups, the EPA-CLP CRDL governs; for SW-846 and EPA 600 groups, the applicable LOQ should be used. If an analyte does not meet this requirement, it must be redigested. Exceptions are:

(1) If the sample true concentration is >10× (CLP and EPA 600) or >20× (SW-846) prep blank concentration, redigestion is not required.

(2) If the sample true concentration is less than the detection limit, redigestion is not required (unless requested by the client).

8. Check the duplicate precision.

a. Acceptable precision is measured as relative percent difference, calculated as follows:

$$RPD = \frac{(U - D)}{(U + D) \div 2} \times 100$$

If the samples are >5× the appropriate LOQ, then the RPD should be <20%. If either the sample or duplicate are <5× the LOQ, then the absolute difference between the two values should be <LOQ. If both values are less than the LOQ, then no criteria is applicable.

b. If >50% of the analytes are outside of the acceptable range, a sampling error should be suspected. For SW-846 and EPA 600 sample groups, the U, D, and R should be redigested.

9. Check the spike and the matrix spike duplicate recoveries. Also check the duplicate precision between the spike and the matrix spike duplicate using Section 8.a.

a. Recovery is calculated as follows:

$$\%Recovery = \frac{(SSR - SR)}{SA} \times 100$$

Where SSR is the Spiked Sample Result, SR is the Sample Result, and SA is the Spike Added. If the measured sample concentration is less than the appropriate LOQ, use 0 for the calculation. Recovery should fall within 100 ± 25% for CLP and SW-846, Method 6010B. For SW-846 6010A and EPA 600 (NPDES), the recovery should fall within 100 ± 20%. For EPA 600 (PW), the recovery should fall within 100 ± 30%. Specific clients may designate other limits.

b. If the spike recovery is outside the acceptable range, and the sample concentration does not exceed 4× the spike concentration, a Post Digestion Spike (PDS) must be performed (exception: Ag for CLP samples). PDS recoveries are calculated as follows:

$$\%Recovery = \frac{(SSR - SR)}{SA} \times 100$$

Where:

SSR = Spike sample reading

SR = Sample reading

SA = Concentration of spike added

Recovery of the PDS should be within 100 ± 25% for CLP, EPA 600 (NPDES), and SW-846. The PDS should be within 100 ± 15% for EPA 600 (PW). No corrective action is required.

An out-of-specification matrix spike and PDS would indicate interference due to the sample matrix.

10. Check the serial dilution recovery.

If the analyte concentration is >50× the IDL, then the % difference should be within <10% (except for EPA 600 [NPDES] which requires <5% difference), calculated as follows:

$$\%Difference = \frac{(5 \times SDR) - SR}{SR} \times 100$$

Where SDR is the serial dilution result and SR is the sample result.

C. Raw data sample checks

1. Starting with the first sample, note any analytes that must be rejected for failing ICV/CCV, ICB/CCB, LCS/LCSD/PB, ICSA/ICSAB. Mark these for reread/redigest as appropriate in the proper column on the raw data.

2. Check to make sure that all results are below the linear range limit. If a sample reading is above the linear range, then reread the sample at an appropriate dilution. (For specific clients, this check requires sample values to be less than the high standard in the calibration.)

3. Check that the **absolute** value of all nondetected analytes is less than the LOQ. A technical decision must be made as to whether a reread is warranted for readings <(-LOQ).

4. Check for carryover between samples. Some clues are:

   a. A high result or overrange for an analyte in a sample, immediately followed by a sample with a low concentration of the same analyte. Boron is particularly subject to carryover effects.

   b. Sample RSD >20%, with the concentrations decreasing from first integration to last.

   Flag any suspect samples for reread.

5. For TCLP and SPLP samples, an MSA (method of standard additions) is required if the sample concentration falls between 80% to 100% of the regulatory limits established in the March 29, 1990, *Federal Register*.

6. For all EW samples (samples from public drinking water sources), check the results against the MCL (maximum contaminant level). If an analyte **exceeds** the MCL, notify a verifier at once so that the supplier can be notified. Suppliers must be notified within 1 hour.

Analyte	MCL (mg/L)
Sb	0.006
As	0.05
Ba	2 (1)**
Be	0.004

Analyte	MCL (mg/L)
Cd	0.005
Cr	0.1 (0.05)**
Se	0.05 (0.01)**
Tl	0.002
Al*	0.2
Cu*	1.0
Fe*	0.3
Mn*	0.05
Ag*	0.1 (0.05)**
Zn*	5.0

* Secondary regulated contaminants

** The federal MCLs for these analytes are greater than Pennsylvania MCLs. The numbers in parentheses are the MCLs effective in Pennsylvania

7. For Trace ICP runs, check the internal standard (yttrium) level for the entire run. If it is <80% or >120% of the value in S0 for any sample, then a technical decision must be made as to whether a reread is needed or not.

D. When complete, check the following:

1. The beginning and end of the raw data are signed and dated by the reviewer.

2. All samples requiring reread/redigestion are listed on the reread/redigestion schedule forms.

3. Reread/redigest request forms are clipped to the front of the run.

4. The data are uploaded to the Wang via INDBMS.

5. The raw data packet is placed in the verification bin. (For samples following Good Laboratory Practices [GLP], the raw data includes the "real-time" printout, as well as the final print file.)

Revision Log:

Initiated Date: 03/06/92

Ver. #	Effective Date	Change
00	09/04/96	Previous Issue

01	01/30/98	Major changes are as follows:
		• References added for SW-846 6010B and EPA 600 200.7/R-94-111
		• Added definitions for SIC and LRL solutions
		• The following throughout the Procedure section:
		  • Added 6010B criteria for ICB, CCV, and CCB
		  • CRI and LRL check added
		  • Modified EPA 600 criteria for the LCS
		  • Added EPA 600 criteria for MS, PDS, and SD
		  • Added GLP "real-time" printout requirement.
		  • Applied changes to Table I

02	FEB 0 4 1999	Major changes are as follows: • Added Personnel Training and Qualifications section • Removed references to LRL and SIC solutions which are no longer used (sections B.8, B.9 in Definitions section, and B.5, B.6 in Procedure section) • Added internal standard check (C.7) • Added 6010B rule for evaluating samples is CCB is >3× IDL (B.4.b.(2))

Prepared by: Jane B. Jellwild Date: 1-19-99

Approved by: Robert Stocks Date: 1.26.99

Approved by: Dorothy M Love Date: 2/3/99

Table I

	Reread	Redigest
A	ICV out - (100 ± 10% for SW-846; CLP) (100 ± 5% for EPA 600)	1 Prep blank out - |PB| ≥LOQ or CRDL
B	ICB out - |ICB| ≥LOQ (SW-846, 6010A, EPA 600) |ICB| ≥3× IDL (SW-846, 6010B)	2 LCS out - (100 ± 20% SW-846, CLP) (100 ± 15% EPA 600) (statistical window for solids)
C	CCV out - (100 ± 10% for SW-846; CLP, EPA 600 [PW]) (100 ± 5% for EPA 600 [NPDES])	3 Out of digestion solution
D	CCB out - |CCB| ≥LOQ (SW846, 6010A, EPA 600) |CCB| ≥3× IDL (SW-846, 6010B) |CCB| ≥CRDL (CLP)	4 Digest error
E	ICS out - (100 ± 20%) (As, Be, Cd, Cr, PB, Se, Ag, Tl >±2× CRDL in ICSA for CLP4.0)	
F	|Sample Reading| <-LOQ	
G	>Linear range	
H	High RSD	
I	Total < Soluble	
J	Leachate (MSA required)	
K	Instrument failure	
L	Sample Handling	
M	High standard out (100 ± 5%)	
N	Saturation of an affecting element	

Thermo Spec Errors

C	Over calibration range
S	Saturation
K	Affected by a saturated element

Appendix I

Definitions and explanations of the codes and symbols used on the raw data. Each heading listed below corresponds to an area labeled in Figure 1.

A. Sample table information

1. The run number
2. The page number
3. The tube number
4. The sample number
5. The first and second asterisks denote whether the sample is a background (U*), duplicate (D*), spike (R*), MSD (M*), post-digestion spike (UP), serial dilution (UL), or not a QC sample (**).
6. The weight to volume or volume to volume digestion ratio - The first number is the sample amount, the second number is the final digest volume.
7. The dilution factor - If the digest solution was diluted prior to analysis, the factor is listed here. An undiluted sample is labeled DF1.
8. Digestion batch number - Set at the time of the digestion, this number is used to track samples and QC prepared together.
9. The protocol by which the data should be reviewed (CLP, SW-846, EPA-600, etc.)

The above information (Items A.4. to A.9.) is entered into the sample table by the analyst prior to the analysis.

10. Date and time of the sample injection into the instrument
11. The ICAP identification number

Appendix I (Continued)

B. The ICP scans all of the elements listed simultaneously during the analysis. The blanks may contain either an "X" or a "√" The X means that the value was reported to the client. The √ is used for quality control samples when the value for an element is used for QC purposes, but not reported to the client because the client did not request that determination. These notations are made by the verifiers.

C. Three error codes preset in the Thermospec software may appear here.

1. S = Saturation - The concentration of the element is greater than the photomultiplier tubes capacity to read it.

2. K = The Elements Affected by a Saturated Element - The concentration listed is not accurate, and a more accurate result can be obtained by running the sample at a dilution.

3. C = Over Calibration Range - The reading of the solution is above the calibration range.

D. The concentration of each element in mg/L is listed here. The result is the average of three integrations.

E. This section shows the individual readings in mg/L plus the %RSD of the three trials. If the RSD is >20% for readings >5 times the detection limit, the samples must be reanalyzed.

F. If a sample must be reanalyzed, a letter will be put next to the element to be reanalyzed. A key with the definition can be found in Table I.

G. If a sample must be redigested, a number is put next to the element to be reanalyzed. The definition of each number is also listed in Table I.

H. Comment Section - Any additional information about the sample or data is noted here.

Figure 1

2. Page: 34

A.

LANCASTER LABORATORIES

1. Run Name: 9602001I11 II. INSTRUMENT ID: 02360

3. Tube: 33 4.2445236 ID: 01/20/96 10:31

5. ****, 50-50, DF1, 960175705005, 3, ,

B. ELEM	C.	D. AVG (ppm)	%RSD	E. INTEGRATIONS #1	#2	#3	F. REREAD	G. REDIG
Ag	k	-0.01604	6.856	-0.01668	-0.01477	-0.01668	N	
Al	k	0.14798	1.943	0.15106	0.14749	0.14537		
As	k	-0.01093	40.31	-0.00839	-0.00838	-0.01602	N	
B	k	2.01667	1.602	2.03599	1.97936	2.03466		
Ba		0.70715	2.080	0.71565	0.69016	0.71565		
Be	k	-0.00026	1.733	-0.00025	-0.00026	-0.00026		
Ca	S	6742.28515	0.000	6742.24169	6742.33251	6742.28076		
Cd		1.00071	1.699	1.01102	0.98107	1.01003		
Co	k	0.03351	14.33	0.03107	0.03904	0.03041		
Cr	k	0.00645	38.19	0.00505	0.00501	0.00930	N	
Cu		1.10923	1.898	1.12196	1.08492	1.12082		
Fe	k	-0.00158	142.5	-0.00085	-0.00412	0.00022		
K	k	108.07760	1.725	109.21997	105.92595	109.08689		
Li		0.12033	2.112	0.12179	0.11739	0.12179		
Mg	k	63.80169	1.599	64.25976	62.63227	64.51304		
Mn	k	4.75433	1.524	4.79001	4.67091	4.80207		
Mo		0.11379	2.598	0.11550	0.11038	0.11550		
Na		190.12321	1.922	191.76280	185.93487	192.67198		
Ni	k	0.20465	1.806	0.20868	0.20142	0.20385		
Pb	k	0.83024	2.541	0.82929	0.80963	0.85180	N	
Sb	k	0.29231	4.260	0.30171	0.29703	0.27819		
Se	k	0.02942	119.6	0.05936	0.03826	-0.00935	N	
Si	k	2.56531	1.059	2.57137	2.53561	2.58897		
Sn	k	0.01599	106.6	0.00793	0.00445	0.03558		
Sr		3.75866	1.967	3.80230	3.67329	3.80039		
Ti	k	-0.00677	20.90	-0.00523	-0.00705	-0.00802		
Tl	k	0.14496	29.40	0.12719	0.11410	0.19360		
V	k	0.00501	38.33	0.00279	0.00611	0.00614		
Zn	k	55.65993	1.024	55.86583	55.01519	56.09877		

11

Ca saturation - Reread @ DF10

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601

Procedural Amendment #1

Number: SOP-IO-014

Title: Reviewing ICP Data for Acceptance

Effective Date (listed on procedure): 02/04/99

Section(s) affected by change: Procedure B.5.a. and b.

Reason for addition(s) or change(s): Client requirement

Change will be effective from (date): 02/04/99

Samples or project affected: All

List change(s) or addition(s) (specify which section):

Last sentence added to a. and b. as follows:

Procedure:

5. Check the ICSA and ICSAB.
   a. Examine only the interferents in the ICSA. Concentrations must fall within ±20% of the true values. For CLP 4.0, the target analytes whose CRDLs are ≤10 ppb must read ≤2× CRDL (Ag, As, Be, Cd, Cr, Pb, Se, Tl). For clients that require it, check that the elements not present in the ICSA are <LOQ.
   b. Examine the target analytes in the ICSAB. All concentrations must fall within ±20% of the true values. For clients that require it, check that the elements not present in the ICSAB are <LOQ.

SOPIO014.DOC
021999

Prepared by: Jane Jellerick Date: 2-19-99

Approved by: Robert Steudl Date: 2.19.99

Approved by: Dorothy M Love Date: 2/24/99

Mercury by Cold Vapor Generation

Reference:

1. Method 7470A (waters) and 7471A (solids), *Test Methods for Evaluating Solid Waste*, USEPA SW-846, September 1994. **(modified)**

2. Method 245.1 (waters) and 245.5 (solids), USEPA CLP SOW No. ILM04.0, Exhibit D, CLP-M, D47-59. **(modified)**

3. Method 245.1, *Methods for Analysis of Water and Wastes*, USEPA 600/4-79-020, March 1979.

Reference Modification:

SW-846 Methods 7470A and 7471A are manual procedures. This SOP is for an automated determination. The chemistries used to do the mercury determinations are the same. No impact on the quality of the data generated using this modification has been observed.

Purpose:

The purpose of this procedure is to detail the instrument parameters used to analyze mercury by cold vapor generation.

Background Information:

The optimum concentration range for this method is 0.2 to 5.0 ppb. The instrument detection limit for the method using the Leeman Labs PS200 and PS200II is 0.01 ppb. The following limits of quantitation are used in accordance with the requirements of the governing regulatory agency.

Limits of Quantitation:

	CRDL	Reference Method
CLP WW	0.2 µg/L	CLP SOW No. ILMO4.0 245.1 CLP-M
CLP SW	0.1 mg/kg	CLP SOW No. ILMO4.0 245.5 CLP-M

	LOQ	Reference Method
Waters	0.2 µg/L	EPA SW-846, Method 7470A
Solids	0.1 mg/kg	EPA SW-846, Method 7471A
PW/EW	0.2 µg/L	EPA 600, Method 245.1
NPDES	0.2 µg/L	EPA 600, Method 245.1

Scope:

This procedure is applicable to the determination of mercury in waters, wastewaters, and leachates (#0259) and soils (#0159).

Basic Principles:

The reaction for the mercury analysis is a simple reduction reaction. The mercury (Hg^{++}) is reduced with stannous chloride (Sn^{++}) to liberate mercury metal and Sn^{+4}. The sample is made acidic with hydrochloric acid to maintain the reducing environment of the reaction. Air or inert gas is used to sweep the volatile mercury into the absorption cell in the optical path of the atomic absorption spectrophotometer.

Reagents:

(Use the following or equivalent.)

1. Nitric acid, 70.0% to 71.0% HNO_3, Baker Instra-Analyzed reagent, 1.428 g/mL; store at room temperature

2. Sulfuric acid, 95.0% to 98.0%, H_2SO_4, 36 N, Fisher reagent, ACS, 1.84 g/mL; store at room temperature

3. Potassium permanganate, $KMnO_4$, Baker Analyzed reagent, ACS

4. Potassium persulfate, 5% $K_2S_2O_8$ Baker Instra-Analyzed reagent, ACS

5. Sodium chloride, NaCl, Fisher, Certified ACS

6. Hydroxylamine hydrochloride, $NH_2OH \cdot HCl$, Fisher, Certified ACS

7. Deionized water, Type 2 or better

8. Stannous chloride solution, 10% SnCl, Baker Analyzed reagent, ACS

9. Hydrochloric acid, HCl, 36.5% to 38.0%, Baker Instra-Analyzed reagent, 1.194 g/mL or equivalent

Safety Precautions:

Refer to SOP-IO-011, "Inorganic Analysis Safety Procedures."

Personnel Training and Qualifications:

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:

Please consult SOP-IO-001, "Preservation and Holding Times for Inorganics Analyses," regarding sample preservation, holding times, and storage conditions. Consult Analysis #0821, 5713, 5714, 5711, 1578, and 0494, for digestion procedures.

1. Preparation of standard solutions

   For detailed procedures on preparation of standard solutions consult SOP-IO-007, "Preparation of Standards and Solutions," Sections E and H.

2. Instrument setup

   For detailed procedures of the Leeman Labs PS200 or PS200II automated mercury analyzer, please refer to MC-IO-014, "Vapor Generation for Cold Vapor Mercury Method Using the Leeman Labs PS200."

3. Program parameters

Leeman Labs PS200 or PS200II

Mercury analyzed using the Leeman Labs PS200 or PS200II - The instrument parameters are preset for the program that was developed for mercury. The parameters for this program are as follows:

	Hg
Instrument mode	Intensity
Calibration mode	Concentration
Sample introduction	Automated
Integration time (sec)	10
Replicates	1

Calibration Standards (µg/L)	Controls	(µg/L)
0.2	ICV	2.0
0.5	CCV	1.0
1.0	CRA	0.2
2.5		
5.0		

NOTE: CCV CONCENTRATION SHOULD BE LESS THAN OR EQUAL THE MAXIMUM CONTAMINATION LEVEL OF 2 µg/L FOR POTABLE (PW) AND DRINKING (EW) WATERS.

NOTE: The concentration values and instrument conditions are for Leeman Labs PS200 or PS200II. All changes from the standard analytical program will be documented on the raw data.

Quality Assurance:

Please consult SOP-IO-005, "Department 23 Quality Control Procedures," for specific QC protocol and procedures.

Calculations:

Please consult SOP-IO-012, "Calculations Used by the Inorganics Group," for calculation procedures.

Revision Log:

Initiated Date: 11/23/97

Ver. #	Effective Date	Change
00	01/18/96	Previous Issue
01	MAY 0 6 1999	Major changes are as follows:

- Removed reference note to add a Reference Modification section
- Changed Background Information, Procedure, and Quality Assurance section to remove references to old instrumentation
- Added reagents to Reagents section
- Added Personnel Training and Qualifications section

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Analysis #0259, 0159
Revision 01
Supersedes Date: 01/18/96
Effective Date: MAY 0 6 1999

Prepared by: _____ Date: 4/20/99

Approved by: _____ Date: 4.21.99

Approved by: _____ Date: 4/22/99

MC-IO-014
Initiated Date: 02/04/94
Effective Date: OCT 0 5 1995

Vapor Generation for Cold Vapor Mercury Method Using the Leeman Labs PS200

Reference:

The method described in this maintenance and calibration is adapted from the following references.

1. Method 7470A (waters) and 7471A (solids), *Test Methods for Evaluating Solid Waste*, USEPA SW-846, modified, September 1994.

2. Method 245.1 (waters) and 245.5 (solids), USEPA CLP SOW No. ILM04.0, Exhibit D, CLP-M, D47-59, modified.

3. Method 245.1, *Methods for Analysis of Water and Wastes*, USEPA 600/4-79-020, March 1979.

4. Leeman Labs suggested operation and maintenance.

Purpose:

The purpose of this procedure is to outline the proper techniques for the operation, calibration and maintenance of the Leeman Labs, PS200 Automated Mercury Analyzer.

Scope:

The Leeman Labs PS200 Automated Mercury Analyzer utilizes continuous flow technology with drying of the sample vapor for the analysis of mercury by automated vapor generation. The dry vapor enters one path of a heated double path optical cell which has been optimized for fast response (small diameter) and

sensitivity (long length). Mercury is measured using a solid state detector with a wide dynamic range and a mercury source which delivers a stable source of emission at 254 nm. The signal is referenced to the simultaneous absorbance of the pure carrier gas flowing through the second optical path under identical conditions. This procedure offers advantages in sensitivity and precision over conventional CV-AAS systems by a factor of approximately 10 times.

This procedure is applicable to the determination of mercury in waters, wastewaters, leachates, and soils by vapor generation.

Standard and Reagent Preparation:

Refer to SOP-IO-007, "Preparation of Standards and Solutions."

Safety Precautions:

Refer to SOP-IO-011, "Inorganic Analysis Safety Procedures."

Procedure:

A. Instrument setup

1. Leeman Labs Automated Mercury Analyzer

The Leeman Labs PS software has been set up using "hotkeys" to help with moving around in the software and macro utilization. (Macros are small programs used to operate the instrument.) The hotkeys are the capitalized letters which are highlighted in red on the computer screen.

a. System conditioning

If the instrument and computer are off, turn on the power to the instrument (green button). Also turn on the power to the computer and printer. Once the computer has initialized and a C:\ICP\ prompt is obtained, type *PS* and press ENTER. This will initialize the PS instrument software. The system is now ready for the initial setup.

If the instrument was shut down completely, the optical cell must be reconnected. To reconnect the optical cell, remove the front cover from the instrument by sliding the cover up and lightly pulling it out. Remove the two screws from the retaining brackets and carefully slide the cell out of its holder. Connect the gas lines on the left side of the cell to their respective connections. Check that all connections are secure for the gas, lamp, detector, and cell heater. Place the cell in its holder and tighten the retaining brackets to hold the cell in place. Replace the front cover to the instrument.

Turn on the power to the mercury lamp (blue button). The lamp will take approximately 20 minutes to stabilize. Press F10 to stop and exit any previously running macro. Press F1 to go to the Main menu. Use hotkeys by typing *U* to select Utilities and then by typing *G* to select diaGnostics. Using the down arrow, move the cursor to Tip Home and press ENTER. This will move the autosampler tip to the rinse position and raise the tip out of the rinse tank. Remove the sample trays and the rinse tank from the autosampler. Rinse the rinse tank with deionized water several times and refill the rinse tank with a 1% nitric acid solution (1% HNO_3).

Change the drying tube (located below the liquid gas separator) with a fresh, loosely packed tube from the prep room desiccator by unscrewing the gray lock nuts, removing the old tube, and replacing it with the fresh tube. Tighten the lock nuts and pull lightly on them to ensure a snug fit. If a seal is not observed, remove the O-rings from the lock nuts and rinse them with deionized water. Replace the drying tube in its holder on the instrument.

NOTE: It is very important that the tube is packed loosely so that the carrier gas can flow freely through the quartz wool and magnesium perchlorate.

Replace the rinse tank and sample racks on the autosampler. Press F1 for Menu, then type *I* for Instrument, *O* for Operations, and then *T* for Tip to Rinse. This will lower the autosampler tip into the rinse tank. Check the pump tubing for excessive wear by flipping the locking hub down and lifting up on the pump cassette. Visually check the tubing for excessive wear. Under normal daily operation, the tubing should last approximately 1 week. To replace the tubing, disconnect old tubing and discard and obtain a new set of tubing from the parts drawer. All tubing should be replaced together. The pump tubing is color coded for easy replacement. The sample tubing is black with a blue tab, the reductant tubing is clear with a red, orange, or yellow tab (0.16 cc/m), and the two waste tubes are clear with a gray tab (1.00 cc/m). The tubes are placed in separate cassettes with the sample tube closest to the instrument, followed by the reductant tube and finally the two waste tubes. To secure the tubing to the pump head, slide the tubing through the plastic clips at the bottom of the cassette until the tab is secure. Hold the tube taut (not stretched) and slide the loaded cassette onto the pump head. Lock the clamp by pulling the locking hub up. Repeat for the remaining tubes and connect the ends to the respective connections. For new tubing the "coldstrt" macro should be used.

Once the above initial system checks are complete there are two system conditioning procedures for the instrument. If the instrument has been shut down completely, usually over a long weekend or if new tubing is used, press F2 for Macro, type *coldstrt*, and press ENTER. The system will run approximately 2 hours, conditioning the pump tubing, lamp, and cell. When the macro is finished it will beep and display "Operation Complete" in red flashing letters at the bottom of the computer screen. The system is now ready to be optimized for automated analysis. If the instrument is in the overnight mode, used when daily operation is necessary or when tubing has previously been conditioned, press F2 for Macro, type *warmstrt*, and press ENTER. The system will run approximately 15 minutes, conditioning the pump tubing, lamp, and cell. When

finished, it will beep and display "System Ready" in red flashing letters at the bottom of the computer screen. The system is now ready to be optimized for automated analysis.

b. Optimization

Once the system is conditioned the cell must be optimized. Remove the front cover of the instrument and the large hex wrench from the lid of the cover. In the computer software, press F1 to go to the Main menu. Select Utilities and then diaGnostics by typing *U* and then *G*. Using the down arrow, move the cursor to Aper Test and press ENTER. A correctly optimized system value is 0. The value displayed on the screen should be ±100. If adjustments are required, turn the lower hex screw clockwise ⅛-turn for a positive adjustment and counterclockwise ⅛-turn if a negative adjustment is required. After the adjustment is done, press ENTER to recheck the value. Adjust the aperture until the value is between ±100. Replace the hex wrench and instrument cover.

c. Run setup

Once the cell has been optimized, the instrument is ready for sample analysis. Press F1 for Menu, type *P* for Protocol, and *G* for Get. It will ask you for a protocol name. The protocols have been set up for those operating the instrument as initials-employee number (e.g., JKG-201). A default protocol has been set up as TEST. Type in the protocol name and press ENTER. It will prompt you for a folder name. The folder name is the file where the run information will be stored. Type in the folder name as follows and press ENTER: year, day, run number (e.g., 9403201 for the first run of day 032 of 1994). Press F1 to return to the Main menu.

d. Autosampler setup

To set up the autosampler, type *A* for Autosampler and *R* for Rack Entry. It will prompt you for a rack name. There are two sample racks called stations, which hold 44 samples each. The standards and check standards are located on their own rack. If less than 44 samples are to be used, only one station is required to be defined. Enter a distinctive rack name such as the day number and 01 or 02 and press ENTER. The INSERT key is used to move between the entry modes. The three entry modes are single cells down ("cell entry"), single cell across ("row entry"), and full column ("column entry"). The current entry mode is displayed at the bottom right corner of the computer screen. In cell entry mode, move the cursor to the cup ID column using the arrow key. Enter the labels, up to 10 characters, and press ENTER. Enter PBW or PBS for blanks, LCSW or LCSS for lab controls, and sample numbers followed by a space then U, D, R, or M for background, duplicate, spike, or matrix spike duplicate, respectively. Once the sample labels have been entered, the extended ID must be entered. The extended ID includes the initial weights or volumes and batch numbers. In the column entry mode, type *R* for Range and enter the range of samples for the first batch. Type *E* for Extended ID (the full range which was selected will be highlighted). Enter the most common initial weight or volume followed by a space and then the full batch number and press ENTER (e.g., 8ml 940250821001). To edit values in either sample or extended ID columns, select either cell or row entry mode and move the cursor to the cell to be edited. Delete the value to be changed, retype the corrected value and press ENTER. Once the entries for all samples and batches are complete, return to the Main menu by pressing F1. The defined sample rack must be loaded into the autosampler setup to be run. Type *A* for Autosampler, *S* for Setup, and *1* for Station Rack Number 1 or *2* for Station Rack Number 2. You will be prompted for a rack name, enter the rack name which corresponds to the sample IDs you will be running. Enter the beginning cup position and the end cup position. Return to the Main menu by pressing F1. Load the standards, check standards, and samples into the appropriate locations (see Figure 1.).

NOTE: When the Leeman autoanalyzer is used in conjunction with the Leeman Labs Automated Preparation System AP200, rack entry occurs at the prep station. To transfer the file from the AP200 to the PS200, type *A* for Autosampler, *M* for rack Maintenance, and *C* for Copy on the prep station PC. Type the rack name to be copied and press ENTER. Insert a diskette into drive A: of the AP200 computer and type *A:* with the rack name and press ENTER. Once the file has been copied, remove the diskette from the AP200 and place it in drive A: of the PS200. At the PS200, press F1 for Menu, then type *A* for Autosampler, *M* for rack Maintenance, and *P* for Path. Type *A:* and press ENTER. Use the copy routine to copy the rack to the C:\ICP directory. Change the path of the PS200 back to C:\ICP. Enter the rack and beginning and end cups as previously described into the Autosampler Setup of the PS200.

2. Analysis of samples

The instrument is now ready for automated analysis. To start the run, press F2 for Macro. You will be prompted at the top of the computer screen for a macro name. Type *CLPHG* and press ENTER. The instrument will go through several checks and setups before starting a calibration. The CLPHG macro has been developed to check the correlation coefficient of the curve, run appropriate check standards at proper intervals, and check the percent recoveries of the calibration check samples. If, for any reason, the checks fall outside the windows required, a recalibration and reread of any associated samples and check samples in the bad blocks will automatically be performed. Recalibration due to failure will repeat three times. If any of the checks fail after the third recalibration, the instrument will stop and an error message will be displayed.

Detailed instructions for the complete instrument setup are found in the *Leeman Labs PS200 Automated Mercury Analyzer Manual*, beginning with Section 3.0.

3. Instrument shutdown and cleanup

   a. Overnight shutdown

      (1) Press F10 to stop and exit any running macro.

      (2) Remove cap from the reductant bottle and place on rim of the rinse tank. Cap the reductant bottle with spare lid.

      (3) Press F2 for Macro, type *overnight*, and press ENTER.

      (4) Turn off the power to the lamp.

      (5) Remove all standard and sample tubes and place them on the washroom cart to be cleaned.

      (6) Clean up any spills which may have occurred during sample pouring or analysis.

   b. Long-term shutdown (more than three days of no operation)

      (1) Press F10 to stop and exit any running macro.

      (2) Remove the rinse tank by following the steps for system conditioning. Rinse the tank with deionized water several times and fill it with deionized water. Replace the rinse tank on the autosampler.

      (3) Remove cap from the reductant bottle and place it on rim of the rinse tank. Cap the reductant bottle with spare lid.

      (4) Turn off the power to the lamp.

      (5) Press F2 for Macro, type *shutdown*, and press ENTER. The system will flush its lines with deionized water for several minutes.

(6) When finished, you will hear a beep and the message "Disconnect optical cell" will appear at the bottom of the computer screen. Disconnect the optical cell by removing the front cover of the instrument. Loosen the screws on the retaining brackets of the optical cell and gently slide the optical cell out of its holder. Disconnect the two gas lines on the left side of the cell and leave them hanging. Replace the optical cell in its holder and lightly tighten the retaining brackets. Replace the front cover of the instrument.

(7) Shut off the power to the computer, printer, and the PS200 instrument.

(8) Remove all standard and sample tubes and place them on the washroom cart to be cleaned.

(9) Clean up any spills which may have occurred during sample pouring or analysis.

4. Maintenance

a. To clean and rejuvenate the drying tube, remove the quartz wool from the ends of the used drying tube and empty the magnesium perchlorate into a beaker. Dissolve the magnesium perchlorate in water and discard this solution as liquid waste in the acid waste carboy located in the washroom. Clean the drying tube by rinsing it with tap water and placing it in a sample rack to dry. Once the drying tube is dry, use a small piece of quartz wool to plug one end of the drying tube. Fill the tube with coarse magnesium perchlorate and plug the other end with the quartz wool. Place the freshly packed drying tube in the desiccator in the prep room.

b. The pump tubing should be replaced weekly under normal daily usage.

c. Lightly spray the pump head weekly with a lubricant.

d. Lubricate the autosampler weekly by placing several drops of a light oil onto a towelette and wiping down the rails.

e. On a monthly basis, check the optical cell and windows, and if needed, clean the optical cell with a soapy solution (one drop of liquid Ivory to 500 ml deionized water) and warm tap water. Rinse with deionized water and dry. To speed the drying of the optical cell, connect the heater plug to the optical cell with the windows off for several minutes. Clean the quartz windows with methanol and a piece of lens paper.

f. Document any maintenance in the Mercury maintenance logbook located next to the instrument.

NOTE: Detailed instructions for the maintenance and troubleshooting of the Leeman Labs Mercury Analyzer can be found in the *PS200 Leeman Labs Mercury Analyzer Manual*.

Quality Control:

1. Follow current Good Laboratory Practices with all procedures.

2. Calibration curves must be composed of a minimum of a blank and three standards.

3. Immediately after calibration of the instrument, an initial calibration verification (ICV) must be analyzed. This control must read ±20.0% for CLP mercury samples and ±10.0% for EPA 600 and SW-846 mercury samples.

4. After the ICV, the reagent blank (initial calibration blank [ICB]) must be verified. The blank must be less than the absolute value of the limit of quantitation.

5. A low-level check (CRA) standard must be read at the limit of quantitation. No control specifications currently exist for these standards.

6. After analyzing ten samples, verify calibration with the continuing calibration verification (CCV) and continuing calibration blank (CCB). The CCV control must read ±20.0% for CLP and SW-846 mercury samples and ±10.0% for EPA 600 mercury samples of the true value of the standard. The CCB must be ± the absolute value of the limit of quantitation for EPA 600 and SW-846 samples or ± the absolute value of the CRDL for CLP samples. If either of the control values fall outside their respective limits, recalibrate the instrument, verify calibration, and reanalyze any samples which are not bracketed by valid calibration checks. After analysis of the last sample, verify the CCV and CCB.

7. For additional quality control please refer to SOP-IO-005, "Department 23 Quality Control Procedures."

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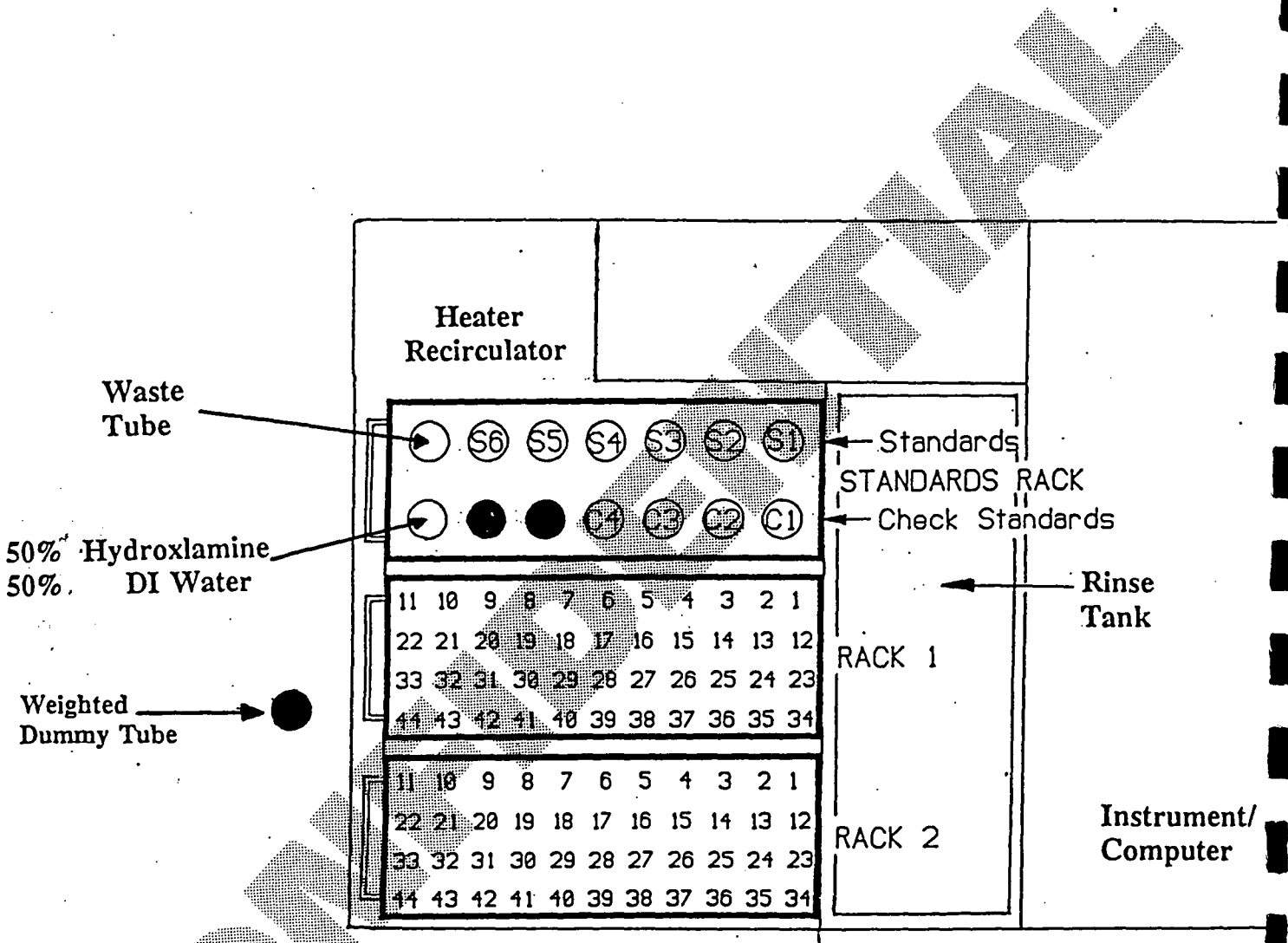
Prepared by: 383 Date: 10-3-95

Approved by: Ramona Nayman 026 Date: 10/3/95

Approved by: 972 Date: 10/3/95

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			

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601

Procedural Amendment #1

Number: 0206

Title: Total Suspended Solids

Effective Date (listed on procedure): 12/10/97

Section(s) affected by change: Procedure 4.b

Reason for addition(s) or change(s): Client request for clarity

Change will be effective from (date): 08/11/99

Samples or project affected: All samples

List change(s) or addition(s) (specify which section):

Procedure:

4. Sample analysis *(Replace Item b as follows)*

   b. Measure 250 mL of sample, or a smaller portion if the sample matrix dictates, using a graduated cylinder or a calibrated pipette. Record sample volume and crucible ID on worksheet.

0206.DOC
081199

Prepared by: Susan Hibner Date: 8/12/99

Approved by: Robert Hussey Date: 8/12/99

Approved by: Daneys R. Clark Date: 8/12/99

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

Hexane Extractable Material (HEM)

Reference:

Method 1664, *Methods for Chemical Analysis of Water and Wastes*, USEPA.

Scope:

This method is for the determination of *n*-hexane extractable material (HEM) in waters. Extractable materials that may be determined are vegetable oils, animal fats, waxes, soaps, greases, and related materials. The limit of quantitation for this method is 5.0 mg/L. The holding time is 28 days.

The referenced method stipulates a solvent evaporation bath temperature of 85°C. Due to the design of the bath being used, this method will utilize a bath temperature of 98°C to enable the hexane to boil.

Basic Principles:

A 1-L sample is acidified to a pH <2 and serially extracted 3× with *n*-hexane in a separatory funnel. The extract is dried over sodium sulfate, the solvent evaporated from the extract, and the residual HEM is weighed.

Apparatus and Reagents:

1. 2-L separatory funnel with a Teflon stopcock
2. 2-L glass beaker
3. Pleated filter paper, Whatman No. 40, or equivalent
4. 125-mL tared distilling flask containing eight to ten boiling chips

5. Concentrated hydrochloric acid (HCl), stable indefinitely at room temperature

6. Sodium sulfate (Na_2SO_4), anhydrous crystal, stable indefinitely at room temperature

7. Vacuum pump or equivalent

8. Glass funnels

9. Analytical balance

10. Solvent capture system or equivalent

11. Desiccator

12. *n*-Hexane - 85% purity, 99.0% min. saturated C_6 isomers, purchased, see label for expiration date. Store at room temperature.

13. Reagent grade acetone, purchased, see label for expiration date. Store at room temperature.

14. Mechanical shaker

15. Hexadecane - 98% minimum purity, purchased, see label for expiration date. Store at room temperature.

16. Stearic acid - 98% minimum purity, purchased, see label for expiration date. Store at room temperature.

17. Hexadecane/stearic acid (1:1) stock solution (4000 mg/L) - Weigh 0.400 ± 0.004 g of stearic acid and 0.400 ± 0.004 g of hexadecane and transfer to a 200-mL volumetric flask. Dilute to volume with reagent grade acetone. Store, well stoppered at 4° ± 2°C. Holding time is 1 month.

18. 40 mg/L standard - Pipette 10 mL of 4000-mg/L stock solution into a separatory funnel containing 1000 mL of deionized water. Prepare fresh daily.

19. Deionized water

NOTE: Alternative weights and volumes may be used for all reagents as long as final concentrations remain the same.

Safety Precautions:

n-Hexane has been shown to have neurotoxic effects. It should be used only in a well ventilated area. *n*-Hexane is also flammable, and care must be taken to ensure that it is always stored in a flammable cabinet and disposed of 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. Obtain approximately 1000 mL of sample, or an aliquot diluted to 1000 mL and mark the sample level on the bottle for later determination of sample volume. Allow sample to come to room temperature before analysis.

2. Acidify the sample to a pH <2 by adding 5 mL of concentrated hydrochloric acid if the sample has not been preserved or if 500 mL or more of deionized water has been added to a reduced aliquot of sample. Verify the pH of the adjusted sample by placing a drop from a glass stir rod on a strip of pH paper.

3. Transfer the sample to a 2-L separatory funnel.

4. Rinse the graduate or sample container with approximately 30 mL of *n*-hexane and then transfer to the separatory funnel.

5. Load the separatory funnels onto the shaker with the stopcocks open. Shake at an intensity of 30 for 30 seconds, then close the stopcocks and shake at an intensity of 45 for 90 seconds. Be sure to vent the separatory funnels before removing from the shaker. Return the funnels to the extraction rack, and allow the layers to separate for a minimum of 10 minutes.

6. Drain the aqueous layer (lower layer) into the original sample container. Drain a small amount of the solvent layer into the sample container to minimize the amount of water remaining in the separatory funnel.

7. Drain the *n*-hexane layer (upper layer) from the separatory funnel through a solvent-moistened pleated filter paper containing approximately 1 g of sodium sulfate into a tared 125-mL distilling flask containing eight to ten boiling chips.

8. Repeat steps 3 to 6 two more times with fresh 30-mL portions of *n*-hexane. Combine all three extracts into the tared distilling flask, and rinse sodium sulfate and filter paper with *n*-hexane, collecting the rinses into the tared distilling flask.

9. Place the flask on a solvent capture system that has been prewarmed to approximately 98°C. Evaporate to dryness (this should take approximately 30 minutes or less).

10. Insert tubing connected to a vacuum source for approximately 2 minutes to remove any solvent vapor from the flask. Wipe the outside of the flask to remove moisture and fingerprints.

11. Cool the flask in a desiccator for 30 minutes and weigh. Redesiccate for 15 minutes and reweigh. If the second weight is more than 1 mg lower than the first weight, redesiccate for 15 minutes more and reweigh. Continue in this manner until a constant weight is achieved.

12. If SGT-HEM (Analysis #8078, "Silica Gel Treated Hexane Extractable Material [SGT-HEM]") is to be determined, save the extract, and all associated QC, for later determination.

Calculation:

$$mg\ oil\ HEM\ grease/Liter = [((A - B) / mL\ of\ sample) \times 1{,}000{,}000]$$

Where:

A = Weight of flask and residue, grams

B = Original weight of flask, grams

Quality Assurance:

Each batch of twenty samples must contain the following QC: a blank, a 40-mg/L LCS (10 mL of the 4000-mg/L HEM standard to 1 L deionized water), a matrix spike, a matrix spike duplicate, and a matrix duplicate. When there is not enough sample to perform the matrix spike and matrix spike duplicate, a LCSD must be performed. See Wang for current quality control acceptance windows.

The analytical balance must be calibrated before and after each batch is weighed using 2- and 1000-mg Class 1 weights. Calibration of the balance must be within ±10% of the true value at 2 mg (i.e., ±.2 mg) and ±0.5% at 1000 mg (i.e., ±5 mg).

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: 12/31/96

Ver. #	Effective Date	Change
01	06/27/97	New
02	08/18/97	Major changes are as follows: • Procedure – Add "allow sample to come to room temperature before analysis"
03	12/29/97	Major changes are as follows: • Scope – Added justification for differing bath temperatures • Reagents – Added storage conditions • QA – Changed batches open from 5 working days to 14-calendar days
04	JUN 3 0 1999	Major changes are as follows: • Apparatus and Reagents – Deleted section #17 and changed mg to g • QA - Revised

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060899

Prepared by: Jaime Jakloneski Date: 6-4-99

Approved by: Kenneth Bell Date: 6/9/99

Approved by: [signature] Date: 6/16/99

APPENDIX E

SOPS FOR FIELD INSTRUMENTS

STANDARD OPERATING PROCEDURES
FIELD MEASAUREMENT OF pH, TEMPERATURE AND SPECIFIC CONDUCTIVITY IN WATER

1.0 PURPOSE

This method is applicable to samples of stormwater, surface water, potable water supplies, and groundwater with measurement occurring at the sampling point. This procedure defines acceptable and consistent methods for measurement of pH, specific conductance, and temperature.

2.0 APPARATUS

The YSI Model 3560 Water Quality Monitoring System, or equivalent instrument, will be used. The instrument is a portable, microprocessor based pH, specific conductivity, and temperature meter.

3.0 REAGENTS

A) pH reference buffer solutions:

1) pH = 4.00
2) pH = 7.00
3) pH = 10.00

B) Specific conductivity buffer solutions:

1) Conductivity standards A or B = 1413 uS or12.88 mS

C) Distilled water

4.0 CALIBRATION PROCEDURES

A) Select sensor (i.e. pH, conductivity)

B) One point calibration

1) Place the sensor in the calibrating medium:

Measurement	Solution	Reading
pH	pH = 7 buffer	7.00 pH (25°C)
Cond	Hold in free air	0.00 μS
TDS	Hold in free air	0.00 mg/L

(2) Press CAL - cal 1 is displayed. After endpointing, the display automatically updates to the calibrated value shown, or the temperature compensated value.

(3) If READ is pressed after cal 1 update, the meter assumes one point calibration only is required. Samples can now be measured.

C) Two point calibration

Follow one point calibration. Place sensor in second calibrating medium: Calibration for pH measurements will utilize the two point calibration option.

Measurement	Solution	Reading
pH	pH 4 or pH 10 buffer	4.00 or 10.01 pH (at 25°C)
Cond	Cond std A or B	1413 uS or 12.88 mS
TDS	Cond std A or B	706 mg/L or 6.44 g/L

2) Press CAL - cal 2 is displayed. After endpointing the display automatically updates to the calibrated value shown or the temperature compensated value.

5.0 SAMPLE HANDLING AND PREPARATION

Samples collected for pH, specific conductivity, and temperature should be obtained directly from the sampling point. Groundwater samples being tested during well purging can be obtained directly from the bailer.

6.0 PROCEDURES

Select sensor for required measurement (i.e. pH sensor or conductivity sensor). Attach sensor to the M90 meter. Calibrate meter to the solution corresponding to type of sensor. Meter is now ready to make a measurement following these steps:

A) Prepare sensor

1) pH - remove the sensor wetting cap and slide the vent sleeve to expose the fill hole.
2) Specific conductivity/TDS - immerse probe to halfway point in solution.
3) Temperature - pH and conductivity sensors automatically measure temperature.

B) Press MODE, READ, CAL, or M to turn meter and start measurement. Place sensor into solution. Automatic endpoint detection freezes the display when plateau is reached; to manually endpoint press READ. Press READ again to start new measurement.

C) After use, close the fill hole and replace the wetting cap (pH).

7.0 TROUBLE-SHOOTING AND MAINTENANCE

A) Use distilled water when transferring from one solution to another.

B) Response time is function of the sensor and the solution. If the solutions are at different temperatures (or ionic strength - pH only), allow more time for the sensor to respond.

C) Avoid handling the sensor tip.

D) Make sure no large air bubbles are trapped under the sensor when making measurements.

E) Do not use calibration standards after the expiration date.

F) Wetting caps should contain:

pH - pH 7 buffer

G) For greatest accuracy, calibrants and samples should be at the same temperature.

H) pH - keep the electrode filled with the appropriate fill solution to prevent reading drift.

I) Conductivity - the sensor shield and probe should be kept clean. Make sure no air bubbles are in the cell chamber during measurement.

8.0 ACCURACY AND PRECISION

Accuracy for pH and specific conductance is addressed by verifying the agreement of two standards/buffers from separate lots. pH should agree within 1 unit. Specific conductance should agree within 10%. Accuracy for pH can also be checked by verifying on a third standard. Calibration would not be adjusted based on the third standard. Accuracy will not be verified for temperature. Temperature will not be used to determine cleanup objectives and will not halt groundwater sampling.

Precision for pH and specific conductance will be verified by checking the measurement on a split of the original sample (i.e., split buffer solution into two jars and verify precision). Measurements should be within 1 unit for pH and 10% for specific conductance. Precision for temperature will not be determined.

Accuracy and precision will be verified during each calibration (i.e., beginning of the day, middle of the day, and as required during sampling) and recorded in the logbook. Corrective action will be taken as required.

9.0 REPORTING

A) pH - report the average value of the replicate measurements to the nearest 0.1 units.

B) Temperature - report the average value of the replicate measurements to the nearest 1°C.

C) Specific Conductivity/TDS - report the average value of the replicate measurements to three significant digits.

APPENDIX F

EXAMPLES OF CHAIN OF CUSTODY FORMS

[redacted] Analysis Request / Environmental Services Chain of Custody

Lancaster Laboratories
A division of Thermo Analytical Inc.

For Lancaster Laboratories use only

Acct. # _____ Sample # _____

Please print. Instructions on reverse side correspond with circled numbers.

(1)

Client: Kerr McGee Acct. #: _____

Project Name/#: Moss-American PWSID #: _____

Project Manager: Tom Graan P.O.# _____

Sampler: TBD Quote #: _____

Name of state where samples were collected: Wisconsin

(2) **Sample Identification** | **Date Collected** | **Time Collected** | (3) **Grab** | **Composite** | (4) **Matrix**: Soil | Water | ☐ Potable ☐ NPDES (Check if applicable) | Other | **Total # of Containers** | (5) **Analyses Requested**: BTEX | PAHs | VOC | Total Metals | Total Mercury | Cyanide | Oil & Grease | TSS | **Remarks** | (6) **Temperature of samples upon receipt (if requested)**

Sample Identification	Date Collected	Time Collected	Grab	Composite	Soil	Water	Potable / NPDES	Other	Total # of Containers	BTEX	PAHs	VOC	Total Metals	Total Mercury	Cyanide	Oil & Grease	TSS	Remarks	Temperature of samples upon receipt (if requested)
MA3-SS05	8/10/99	10:00	X		X				4	X	X								
MA3-TW-N020700-01	8/10/99	12:00	X			X					X	X	X	X	X	X	X		
MA3-TW-TB040700-01	8/10/99	11:00	X			X						X							

(7) **Turnaround Time Requested** (TAT) (please circle): (Normal) Rush

(Rush TAT is subject to Lancaster Laboratories approval and surcharge.)

Date results are needed: 8/25/99

Rush results requested by (please circle): Phone Fax

Phone #: _____ Fax #: _____

(8) **Data Package Options** (please circle if requested)

QC Summary | Type VI (Raw Data)

Type I (Tier I) | GLP

Type II (Tier II) | Other

Type III (NJ Red. Del.)

(Type IV (CLP))

SDG Complete? Yes (No)

Site-specific QC required? Yes No

(If yes, indicate QC sample and submit triplicate volume.)

Internal Chain of Custody required? Yes No

(9)

Relinquished by:	Date	Time	Received by:	Date	Time
TBD	8/10/99	16:00			
Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by:	Date	Time

CLIENT

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

MA3-TW-TB040700-01

SAMPLE IDENTIFICATION / LOCATION CL. RES:

Groundwater Sample

COLLECTION INFORMATION:

☐ COMPOSITE
☒ GRAB

DATE 8/10/99 TIME 11:00 BY: weston

TESTING REQUIRED	PRESERVATIVE(S) ADDED
VOC analysis	HCL

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

LL # 16473-2