REM IV Remedial Planning Activities at Selected Uncontrolled Hazardous Waste Sites–Zone II

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FINAL

Environmental Protection Agency
Hazardous Site Control Division
Contract No. 68-01-7251

QUALITY ASSURANCE PROJECT PLAN

PHASE I
REMEDIAL INVESTIGATION/
FEASIBILITY STUDY
VOLUME I OF II

Moss-American
Milwaukee, Wisconsin

EPA WA 5-5LM7.0

CH2M HILL
Black & Veatch
ICF
PRC
Ecology and Environment

QUALITY ASSURANCE PROJECT PLAN

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October 15, 1987

Remedial Planning Activities
(REM/IV) ZONE II
Contract Number 68-01-7251

QUALITY ASSURANCE PROJECT PLAN (QAPP)

Project Title: Moss-American, Wisconsin

EPA No.: 5-5LM7.0

EPA Remedial Project Officer: Gregg Kulma (Acting RPO)

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EPA Remedial Project Manager

Reviewed _____ Date: 10/29/87
EPA Director, Central Regional Laboratory

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EPA Quality Assurance Officer

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1.0 INTRODUCTION

The United States Environmental Protection Agency (U.S. EPA) requires participation of all U.S. EPA contractors in a centrally managed quality assurance (QA) program. This requirement applies to all environmental monitoring and measurement efforts mandated or supported by U.S. EPA.

Each contractor generating data has the responsibility to implement minimum procedures to assure that the precision, accuracy, completeness and representativeness of its data are known and documented. To ensure the responsibility is met uniformly, each U.S. EPA contractor must prepare a written Quality Assurance Project Plan (QAPP) covering each project it is contracted to perform.

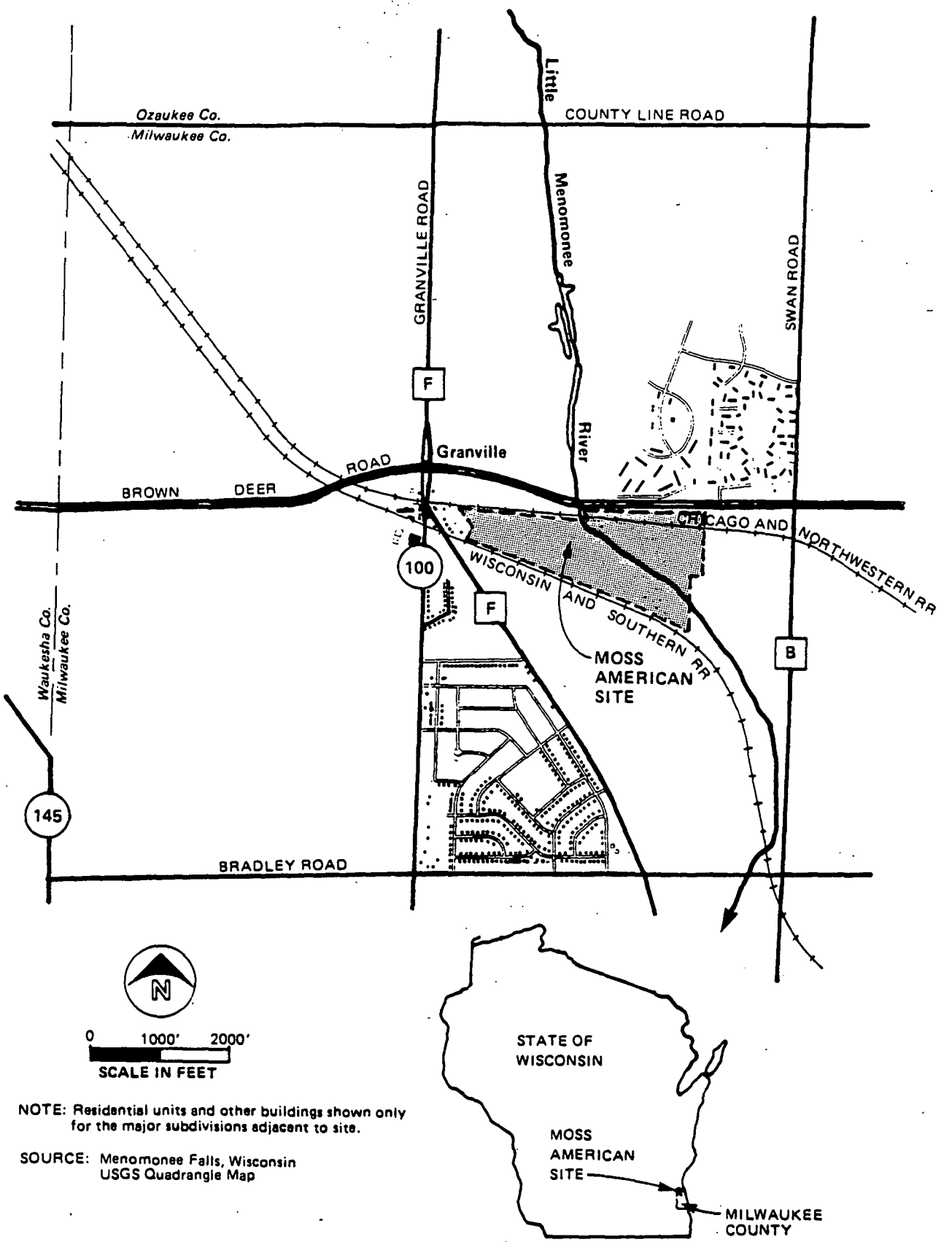
This QAPP presents the organization, objectives, functional activities and specific QA and quality control (QC) activities associated with the Remedial Investigation/Feasibility Study (RI/FS) at the Moss-American site near Milwaukee, Wisconsin. This QAPP is designed to achieve the specific data quality goals of the RI/FS.

2.0 PROJECT DESCRIPTION

The Phase I remedial investigation (RI) portion of the RI/FS is designed to gather specific information necessary to determine if the site presents a hazard to human health or welfare or to the environment. All tasks and subtasks are directed toward accomplishment of these primary objectives. The Phase I RI will determine whether additional phases are needed to perform a feasibility study (FS).

2.1 BACKGROUND

The Moss-American superfund site is in the northwestern part of the City of Milwaukee, one-quarter mile east of 107th Street (State Highway 100) on Brown Deer Road. It is located in the northwest 1/4 of Section 8, Township 8 North, Range 21 East, which is covered by the Menomonee Falls, Wisconsin USGS 7½-minute quadrangle map (Figure 1). The 88 acre site is bounded by the Chicago and Northwestern Railroad and Brown Deer Road to the north, the Wisconsin and Southern Railroad to the south, the west edge of the Chicago and Northwestern



NOTE: Residential units and other buildings shown only for the major subdivisions adjacent to site.

SOURCE: Menomonee Falls, Wisconsin USGS Quadrangle Map

FIGURE 1
LOCATION MAP
MOSS AMERICAN QAPP

Railroad automobile storage lot to the west, and a north-south line approximately 3,500 feet east of 107th Street. The Little Menomonee River enters the site through the northern boundary and leaves through the eastern boundary.

The site is the former location of Moss-American Co., Inc., a creosoting plant where railroad ties and other wood products were treated and stored. The wood preserving plant was established in 1921 by the T.J. Moss Tie Company. Kerr-McGee purchased the T.J. Moss facility in 1963 and the American Creosote Co. in 1964. The two companies were consolidated in 1965 and became known as the Moss-American Company. The name was changed to the Kerr-McGee Chemical Corporation--Forest Products Division in 1974. Operations at the site ceased in June 1976 and all buildings and equipment were subsequently dismantled and removed.

A series of ditches collected spilled oil and creosote and rainwater and snow melt runoff. The ditches discharged directly into the Little Menomonee River. Sometime before 1941, the ditch system was modified to include a series of settling ponds and an oil separator system. In 1952, the plant yard was resurfaced. About 20 acres were covered with gravel and were used to store untreated wood. Another 10 acres were covered with cinders and were used to store the treated wood products. Subsurface drain tiles under the newly surfaced yard emptied into an open ditch which eventually emptied into the Little Menomonee River. Bales of straw were added to the ditch as oil filters at the request of the City of Milwaukee in 1954. No major changes were made to the treatment system until 1966 when the Milwaukee Sewerage Commission advised Moss-American to repair a pond that was leaking oil to the Little Menomonee River.

The site received attention in 1968 when a dump upstream of the site burned out of control for over a year. Water poured on the fire caused the Little Menomonee River to become anaerobic. Subsequent studies by EPA found that the effluent discharged to the river from the Moss-American site was of undesirable quality. The City of Milwaukee ordered a cleanup and Moss-American complied by installing coke filters to pretreat the waste. In 1971, all the industrial and domestic wastes from the Moss-American facilities were diverted into the Milwaukee Metropolitan sewage system.

State and national attention was brought to the site in 1971 when a group of young people received chemical burns while cleaning debris from a stretch from the Little Menomonee River more than three miles downstream of the Moss-American site. Subsequent studies by EPA determined that creosote

originating at the Moss-American facilities was the cause of the chemical burns. As a result of these findings Kerr-McGee dredged and backfilled eight interconnected waste ponds with clean fill. These were located in series along the drainage ditch in the vicinity of the largest of two more recent ponds identified and shown in Figure 2. Additionally, 1,700 feet of river were dredged and the contaminated sediments removed and placed along the river embankment or in a small landfill in the northeast corner of the site as shown in Figure 2. An underground clay wall was constructed between the ponds and the river.

In 1972, EPA awarded contracts to Rexnord, Inc. and Biotest, Inc. for demonstration test of removal and treatment of creosote-contaminated sediments from the river bottom. The Rexnord demonstration was conducted near the site. The other demonstration by Biotest was conducted on sediments 1½ miles downstream near Calumet Road (Figure 3). The Rexnord method was selected for continued use. About 4,000 feet of river bottom sediments extending south from the Chicago and Northwestern Railroad bridge were cleaned before funding ran out.

In 1974, EPA filed suit against Kerr-McGee, seeking recovery of costs incurred for the experimental projects and cleanup of the entire river as well as civil penalties. The suit was dropped as a result of falsification of data in one study by an EPA investigator. In 1978, Milwaukee County agreed to accept ownership of a portion of the site in return for dropping its lawsuit against Kerr-McGee.

Currently, the western 23.3 acres of the site are owned by the Chicago and Northwestern Railroad and have been developed as an automobile storage and loading area. The remaining 65 acres are owned by Milwaukee County and are used as a public park.

Documented contaminants generated at the site include polynuclear aromatic hydrocarbons (PAH's) and phenols present in creosote. Offsite, oily compounds, presumably derived from creosote and No. 6 fuel oil (also used for wood preservation), have been observed in the bed of the Little Menomonee River from the site to the confluence of the Menomonee River, approximately 5.75 miles downstream (Helvig, 1975). Onsite, such compounds are suspected to exist in the soils of former processing areas, drip tracks, treated railroad tie storage areas, waste ponds and effluent ditches, the sludge disposal area, and the dredgings landfill area as well as in the river bed. These potential contaminant source areas and other important site features are shown in Figure 2.

Analysis of onsite river bottom muds for hexane extractables (present in creosote and No. 6 fuel oil) in 1973 showed concentrations ranging from below detection limits to 43,480 mg/kg (Rexnord, 1973). The median sample concentration detected was 2,280 ug/kg. Onsite soils contained phenol in concentrations ranging from 15 to 211 ug/kg, (Martin, 1977). Methylene chloride extractables (present in creosote and fuel oil) were found in concentrations that ranged from below detection limits to 279,000 ug/kg (NEIC, 1977). Detectable levels of contaminants were found in soils at depths to to 15 feet below ground surface.

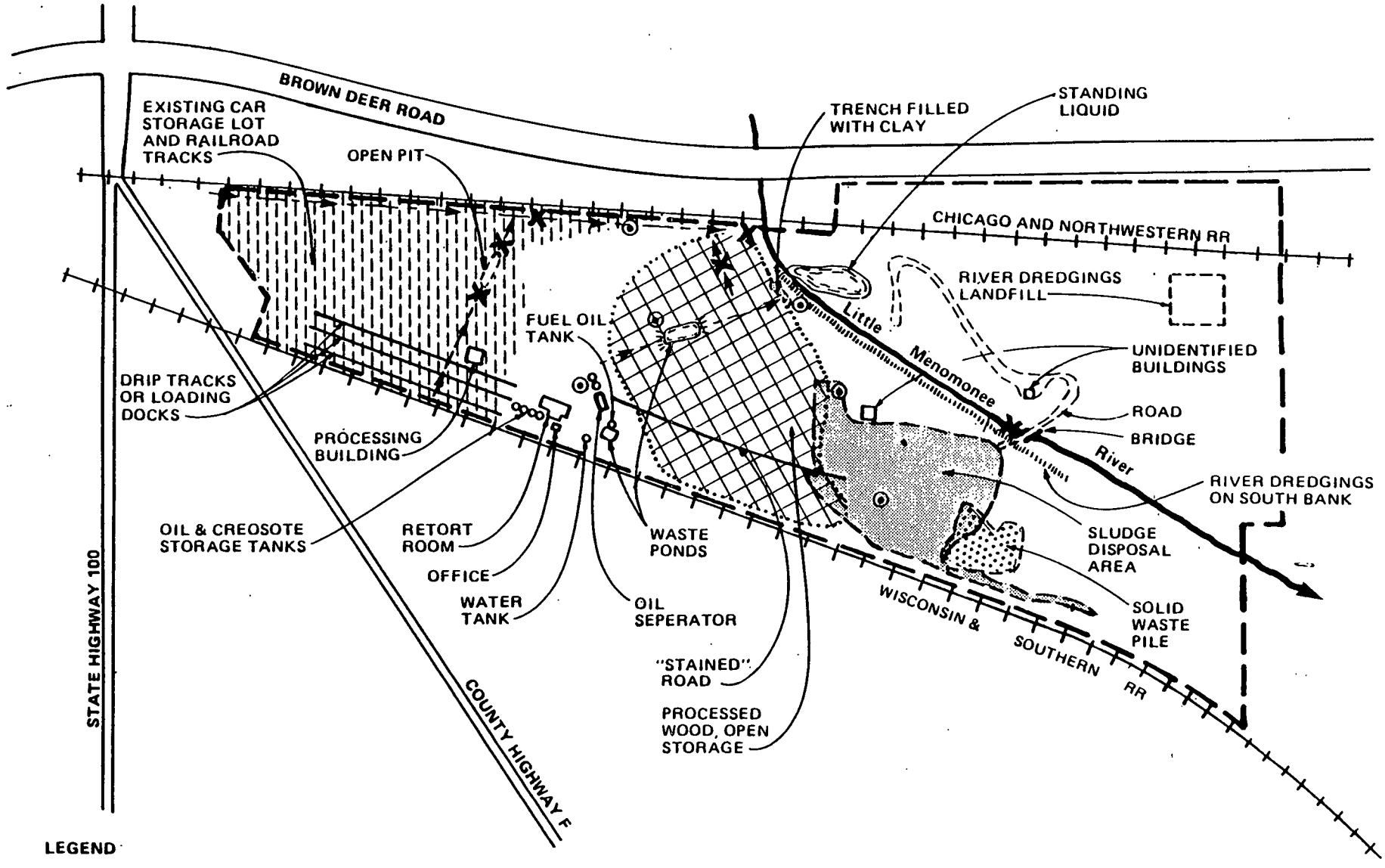
Onsite soils were sampled by Marquette University in April 1984 at the request of the Task Force on Pesticide and Herbicide Use of Milwaukee County. Concentrations of methylene chloride extractables in the samples ranged from below detection limits to 481,000 mg/kg (letter from Clifford J. Crandall to Penny Podell, June 5, 1984). Results of these analyses are presented in Appendix A.

Analysis of well samples by NEIC (1977) suggests that onsite groundwater is contaminated. Concentrations of methylene chloride extractables measured in 1977 ranged from below detection limit to 4,190 mg/l. Contamination may have resulted from creosote and fuel oil by-products being leached from the surface soils by infiltration of surface water or by direct contact between groundwater and contaminated soils.

Surface water samples have been collected from the Little Menomonee River and analyzed for phenol (WDNR, 1969; Rexnord, 1971; and USACE, 1971) and for oil and grease (Rexnord, 1971). Phenol concentrations as high as 4 mg/l have been detected near the outfall from the Moss-American site. Trace amounts have been detected elsewhere downstream. Oil and grease concentrations as high as 11 mg/l have been observed in surface water samples downstream of the site.

2.2 PROJECT OBJECTIVES

The overall objective of the RI is to determine the nature and extent of the threat posed by the release or threat of release of hazardous substances and to evaluate proposed remedies. Before alternatives for remedial actions can be considered in the FS, there must be sufficient information available to develop, screen, and evaluate potential alternatives. With this in mind the following project objections have been identified for the Moss American Site:



NOTE: This is a composite map of features that at one time had been on the site. Many of these have since been dismantled or covered.

FIGURE 2
POSSIBLE CONTAMINANT SOURCE AND OTHER IMPORTANT FEATURES
MOSS AMERICAN QAPP

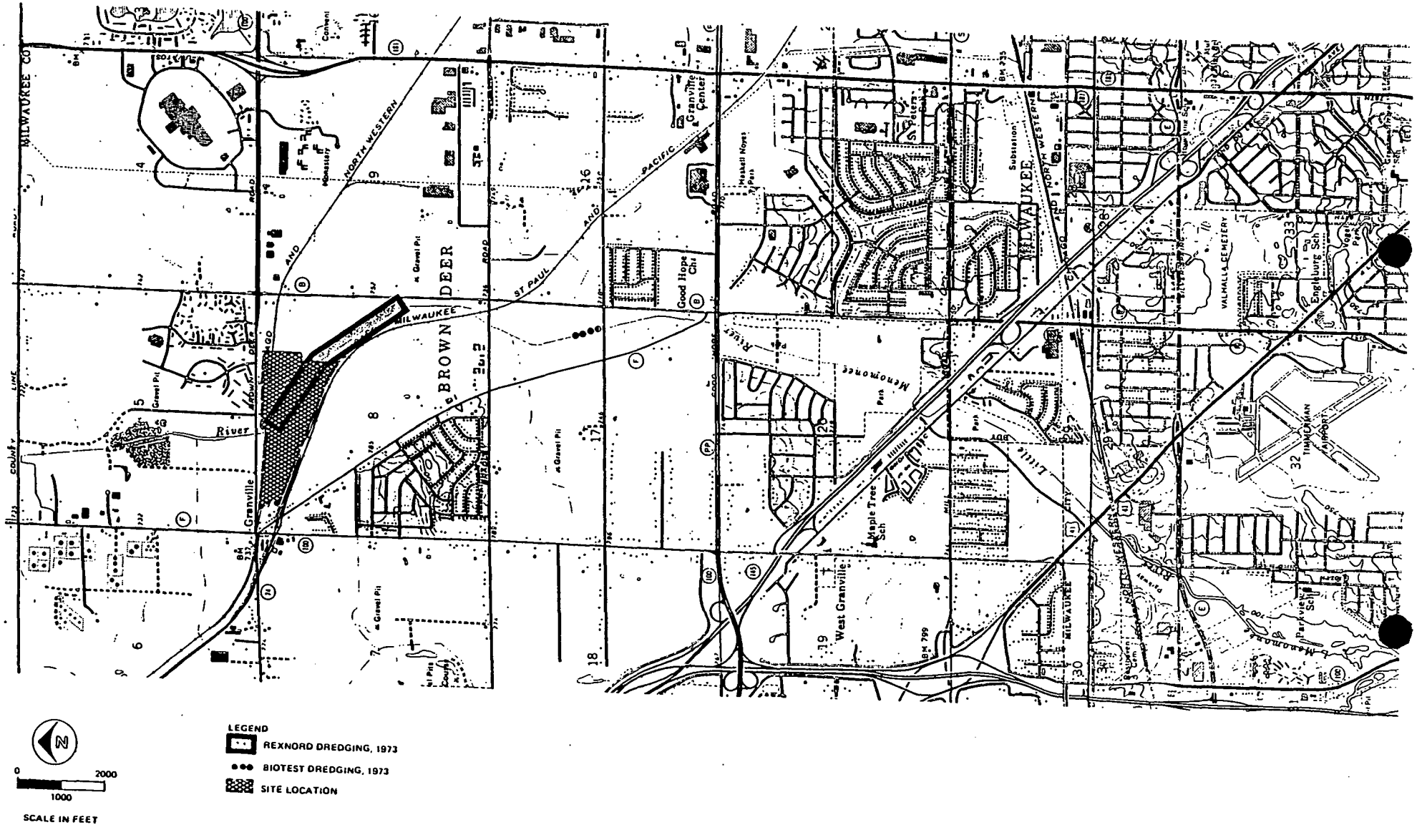


FIGURE 3
RIVER BOTTOM
DREDGING LOCATIONS
MOSS-AMERICAN

- Determine the nature and extent of creosote and oil contamination in onsite soil, groundwater, surface water and river sediment
- Identify and evaluate potential routes of contaminant release and migration.
- Determine if groundwater contamination has the potential to migrate offsite in deeper aquifer zones and affect water supplies.
- Determine the chemical nature of the groundwater and surface water to evaluate the feasibility of various treatment technologies.
- Determine soil properties on the site and in the river to evaluate physical and chemical parameters that may affect potential remediation technologies.
- Determine the nature of hydrogeologic units beneath the site to evaluate contaminant migration and physical and chemical parameters that may affect potential groundwater remediation, barrier, or containment technologies.

Evaluation of existing data (Appendix A) from previous investigations of the site and surrounding area indicates that the project objectives discussed above have not been satisfied. The last of several investigations of the Moss-American site was completed in 1977. Since these investigations were completed, conditions have changed significantly onsite and in the river. River sediments have been transported and deposited, part of the site has been paved over or covered with new material, and new vegetation has grown over old storage, treatment, and disposal areas.

Once the Phase I RI data have been obtained and evaluated in the RI report and it is determined that no additional phases of remedial investigations are required, the following tasks will be performed as part of the feasibility study:

- Develop and evaluate remedial action alternatives.
- Recommend a cost-effective remedial action(s) for the site.

2.3 DATA USES AND DATA NEEDS

Consistent with the project objectives, sampling and analysis activities during the Phase I RI have been developed for the

purpose of characterizing the site (determining the horizontal and vertical extent of contamination), providing input to the risk assessment, and providing information for screening and evaluating remedial alternatives. Most of the data necessary to meet the project objectives can be satisfied by performance of a Phase I investigation program that incorporates endangerment, hydrogeologic, hydrologic and remedial technology data collection activities. Data will be required to:

- Assess the nature and extent of groundwater, surface water, soil, and sediment contamination to evaluate needs and limitations of removal, containment, and/or treatment technologies.
- Determine the contaminant loading to the Little Menomonee River from release through groundwater recharge or surface water routes.
- Assess the nature of the surface water regime to evaluate contaminant and sediment transport and physical and chemical parameters that may affect potential surface water and sediment remediation.
- Estimate the potential impact and risks to human health and the environment from the presence or release of creosote and oil from the site or river sediments.
- Define cost-effective remedial measures to reduce the risk or threat posed by the presence or release of contaminants onsite, away from the site or in river sediments.

CH2M HILL will execute sampling and analysis activities as Phase I of the RI. The intended use of the data collected during the Phase I RI will be to:

- Perform a characterization and hydrogeologic assessment of the site;
- Determine the level and volume of contaminated soil on site and sediment in the river;
- Evaluate the potential for endangerment to the public health, welfare, or environment;
- Perform additional screening of remedial alternatives; and

- Determine whether any additional phases of field investigation are needed to perform a FS.

It is intended that sufficient data will be provided by the Phase I scope of work to proceed directly to the FS. If insufficient data or unforeseen conditions warrant, a second phase of the RI may be necessary.

2.4 DATA QUALITY OBJECTIVES

The Data Quality Objectives (DQO's) to address the data needs and data uses for the Moss-American Phase I RI are:

1. To define outer boundaries of creosote contamination in onsite soils and river sediment by semiquantitative and real-time field screening methods in a cost-effective and timely manner.

2. To define relative creosote contaminant concentrations such that submittal of high concentration samples (as known to exist at the site) can be controlled to avoid instrument interference or failure, and such that samples later sent for analysis can be properly prepared and handled.

3. To verify in a cost-effective manner that the screening method has identified contaminants of concern (creosote and fuel oil constituents).

4. To quantitatively identify (in fewer, but statistically sufficient number of samples) constituents of the creosote contamination for remedial needs, and to support the public health assessment.

5. To analytically confirm and document for litigative purpose levels of creosote constituents present in site soil, river sediments and groundwater.

6. To support selection of remedial alternatives in the FS.

To meet these six DQO's the Work Plan proposes the use of analytical techniques in accordance with the analytical levels defined in "Data Quality Objectives for Remedial Response Action" (EPA 540/G-87/003A). To facilitate site characterization, Level I field screening techniques will be instituted; this method involves OVA/HNu analysis conducted by CH2M HILL.

Level II field screening techniques have been selected to satisfy DQO's 1 and 2 above. This method involves total carbon (TC) screening utilizing the U.S. EPA Close Support Laboratory (CSL) to handle the large number (600) of samples proposed. See Appendix B for a discussion of the methodology.

To meet the DQO's 3 and 4 above, a Level III analytical method using gas chromatography with flame ionization detection (GC/FID) has been developed by CH2M HILL's Montgomery Laboratory. The standard operating procedure and back-up data for this method is presented as Appendix C.

Analytical confirmation methods to satisfy DQO's will require use of the Contract Laboratory Program (CLP) in accordance with Level IV analytical support needs.

Additional nonstandard or special analytical services (SAS) will be necessary to accomplish DQO No. 6. These analyses are considered Level V analytical support methods.

3.0 RI TASKS

3.1 PHASE I SUPPORT

The following tasks are intended to provide support throughout all phases of the RI:

Task PM--Project Management. Project management activities will be handled through CH2M HILL's office in Milwaukee, Wisconsin. Contact will be maintained with the U.S. EPA Remedial Project Manager (RPM) during all phases of the project.

Project management activities during Phase I will include preparation of monthly reports to keep the U.S. EPA informed of the technical, financial, and schedule status of the project. Other responsibilities include controlling budgets and schedules; selecting, coordinating, and scheduling staff and subcontractors for task assignments; maintaining project quality control and assurance programs; and outlining the scope of work for a Phase II RI (if additional field investigative work is necessary) or the FS.

Task FK--Field Work Support. Facilities for decontamination, equipment and sample storage, onsite laboratory analyses and office space for all field personnel during the RI/FS will be established. Selection of storage, decontamination, onsite laboratory, and office areas will be accomplished during a site visit performed by CH2M HILL and EPA personnel.

Provisions for utility connections will also be made during this task.

Other support work to be accomplished under this task will include implementation of the Site Safety Plan.

Task QC--Quality Control. Periodic review of project files, project deliverables, field audits during the field activities, and project progress will be conducted by a review team throughout Phase I of the project. The team will consist of up to four professionals from appropriate disciplines with experience related to the problems and investigations at the site. Project deliverables to be reviewed will include Technical Memoranda, Draft RI Report and Final RI Report.

Task SM--Sample Management. The objective of this task is to track and manage information received from laboratory analyses of samples. Laboratory space and time will be scheduled and analytical data will be tracked.

Task FM--Field Work--Mapping and Surveying. Prior to initiation of field sampling efforts, a site and river survey will be conducted, to inspect existing conditions in order to determine actual sampling locations. Figures 4 and 5 show the approximate locations. Evaluations of the entire 5-mile length of the Little Menomonee River adjacent to and downstream of the site will be included in the Phase I RI. Investigation of contamination within the Menomonee River downstream of the confluence with the Little Menomonee will be reserved for a second phase, if determined to be necessary following Phase I. Because several years have passed since investigation of river sediment contamination, changes in sediment location and river channel configuration are expected.

The river surveying and mapping will include:

- Reconnaissance from 500 feet upstream of the site downstream to the Menomonee River to determine variations in stream channel configuration and delineation of stream segments based on width and depth of channel, channel and bank alignment (straightness), degradational and aggradational zones. This will be accomplished using measuring tapes, sounding rods and probes. Observations will be logged and plotted on 1"=400' photos aerial obtained from the Southeast Wisconsin Regional Planning Commission.

- Delineation of potential dredge disposal areas along the river embankment will be made by comparison of field observations to aerial photographs.

- Identification of other major discharge points to the river downstream of the site, including storm sewers, combined sewers and surface runoff courses.

- Channel profiling of 26 typical sections based on results of stream characterization.

- Development of river sediment and surface water sampling locations based on review of typical stream segment configurations observed. Points will be established to provide cover of aggradational as well as degradational sediment zones, and immediately above and below major discharge points.

To establish initial sampling locations on the site, a more detailed evaluation and inspection of the site will be made. The site surveying and mapping will include:

- Comparison of previous air photos, county planning topographic surveys and topographic surveys obtained during the Interim Authorization phase will be made to evaluate changes to the site since its last evaluation.

- Inspection of the site using a hand auger and shovel to visibly evaluate surface soil conditions up to a depth of 2 feet within areas of suspected contamination.

- Surveying the site, staking 100-foot grid points to mark potential surface soil sampling locations.

3.2 SOIL AND SEDIMENT SCREENING

Because of the large number of samples necessary to define the level and extent of oil and creosote, a two-stepped approach will be made to conduct screening and analysis of contaminated soil onsite and sediment in the river. Tasks will be designated as G (ground) for onsite soil sampling, and S (sediment) for river sediment sampling. The two steps will be designated by (1) for initial sampling; and, (2) for confirmatory (detailed) analysis. Details of each Task G1 and G2 and S1 and S2 are presented below. Initial screening, based on two analytical methods developed by CH2M HILL laboratories, will provide for gross identification of the quantity

of creosote and oils distributed on the site and in the river. Confirmatory analyses by the U.S. EPA Contract Laboratory Program (CLP) using GC/MS techniques will provide confirmation of contaminant identification and levels for litigative purposes, and endangerment assessment. Additional parameters tested for during confirmatory analyses will support evaluation of remedial alternatives.

Total Carbons (TC) Screening Analyses

The first analytical method selected for initial screening is intended to analyze a large number of samples quickly and cost-effectively in the field. Total carbon (TC) analysis according to the procedure given in Appendix B will be performed on approximately 600 total surface soil, subsurface soil, and sediment samples. This analysis is considered to be a Level II analytical method as defined by U.S. EPA's DQO's.

The TC analysis will be performed at the Moss-American site by the U.S. EPA-owned Close Support Laboratory (CSL). The purpose of using the mobile laboratory for in-field TC screening is to expedite the analyses and direct sample collection activities. Proposed surface soil and sediment sample locations may be modified based on the results of the TC analyses. CH2M HILL will be responsible for the management and operation of the CSL.

Gas Chromatography With Flame Ionization Detection (GC/FID) Analysis

The second analytical method selected for the initial screening (Task G1 and S1) of soil and sediment samples uses capillary gas chromatography with flame ionization detection (GC/FID). The standard operating procedure (SOP) for the GC/FID analysis is given in Appendix C. The GC/FID data will be used for the purpose of screening samples, that is, analytically looking for compounds that are indicators of contamination at the Moss-American site, in accordance U.S. EPA DQO's for Level III analytical methods. The indicator compounds selected (Appendix C) are polynuclear aromatic hydrocarbon and phenolic compounds which are common constituents of creosote and have been previously detected at this site. The GC/FID screening results will yield quantitative concentrations to verify the TC screening and further define the boundaries and nature of contamination. These results will also be used in determining the locations of samples to be selected for qualitative analyses by U.S. EPA CLP protocols. A total of approximately 120 surface soil and sediment samples will be analyzed using this

method. The GC/FID analyses will be performed by the CH2M HILL Montgomery laboratory where this soil/sediment screening method was developed and tested using selected soil samples from the Moss-American site.

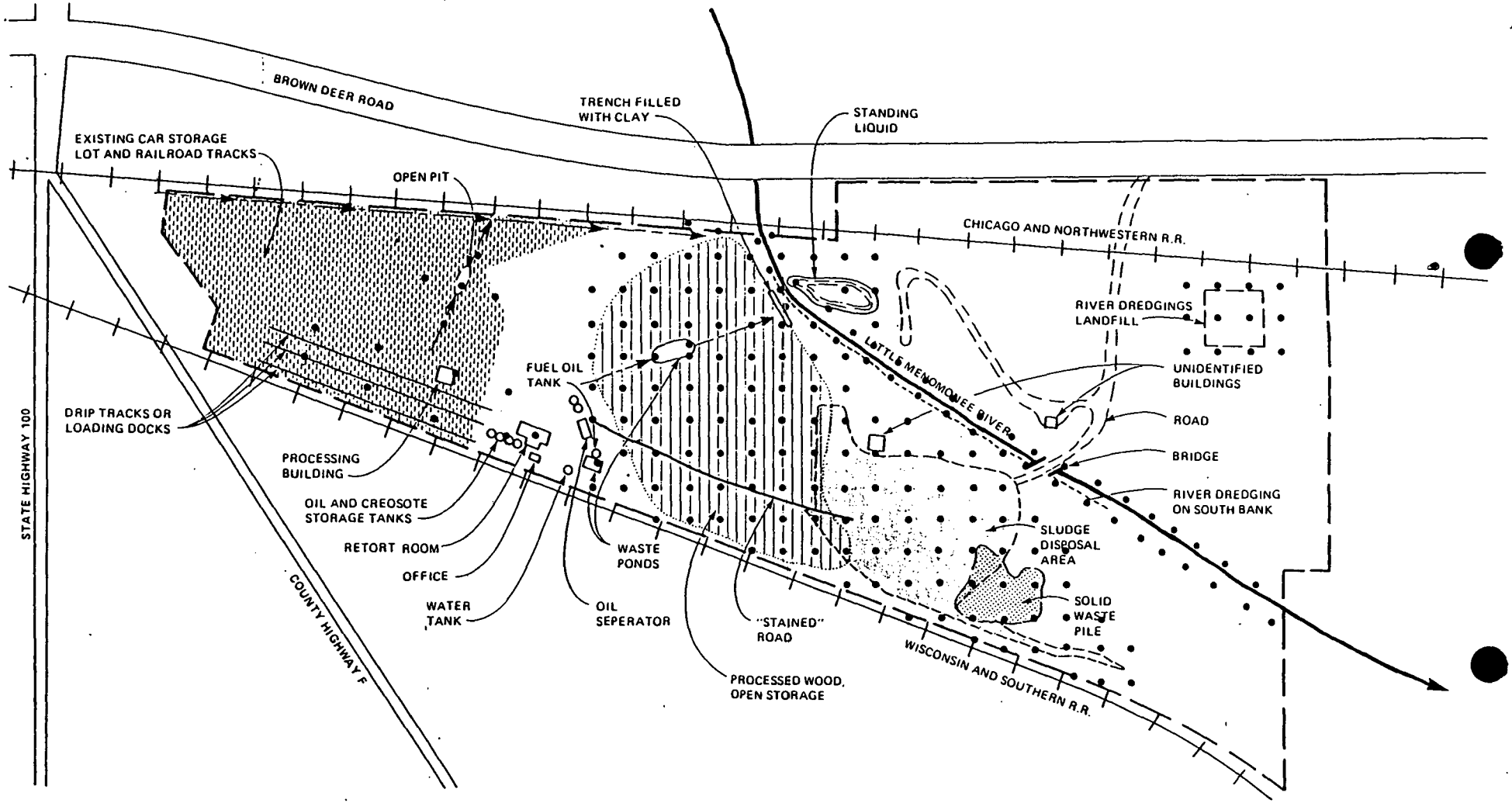
Task G1--Initial Soil Screening. Utilizing a modified 100-foot grid based on apparent past site activities and detailed site inspection (see Figure 4 for proposed grid), a first round of sampling will be initiated within the grid on a 200-foot spacing. Soil samples will be obtained using a trailer-mounted drill rig and split-spoon soil sampler. Continuous split-spoon samples will be obtained to a depth of 4 feet. Samples will be scanned with an organic vapor analyzer (OVA) and photoionization detector (HNu) and descriptions of the samples logged. The OVA and HNu readings will be recorded and used to evaluate the potential for air transport of volatile contaminants.

Based on the results of HNu, OVA readings, and the visual observations of soil samples obtained at each grid point, a portion of the sample from each location (approximately 118 points) will be selected for analytical screening using the CSL and TC method. Results of these first 118 samples will be evaluated and utilized to modify the locations of a second round of sampling of another 118 points on a closer (100-foot) grid spacing. A total of approximately 236 points are to be screened using the TC method. The unused portion of each split spoon sample will be containerized, labeled, and stored for future analysis using more sophisticated procedures. These samples will be stored in a refrigerator located onsite in a secure area for the duration of the Phase I investigation.

As indicated in Figure 4, approximately 190 of the 236 sample locations are planned within areas of apparent disposal, spillage and processing. The other 46 sample locations will be selected to test for the presence of contamination in unknown or unsuspected processing areas. In addition, seven sample locations will be identified to measure levels in the active railroad beds, and 7 to measure offsite background levels, for a total of approximately 250 surface soil sample locations.

Samples obtained from beneath the parking areas of the railroad's automobile storage facility may require coring and patching, to allow penetration by the split-spoon sampler and prevent contamination of underlying soils with asphalt.

The results of the TC screening will be fully evaluated within 2 weeks after the completion of the field sampling.



NOTE: This is a composite map of features that at one time had been on the site. Many of these have since been dismantled or covered.

FIGURE 4
SURFACE SOIL SAMPLE LOCATIONS
MOSS AMERICAN

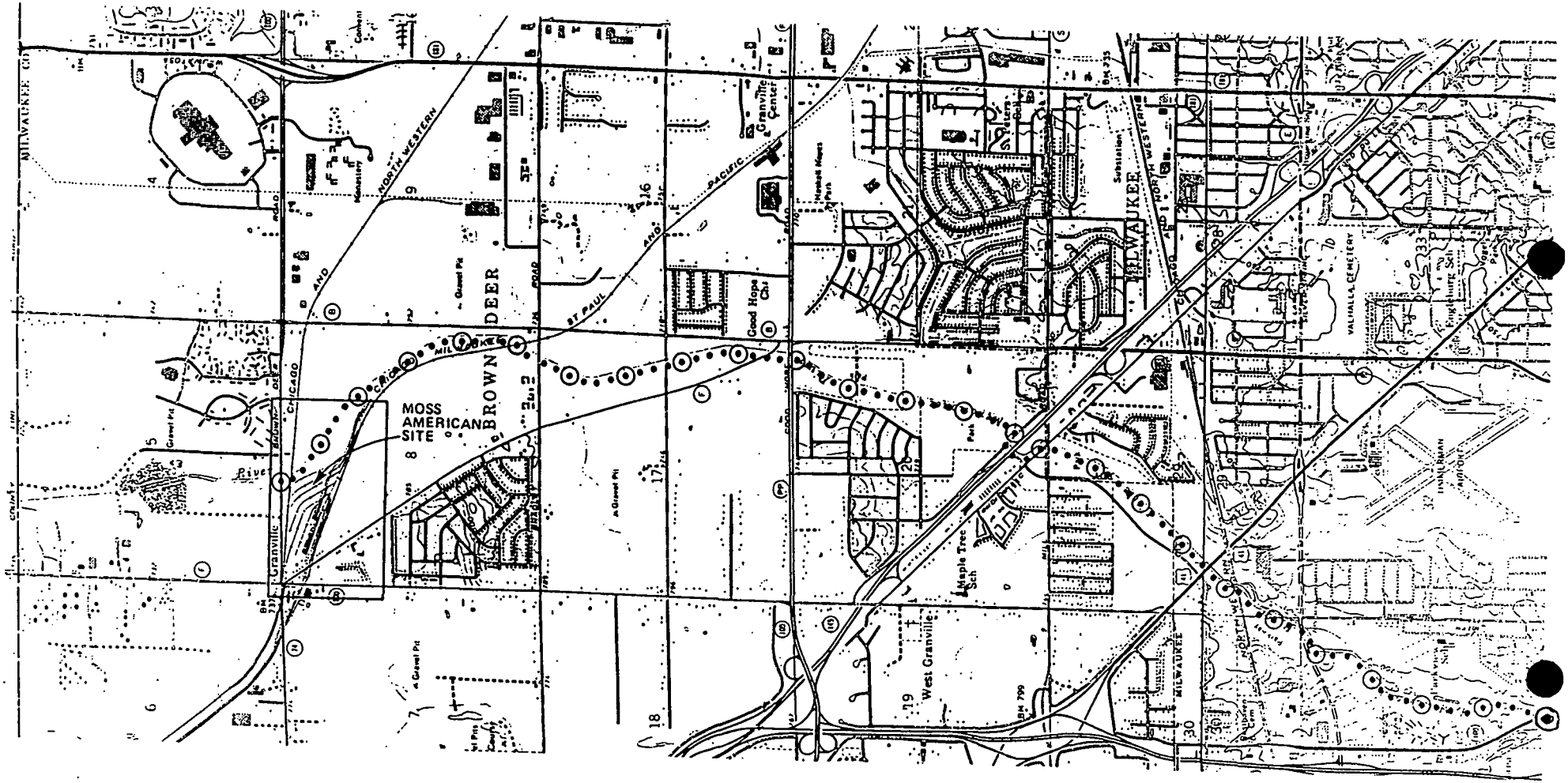


FIGURE 5
SEDIMENT SAMPLE LOCATIONS

This data will be used to select 60 surface soil samples to be taken from onsite storage and sent to the CH2M HILL Montgomery laboratory for GC/FID analysis.

Task S1--Initial Sediment Screening. Sediment samples will be obtained at and along profiled cross sections on the river. It is estimated that 26 such sections will be required for initial characterization and quantification of river sedimentation. As shown on Figure 5, general distribution of cross sections will be at approximately 1,200-foot stations, with modifications made based on the survey efforts of Task FM. Six samples will be taken from three locations at each section, for a subtotal of 156 samples. Three samples will be collected at a sediment depth of 0 to 1 foot, and three samples will be collected at a 1- to 2-foot depth. As in Task G1, samples will be scanned with an organic vapor analyzer (OVA) and photoionization detector (HNu) and descriptions of the samples logged. The OVA and HNu readings will be recorded and used to evaluate the potential for water transport of volatile contaminants. Visual descriptions and location of samples will be logged for use in quantifying the visibly identified free product (oils). Sediment samples will be submitted to the CSL for analysis by TC methods.

After these 156 samples have been collected and screened, the data will be evaluated. An additional 76 channel samples may be collected from sampling locations planned at 300-foot stations. Another 25 sampling locations will be selected for sampling river dredgings, floodplain deposits, and background locations upstream; the final locations to be based on field surveys and the initial TC results. The total number of sediment samples for TC screening is approximately 257.

The remaining portion of the sediment samples collected will be labeled and stored onsite in a refrigerator for future analysis. Upon completion of the TC screening and review of the results, approximately 60 unused portions of the sediment samples will be selected for GC/FID analysis by the CH2M HILL Montgomery laboratory.

Task G2--Confirmatory Soil Analyses. Upon completion of Task G1, and following review of the data, a field sampling team will return to the site to collect 16 additional surface soil samples. A minimum 10 onsite soil samples will be collected from selected areas representative of the most contaminated soil (based on GC/FID results). Six apparent background or noncontaminated samples will also be collected. These 16 samples will be submitted to the CLP for full scans of organic compounds, metals, and cyanide. To facilitate

evaluation of remedial alternatives in the Feasibility Study, additional analyses will include: carbon, hydrogen, sulfur, oxygen, nitrogen, moisture content, ash content, volatile matter, fixed carbon, total organic carbon (TOC), water soluble chlorides, heating value, flash point, and pH. At the request of the State of Wisconsin, soil samples will also be analyzed for dioxin (all isomers).

Task S2--Confirmatory Sediment Analyses. Similar to Task G2, a field sampling team will return to the site to collect 10 sediment samples and 6 background samples following Task S1 for submittal to the CLP for detailed analyses including full scans of: organic compounds, metals, and cyanide. The following parameter will also be analyzed to support the selection of remedial alternatives in the FS: carbon, hydrogen, sulfur, oxygen, nitrogen, moisture content, ash content, volatile matter, fixed carbon, TOC, water soluble chlorides, heating value, flash point, and pH. At the request of the State of Wisconsin, sediment samples will also be analyzed for dioxin (all isomers).

3.3 HYDROGEOLOGIC INVESTIGATIONS

The hydrogeologic investigations proposed for this Phase I RI are based on key assumptions derived from initial evaluation of existing data:

- The site is probably underlain by thick till deposits of low hydraulic conductivity such that deep migration of contaminants probably has not occurred.
- The horizontal hydraulic conductivity of overlying weathered till, alluvium and fill deposits is greater than that of the lower till, such that groundwater flow and contaminant migration is probably toward peripheral drainage ditches and the Little Menomonee River.
- The Little Menomonee River acts as a groundwater discharge zone such that all groundwater flow from the site eventually discharges to the river.

The purpose of the hydrogeologic investigations will be to (1) verify or modify interpretations of the assumed hydrogeological regime; (2) determine the extent and fate of groundwater contaminants derived from past site operations; and, (3) gather that information necessary to evaluate potential remedial measures to alleviate public health and environmental impacts resulting from groundwater contamination.

One of the objectives of the soil screening tasks discussed earlier will be a horizontal delineation of onsite source areas of contamination. These data will, however, be shallow in nature, and may require further investigation at depth to develop the three-dimensional distribution of the contaminants to support evaluation of alternative remedial actions. Determination of contaminated soil thicknesses will require soil boring operations. These will be accomplished along with other subsurface investigations required to evaluate the hydrogeologic regime.

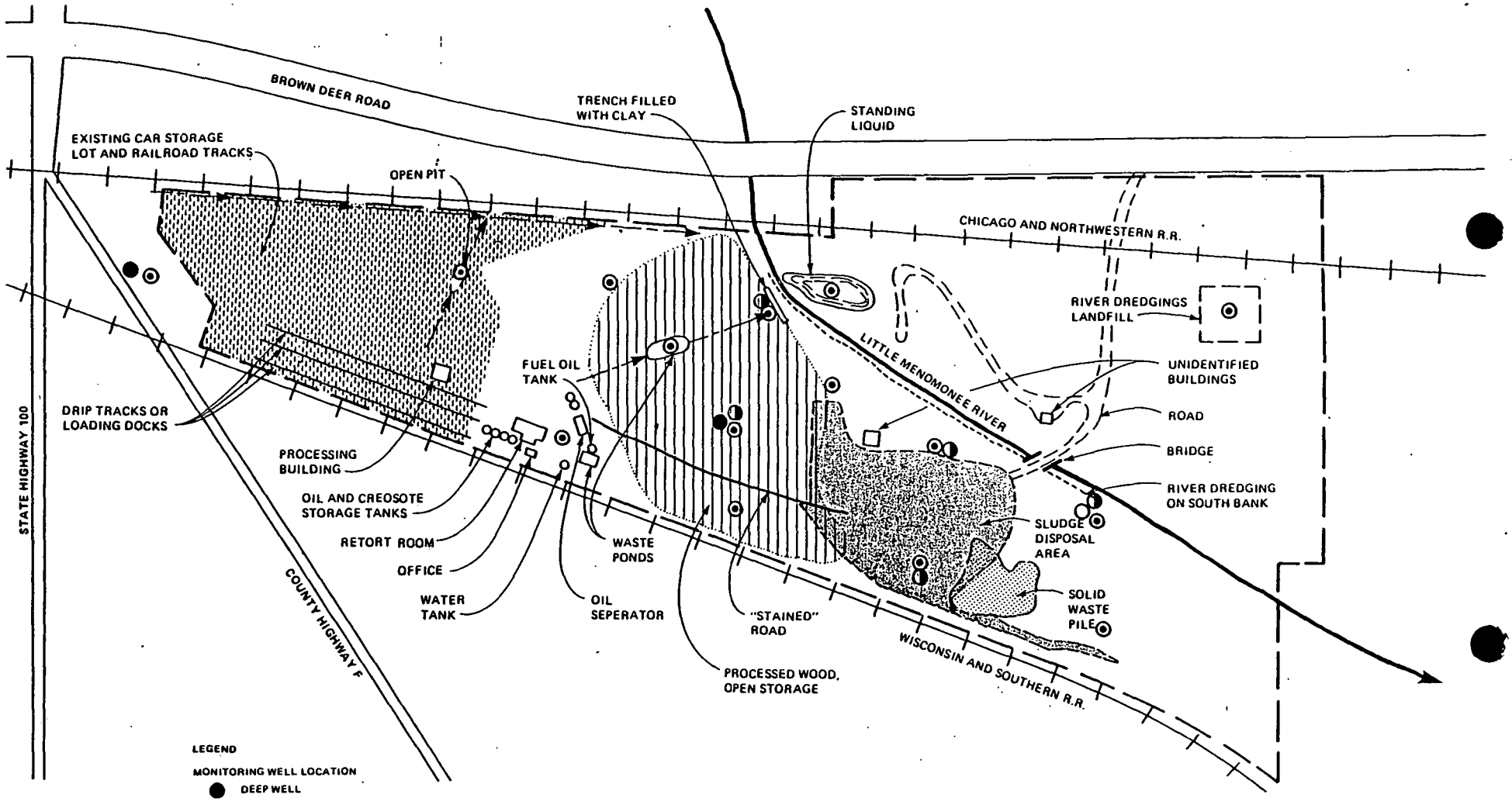
Task FI--Well Installation. While the till beneath the Moss-American site is estimated to range as deep as 180 feet, it is anticipated that groundwater flow and contaminant migration from the surface will not penetrate that depth, and that shallow investigations coupled with variable depth monitoring will accomplish verification and understanding of the flow regime. The proposed subsurface program, therefore, calls for three different depth intervals of investigation: shallow (0-15 feet), intermediate (30-35 feet) and deep (55-60 feet).

Because the thick deeper till is anticipated to be more homogeneous than the overlying weathered till, alluvial and fill deposits, variability of formation properties should be less at depths greater than 20 feet than at the surface. The shallow zone of fill, peat, alluvial deposits and variations due to past surface activities, on the other hand, is expected to present a greater variety of physical as well as chemical properties. This necessitates more numerous shallow groundwater monitoring locations than deep locations to interpret the extent of contamination as well as definition of the hydrogeologic regime.

Initially planned monitoring well locations are indicated in Figure 6. Twenty-three wells are currently planned with the depth distributions as follows:

- Fifteen--Shallow (0-15 feet)
- Five--Intermediate (30-35 feet)
- Three--Deep (55-60 feet)

Based on the results of the horizontal distribution of contamination determined from surface soil screening, final groundwater monitoring well locations will be determined. Some of the wells will be grouped in clusters to provide vertical gradient and contaminant level data at the same points. The three deep wells will be distributed to provide upgradient water quality data as well as more regional groundwater gradient conditions. Intermediate depth wells will be installed



NOTE: This is a composite map of features that at one time had been on the site. Many of these have since been dismantled or covered.

FIGURE 6
PROPOSED GROUNDWATER MONITORING WELLS
MOSS AMERICAN OAPP

to intercept potential contaminant migration in areas of heaviest soil contamination based on the results of GC/FID analysis of surface soil samples (Task G1). The shallow monitoring wells will be distributed to provide broad based data points for contaminated as well as clean areas, and to assist in quantification of contaminated groundwater. All well screen will be placed in sand or gravel seams, if encountered.

Wells will be installed using hollow-stem augers or spun-in casing. Split-spoon soil samples and HNu and OVA readings will be obtained during drilling. Continuous split-spoon samples will be obtained from the ground surface to a depth of 20 feet (or the bottom of the well). Split-spoon samples will be obtained at 5-foot intervals thereafter. In each cluster of wells, only the deepest well will be split-spoon sampled. Approximately 130 subsurface soil samples will be submitted to the CSL for TC screening. This data will be used to determine the vertical extent and level of contamination at various locations on the site. The remaining portions of the samples will be retained onsite for CLP analyses discussed under Task FS.

Well construction will consist of 2-inch diameter stainless steel screens and risers. All screened zones will be sand packed. Annular well seals will consist of 5 feet of bentonite clay above the well screens, and bentonite-cement slurry grout to the ground surface. Protective pipes will be grouted in and the wells equipped with locking caps.

Details of well installation, soil sampling, and HNu and OVA monitoring are provided in the Sampling Plan (Appendix D).

Task FS--Subsurface Soil Testing. Based on HNu and OVA readings, visual observations, and TC screening results an estimated 30 soil samples (collected during monitoring well installation) will be selected for submittal to the CLP for detailed analysis. These samples will be analyzed for organic compounds, metals, and cyanide. In addition, an estimated ten selected subsurface soil samples collected during drilling will be laboratory analyzed for particle-size distribution and Atterberg limit determinations.

Task FQ--Field Work Groundwater. Upon satisfactory development of all wells, in situ hydraulic conductivity (K) measurements will be obtained in each well by slug test methods. Hydraulic conductivities will be estimated from the particle-size distributions using Hazen's Law and the Kozeny-Carmen equation. The estimates from the well testing and the

particle-size distributions will be compared and correlated where possible.

One round of samples from groundwater monitoring wells will be obtained upon completion of aquifer testing. Static water levels will be recorded before any sampling activity. When practical, a minimum of five bore volumes of water will be purged from each well, using a peristaltic pump or a submersible pump, before collecting samples. Samples will be analyzed for pH, conductivity, and temperature in the field, and will be sent to the CLP for analysis of the organic (acid and base/neutral extractables, volatiles, PCB's, and pesticides) parameters and inorganic (metals and cyanide) constituents as defined in the User's Guide to the Contract Laboratory Program (1984). Monitoring well samples will also be analyzed for biochemical oxygen demand (BOD), chemical oxygen demand (COD), total organic carbon (TOC), sulfate, total phenols, total dissolved solids (TDS), total suspended solids (TSS), and alkalinity/acidity to evaluate the feasibility of remedial alternatives.

Samples to be analyzed for organic compounds will be collected using a pump and Teflon tubing. Samples to be analyzed for volatile organic compounds will be collected using a Teflon or stainless steel bailer. Two sets of samples will be collected for metals analysis. One set will be filtered in the field and the other will be unfiltered. A detailed description of groundwater sampling procedures is included in the Sampling Plan (Appendix D). Twenty-three groundwater samples, plus replicate samples and field blanks, will be submitted for analysis.

3.4 SURFACE WATER EVALUATION

Task FW--Field Work--Surface Water. Eight surface water grab samples will be collected from the following planned locations:

- In the drainage ditch on the north side of the site (1) where the ditch enters the site, and (2) just before the ditch discharges to the Little Menomonee River.
- In the Little Menomonee River (3) upstream of the site; (4) just below the confluence with the drainage ditch; (5) where the Little Menomonee exits the site; (6,7) at downstream locations to be selected after the river survey is complete; and, (8) just before the confluence with the Menomonee River.

A total of 10 water samples (including one replicate and one field blank) will be collected and submitted to the CLP. Samples will not be taken immediately after any major precipitation event to avoid masking or dilution of contaminant concentrations. All samples will be analyzed for organic parameters and inorganic constituents listed in the User's Guide to the CLP as well as BOD, TOC, COD, TDS, TSS and alkalinity/acidity, sulfates, and total phenols. Field measurements for pH, temperature, and conductivity will also be conducted.

3.5 EVALUATION OF FIELD AND LABORATORY DATA

Task DV--Data Validation. Quality assurance reviews of analytical data received from the U.S. EPA Central Regional Laboratory (CRL) Contract Laboratory Program, CH2M HILL Montgomery laboratory, the CSL, or subcontract laboratories will be evaluated by CH2M HILL. The appropriate use of the analytical data for RI/FS purposes will be evaluated based on the CRL or CLP QA/QC comments. Limitations of the analytical data will be presented and explained in the RI Report (Task RR).

Task DE--Data Evaluation. Data from the Phase I RI field tests will be summarized and evaluated. An appropriate data base system will be developed to allow for effective data comparisons and sorting capabilities based on factors such as type of sample, location, parameter analyzed, and concentration. Figures and graphic presentations will be developed to assist in data evaluation and explanations of contaminant concentration surveys hydrogeologic analyses. RI objectives will be reviewed to determine if the gathered data provide the specific information required by each task. Additional needs will be identified and incorporated into a Phase II RI work planning, if necessary.

Specific analyses and evaluations to be performed are:

- Generation of groundwater gradients for shallow, intermediate, and deep zones of the shallow aquifer and corresponding flow directions
- Identification of recharge and discharge areas
- Determination of vertical hydraulic gradients with respect to integrity of the till as a confining layer
- Generation of a site water balance

- Determination of the extent and level of groundwater and surface water contamination
- Estimation of surface and groundwater contaminant loadings to the Little Menomonee River
- Determination of the location and extent of contaminated soil onsite
- Determination of the location and extent of contaminated sediment in the river
- Identification of contaminant transport pathways and receptors
- Identification of general groundwater and surface water remedial technology constraints
- Identification of general soil and sediment remedial technology constraints

Task PH--Public Health Assessment. Based on the determination of potential contaminant migration to water resources, estimated contaminant loadings and potential for direct contact with contaminated soils and sediments; a public health assessment will be performed. The level of contaminants detected will be compared to acceptable limits and standards, and the needs for specific areas of remediation defined.

Task RR--RI Report. A draft technical report summarizing Phase I activities, results, and conclusions will be prepared. Six copies will be submitted to U.S. EPA and two copies to the Wisconsin Department of Natural Resources (WDNR). The draft report will provide documentation of data obtained for Phase I tasks, as well as data evaluation and identification of additional tasks and information needs for the Phase II investigations. Phase II RI work may be required, if it is determined that additional data are necessary as follows:

- Discovery of an environmental or health risk needing further clarification
- Determination that subsurface or river conditions are substantially different than originally assumed and must be investigated in more detail
- Determination that significant and discrete areas of contamination warrant delineation to develop

more cost-effective remedial technologies for those discrete problems

- To support selection of remedial technologies

After meeting with U.S. EPA and WDNR to review the Draft RI Report, a Final RI Report will be prepared, with 15 copies submitted to the U.S. EPA and 5 to the WDNR.

3.6 COMMUNITY RELATIONS

Task CR--Community Relations. Upon approval of the Work Plan, the Community Relations Plan previously prepared by CH2M HILL will be executed throughout the Phase I RI activities. The major objectives of the community relations program are to explain the Superfund process and how this applies to the Moss-American site, to provide mechanisms to inform the public of progress at the site, to provide opportunities for local input into the decisionmaking process, and to keep community leaders informed at key points during the process.

Task CS--Technical Support. Under this task the technical staff will assist in review of Community Relations documents and provide technical support at public presentations.

4.0 PROJECT ORGANIZATION AND RESPONSIBILITY

CH2M HILL has overall responsibility for all phases of the RI/FS. CH2M HILL will perform the field investigations and prepare the RI report. Subsequently, CH2M HILL will prepare the Feasibility Study. Project management will be provided by CH2M HILL.

The following responsibilities have been assigned for the project:

- Remedial Project Manager (RPM)
  Frank Rollins (U.S. EPA Region V)
- Site Manager (SM)
  Andrew Diefendorf (CH2M HILL)
- Regional Manager (RM)
  Mike Jury (CH2M HILL)
- Quality Assurance Manager (QAM)
  Greg Peterson (CH2M HILL)
- CH2M HILL Review Team Leader (RTL)
  Randy Videkovich

- Sample Team Leader (to be identified)
- Offsite Laboratory Operation
  U.S. EPA Contract Laboratory Program (CLP)
  and CH2M HILL Montgomery laboratory
- Onsite Laboratory Operations
  U.S. EPA Close Support Laboratory (CSL)
  managed by CH2M HILL
- System/Performance Audits
  CH2M HILL QA Manager (field), U.S. EPA EMSL--Las Vegas (CLP), Contract Project Management Section (CPMS), CRL--Montgomery Laboratory
- Special Analytical Services Requests Preparation
  CH2M HILL
- Review of Tentatively Identified Compounds
  CH2M HILL
- Review of Physical Soil Testing Data
  CH2M HILL
- QA/QC of CLP Data
  U.S. EPA Region V, Contract Project Management Section (CRL)
- QA/QC of SAS Data
  U.S. EPA Region V, Contract Project Management Section (CRL)
- CLP Data Completeness
  CH2M HILL
- QA/QC of CH2M HILL Montgomery Laboratory
  CH2M HILL
- QA/QC of CSL
  CH2M HILL

The PM and RTL will have the responsibility of assigning competent personnel to tasks which have been designated as CH2M HILL's responsibility. Primary responsibility for project quality rests with the SM. Independent quality assurance review is provided by the QA reviewers. A project organization chart is presented in Figure 7.

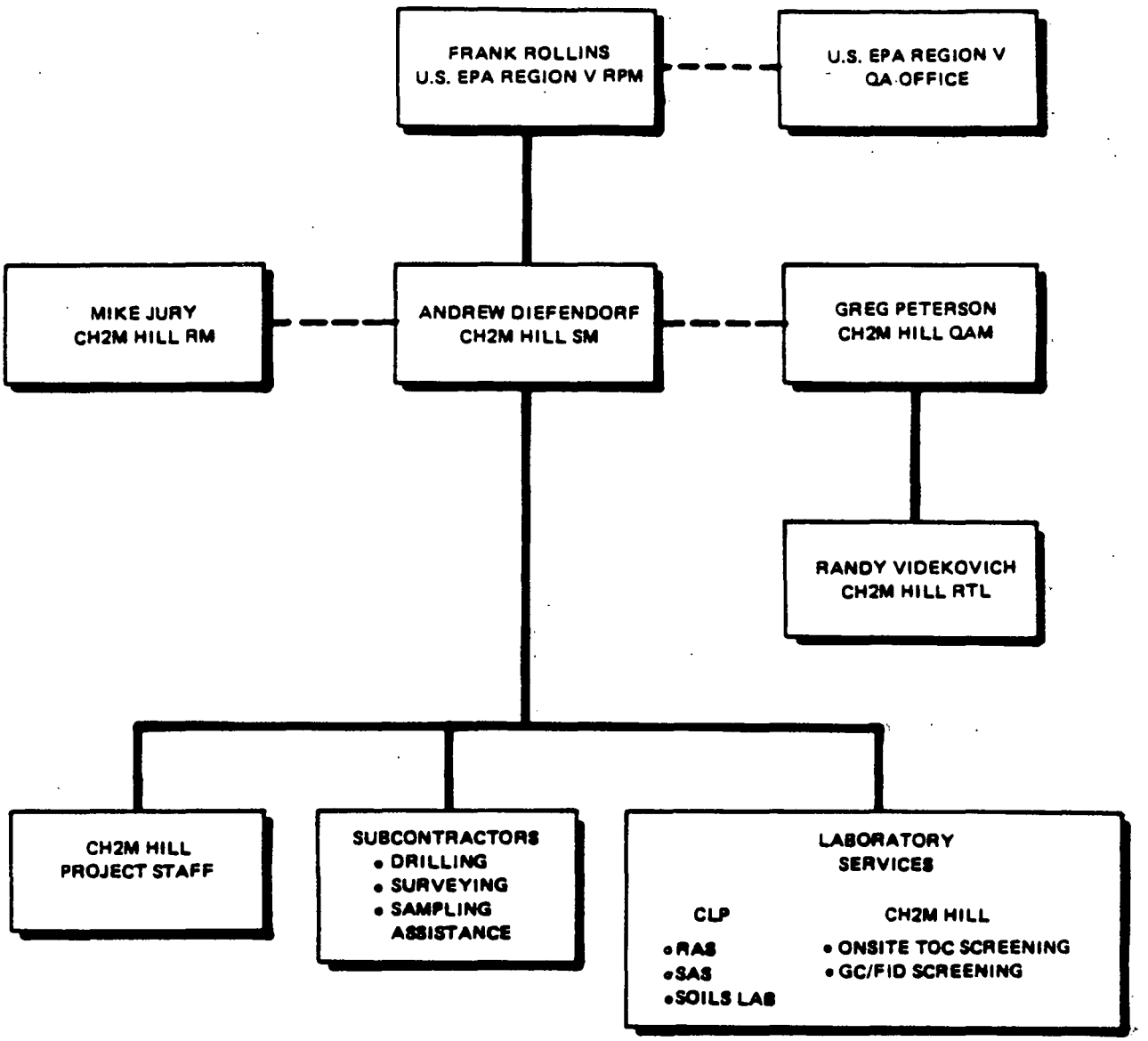


FIGURE 7
PHASE I RI
PROJECT ORGANIZATION
MOSS AMERICAN QAPP

5.0 SAMPLING PROCEDURES AND SAMPLE CUSTODY

Detailed sampling procedures are provided in the Sampling Plan included in this QAPP as Appendix D while sample custody protocols are included as Appendix E. Sample custody protocols for this project will be in accordance with the procedures detailed in NEIC Policies and Procedures, EPA-330/-9-78-001-R, revised June, 1985.

6.0 QUALITY ASSURANCE OBJECTIVES

The overall QA objective is to develop and implement procedures for field sampling, chain of custody, laboratory analysis and reporting that will provide legally defensible results in a court of law. Specific procedures to be used for sampling, chain of custody, calibration, laboratory analysis, reporting, internal quality control, audits, preventative maintenance and corrective actions are described in the appendixes of this Quality Assurance Project Plan.

6.1 FIELD QC AUDITS

Accuracy and reproducibility standards for survey activities will be consistent with those given in the standard surveying reference Manual of Surveying Instructions 1973 prepared by the Bureau of Land Management. Site and river surveying and mapping is a field activity where samples will not be collected, but involves measurements where quality assurance concerns are appropriate. The primary QA/QC objectives in activities where samples are not collected is to obtain reproducible measurements to a degree of accuracy consistent with the intended use of measurements and to document measurement procedures.

To assess the quality of data from field sampling efforts, replicate and field blank samples will be submitted. Blank samples prepared in the field will be analyzed to check for procedural contamination and/or ambient conditions at the site which are causing sample contamination. Preparation of replicate samples entails collecting a sample and dividing it into two portions for separate analysis. Replicate samples will provide information on data precision.

Depending on the extent and magnitude of contamination found, further exploration might be recommended, and at that time QA/QC aspects would also be revised for any additional sampling.

6.2 ACCURACY, PRECISION, AND SENSITIVITY OF LABORATORY ANALYSIS

The groundwater, surface water, soil, and sediment samples taken at the Moss-American site will be analyzed using the U.S. EPA Contract Laboratory Program (CLP).

The QA objectives for the CLP Routine Analytical Services (RAS) analyses are to achieve QC criteria stated in the CLP Invitation for Bid (IFB), number WA-85-J644/J680 for organic chemical analyses, and IFB number WB-85-J838/J839 for inorganic chemical analyses. Detection limits provided by CLP RAS for low and medium level soil and water parameters are given in Appendix F. QA objectives for parameters that are not part of the RAS are stated on the CLP Special Analytical Services (SAS) request forms and the appropriate attachments (defining laboratory protocol) are found in Appendixes G and H. QA objectives for field measurements, such as pH and specific conductance, includes proper operation of the field equipment based on the operator's manual instructions included in Appendix I.

7.0 ANALYTICAL SERVICES

Water samples, soil, and sediment samples will be analyzed using RAS or SAS of the CLP in addition to the initial screening performed by the CSL. Several field measurements will also be performed. QAPP elements for each are listed below and are documented in the references cited.

QAPP Element	RAS	SAS	Field Analysis
Calibration Procedures	PD	Appendixes G & H	Appendix I
Analytical Procedures	PD	Appendixes G & H	Appendix I
Internal QC	PD	Appendixes G & H	Appendix I
Data Reduction/ Validation	PD	Appendixes G & H	Appendix I
Performance/System Audit	PD	Appendixes G & H	Appendix I
Data Assessment	PD	Appendixes G & H	Appendix I
Accuracy/Precision Definitions	PD	Appendixes G & H	Appendix I
Corrective Action	PD	Appendixes G & H	Appendix I

PD = Predetermined in CLP, IFB Nos. WA-85-J644/J680 for organic chemical analyses and IFB Nos. WA-85-J838/J839 for metals and cyanide.

7.1 CLP ROUTINE ANALYTICAL SERVICES

Analytical and Calibration Procedures

All samples collected will be analyzed for Hazardous Substances List Organics by the CLP. All testing of soil, groundwater, surface water, and sediment samples will conform to the guidelines in the User's Guide to the U.S. EPA Contract Laboratory Program and to those specified in IFB's WA-85-J664/J680 for organics and WA-85-J838/J839 for metals and cyanide.

Computer-assisted library searches will be made to tentatively identify as many as 30 organic compounds in addition to those listed in the Sampling Plan (Appendix D). However, no more than 4 hours per sample will be spent in the search for the identity of unknowns. The three most matched compounds will be reported via a computer mass spectral library search. Positive peak identification requires at least a five major-peak match including the base peak and molecular ion peak. The relative intensities of these peaks should not vary by ± 20 percent compared to the suspected compound. Compounds still unidentified after 4 hours are labeled as UNKNOWN #XXX, where XXX is the scan number where the unknown appears. Purity should also be included.

Internal Quality Control

Internal quality control procedures for groundwater, surface water, sediment, and soil samples will follow the guidelines of the CLP specified in the IFB's WA-85-J664/J680 for organics and WA-85-J838/J839 for metals and cyanide. Field blanks and replicates will be collected to check for any sample contamination resulting from field sampling equipment and to check data precision, respectively.

Data Reduction/Validation and Reporting

Data validation will be performed by the CPM Section of the CRL to ensure the accuracy of data reporting and the acceptability of data reductions. Data handling standards guarantee the integrity of raw data and create a traceable audit trail from chain-of-custody through data reduction. The data base is assessed for numerical reasonableness and acceptability. Extremes are eliminated on the basis of review by qualified and experienced individuals. Validation procedures provide for the generation of defensible analytical data acceptable for use as evidence in legal proceedings. The raw data collected from project sampling tasks and used in

project reports will be appropriately identified and will be included in a separate appendix within the final report.

Data reduction will be documented so checks can be accurately made to monitor the validity of the reduction process. Data reduction includes all processes which change the values or numbers of data items. Documentation will address the reliability of computations, appropriateness of the model(s) as a framework for investigating the study questions and the overall correctness of the data reduction. Individuals responsible for work performed on data will date and sign (or initial) the generated material so questions about conclusions or methods can be quickly referenced and resolved.

Reports will record the performance of all tasks and results. Missing data will be explained and the validation of data will be demonstrated each time data are recorded, calculated, or transcribed. Internal checks will be made to uncover or avoid errors in the data collection, recording, or transfer process.

Performance/System Audit

Performance and systems audits for CLP, RAS, are the responsibility of the Support Services Branch, OERR, EPA and of EMSL--Las Vegas, EPA. Audits are performed as described in IFB's WA-85-J664/J680 for organics and WA-85-J838/J839 for metals and cyanide.

Data Assessment

Data assessment is the responsibility of CPMS, CRL. Data completeness will be checked by CH2M HILL and the SMO.

Accuracy/Precision Definitions

Accuracy and precision definitions for analyses performed by CLP, RAS, are listed in IFB No.'s WA-85-J664/J680 and WA-85-J838/J839.

Corrective Actions

If quality control audits result in detection of unacceptable conditions of data, the SPM will be responsible for developing and initiating corrective action. The RM and QAM will be notified if nonconformance is of program significance or requires special expertise not normally available to the project team. Corrective action may include:

- Reanalyzing samples if holding time criteria permit;
- Resampling and analyzing;
- Evaluating and amending sampling and analytical procedures; and
- Accepting data acknowledging level of uncertainty.

7.2 CLP SPECIAL ANALYTICAL SERVICES

Analytical and Calibration Procedures

The groundwater and surface water samples will be analyzed by the CLP using SAS protocols for BOD, COD, TOC, TDS, TSS, sulfates, total phenols, and alkalinity/acidity. Soil and sediment samples will be analyzed using SAS protocols for proximate analysis (moisture content, ash, volatile matter, and fixed carbon), ultimate analyses (carbon, hydrogen, sulfur, oxygen, and nitrogen), heating value, flash point, pH, TOC, water soluble chlorides, and dioxin (all isomers). In addition, selected subsurface soil samples will be analyzed for particle-size distribution and Atterberg limits. Analytical and calibration procedures for these analyses are specified in Special Analytical Services--Regional Request forms (Appendixes G and H).

Internal Quality Control

Quality control requirements for each of the CLP, SAS analyses are specified in Appendixes G and H. Field blanks and replicate samples will be collected and submitted to CLP, SAS, for analysis to determine if any sample contamination is due to field sampling equipment and to check data precision, respectively.

Data Reduction/Validation, and Reporting

Data validation will be performed by the CPM Section of the CRL to ensure the accuracy of data reporting and the acceptability of data reductions. Data handling standards guarantee the integrity of raw data and create a traceable audit trail from chain-of-custody through data reduction. The data base is assessed for numerical reasonableness and acceptability. Extremes are eliminated on the basis of review by qualified and experienced individuals. Validation procedures provide for the generation of defensible analytical data acceptable for use as evidence in legal proceedings. The raw data collected from project sampling tasks and used in

project reports will be appropriately identified and will be included in a separate appendix within the final report.

Data reduction will be documented so checks can be accurately made to monitor the validity of the reduction process. Data reduction includes all processes which change the values or numbers of data items. Documentation will address the reliability of computations, appropriateness of the model(s) as a framework for investigating the study questions and the overall correctness of the data reduction. Individuals responsible for work performed on data will date and sign (or initial) the generated material so questions about conclusions or methods can be quickly referenced and resolved.

Reports will record the performance of all tasks and results. Missing data will be explained and the validation of data will be demonstrated each time data are recorded, calculated, or transcribed. Internal checks will be made to uncover or avoid errors in the data collection, recording, or transfer process.

Performance/System Audit

System audits and required performance limits are specified for each CLP, SAS, analysis in Appendixes G and H.

Data Assessment

Data Assessment is the responsibility of CPMS, CRL. Data completeness will be checked by CH2M HILL and the SMO.

Accuracy/Precision Definitions

Accuracy and precision definitions are specified for each CLP, SAS, analysis in Appendixes G and H.

Corrective Actions

If quality control audits detect unacceptable conditions or data, samples should be reanalyzed if holding time criteria permit. The Program Coordinator of the Contract Project Management Section should be contacted if requirements are not met upon reanalysis of samples.

7.3 CH2M HILL MONTGOMERY LABORATORY SCREENING

Analytical and Calibration Procedures

Surface soil and sediment samples will be analyzed for PAH and phenolic compounds at the CH2M HILL Montgomery

laboratory. Analytical and calibration procedures for the analysis of these compounds are included in the GC/FID analysis SOP (Appendix C).

Internal Quality Control

Quality control requirements for this analysis are also specified in the GC/FID analysis SOP (Appendix C). Field blanks and field replicates will be analyzed as a check on decontamination procedures and data precision.

Data Reduction/Validation and Reporting

All data generated during this screening task will be initially validated by a qualified chemist, other than the analyst, before turning the data over to the engineering staff. This will ensure the accuracy of the data reporting and acceptability of the data reductions.

Then on a weekly basis the laboratory will turn over to the site manager all bench records and calculations for samples, blanks, replicates, spikes, standards, etc. The site manager will see to it that a qualified chemist, independent of the laboratory reviews the data.

Data handling standards guarantee the integrity of raw data and create a traceable audit trail from chain-of-custody through data reduction. The data base is assessed for numerical reasonableness and acceptability. Extremes are eliminated on the basis of review by qualified and experienced individuals. Validation procedures provide for the generation of defensible analytical data acceptable for use as evidence in legal proceedings. The raw data collected from project sampling tasks and used in project reports will be appropriately identified and will be included in a separate appendix within the final report.

Data reduction will be documented so checks can be accurately made to monitor the validity of the reduction process. Data reduction includes all processes which change the values or numbers of data items. Documentation will address the reliability of computations, appropriateness of the model(s) as a framework for investigating the study questions and the overall correctness of the data reduction. Individuals responsible for work performed on data will date and sign (or initial) the generated material so questions about conclusions or methods can be quickly referenced and resolved.

Reports will record the performance of all tasks and results. Missing data will be explained and the validation of data

will be demonstrated each time data are recorded, calculated, or transcribed. Internal checks will be made to uncover or avoid errors in the data collection, recording, or transfer process. The CPMS CRL may, at their discretion perform an audit of these data reviews. The data reviews will be summarized in the final RI report.

Performance/System Audits

System audits and required performance limits are specified in Appendix C.

Data Assessment/Completeness

Data Assessment will be performed by the CH2M HILL Montgomery laboratory staff before releasing the sample results to the data users. Data completeness will be monitored by the data users.

Accuracy/Precision Definitions

Accuracy and precision definitions are specified in the analytical procedure for the GC/FID analysis in Appendix C.

Corrective Action

If variability among multiple readings of replicate samples is judged excessive, instruments will be recalibrated (if necessary) and the measurement repeated. If variability remains unacceptable high and instruments fail to properly calibrate, the QAM will be notified.

7.4 CLOSE SUPPORT LABORATORY SCREENING

Analytical and Calibration Procedures

Surface soil, subsurface soil, and sediment samples will be analyzed for TC in the close support laboratory. Analytical and calibration procedures for the analysis of these compounds are included in TC screening procedures (Appendix B).

Internal Quality Control

Quality control requirements for this analysis are also specified in the Appendix B. Field blanks and field replicates will be analyzed as a check on decontamination procedures and data precision.

Data Reduction/Validation and Reporting

Chain-of-custody records will be completed by the sampling team. Samples will be relinquished to the CSL staff. The CSL sample documentation coordinator will then log all samples in a laboratory notebook. Samples will be maintained under the sample documentation coordinators custody until disposal. Samples will be kept in a locked secured area at the end of each day.

Following the completion of the analysis and validation process the unused portion of the sample will be placed in a Department of Transportation (DOT) approved 55-gallon drum. The drums will be labeled and temporarily stored onsite for later disposal. All sample aliquots used for analysis by the CSL will ultimately be discarded in a drum labeled "High Concentration Lab Wastes Only."

Should the CSL staff find any deficiencies in sample documentation or possibly confusing or conflicting information, they will report it immediately to the SMT for prompt correction or resolution.

Data Reduction. All data will originate from the CSL data books, or be recorded on CSL data sheets. The analytical records including, bench work sheets, data sheets and the strip charts will be kept in the CSL trailer throughout the duration of the investigation. Copies of this data will be made available for data validation purposes in accordance with the section "DATA VALIDATION." The actual analytical values produced by the CSL will be entered into a microcomputer using a Lotus-type data base by the attending analysts. The data base will also include information such as sample identity, sample type, date received and analyzed, analytical method and procedure used, and data validation status. All data will be stored and updated on a floppy disk. A backup of this disk will be made and continually updated.

CSL data will be reviewed and validated in accordance with the data validation process described under "DATA VALIDATION." The CSL data will be reported in accordance with the section "DATA REPORTING." Ultimately the data will be stored and archived along with the master project files at CH2M HILL's Milwaukee office.

Data Reporting. Upon completion of each days' batch of samples, all information pertaining to the samples will be entered into the data base. A copy of each day's data will be provided to the SMT for his review and distribution. It

will be the duty of the SMT to report this data to the FTL and the appropriate task leaders. Also, at the end of each day all technical and nontechnical events will be entered into a site diary.

At this point the data will not have passed the data validation check, as described in the section "DATA VALIDATION," and as such will be designated preliminary. Data will be validated by the CSL operations manager. Upon approval of the CSL operations manager, data will be clearly designated in the data base as "validated."

Quality Assurance/Quality Control. The CSL quality assurance (QA) protocol is designed to assure that data quality objectives for monitoring and measurement are met. The system integrates quality planning, quality assessment, and quality improvement. Elements of the overall QA program are described in CH2M HILL's REM IV quality assurance documents.

Documentation of corrective action steps will include: problem identification, investigation responsibility assignment, investigation, action to eliminate the problem, increased monitoring of the effectiveness of the corrective action, and verification of problem elimination.

Data Validation. An objective of the CSL is to provide timely data of an appropriate level of quality as defined by the DQO. To satisfy this objective, the CSL data will undergo two levels of review and validation.

The first level of review will be performed by the CSL staff, which generated the data, prior to its release to the SMT. This is the customary "double checking" of the data for its conformance with protocols detailed in this SOP and the analytical methods.

The second level of review is carried on outside of the CSL. Each week a batch of data copies along with copies of the CSL diary will be submitted to the regional CSL operations manager for data validation and checking of data reduction and reporting.

Data validation consists of the steps taken so that the reported results correctly represent the samples and the analyses performed. There are two basic validation activities:

- The checking of sample results and QC sample results to demonstrate that the analyses are within

prescribed criteria for precision, accuracy, completeness, method detection limit, understandability, and legibility.

- o The process checking of 10 percent of the numerical computations for correct data and correct reporting of data.

For this project, the data will be validated for consistency with the DQO and the items of importance in the analytical procedures used. In addition to numerical evaluation, the reviewer will evaluate the performance of the analytical system by reviewing the strip chart for base-line noise, drift, and relative peak height. Emphasis will also be placed on blanks duplicates, and matrix spike analysis.

The following data qualifiers will be used as a means to classify the data as to its conformance to QC requirements:

J = Quantitatively Suspect
R = Unable to calculate due to interference
U = Not detected at the specified detection limits

Calibration problems, blank contamination, poor spike recoveries, instrument noise, or sample matrix interferences are possible reasons for a "J" qualifier. After the data validation process, the data reviewer will return the data to the FTL. Validated data will be considered as final data. If operational problems become evident during the validation process, the CSL Operations Manager will be responsible to determine the source of the problem, develop a plan, take corrective action, and report the results of his investigation to the FTL.

Performance/System Audits

System audits and required performance limits are specified in Appendix B.

Data Assessment/Completeness

Data Assessment will be performed by the close support laboratory staff before releasing the sample results to the data users. Data completeness will be monitored by the data users.

Accuracy/Precision Definitions

Accuracy and precision definitions are specified in the analytical procedure for the TC screening in Appendix B.

Corrective Action

If variability among multiple readings of replicate samples is judged excessive, instruments will be recalibrated (if necessary) and the measurement repeated. If variability remains unacceptable high and instruments fail to properly calibrate, the QAM will be notified.

7.5 FIELD ANALYTICAL SERVICES

Analytical and Calibration Procedures

Groundwater and surface water samples will be analyzed for pH, specific conductance, and temperature. Permeability measurements will be made at all monitoring wells. Specific operating and calibration procedures for the pH and conductivity meters to be used in the field are contained in Appendix I. The procedure used in determining permeability is also given in Appendix I. Operation of the data logger, if required, used in determination of permeability will be in accordance with the manufacturer's recommendation as specified in Owner's Manual Hermit Environmental Data Logger, Model SE10008, In-Situ, Inc., Laramie, Wyoming, 82070.

Internal Quality Control

Field analyses are performed onsite and do not involve samples that are collected and retained. The primary QA/QC objective is to obtain reproducible measurements to a degree of accuracy consistent with limits imposed by analytical methodologies used and with the intended use of the data. Quality control procedures will be limited to checking the reproducibility of measurements by taking multiple readings and by calibration of instruments (where appropriate).

Data Reduction, Validation, and Reporting

All field recording sheets, instruments outputs, and worksheets for calculating results will be retained. Summarized raw data will be appropriately identified in reports and included in a separate appendix of the final RI report. CH2M HILL will perform data validation. Any method used for data reduction will be described and will be part of the data package.

Performance/System Audit

All instruments used in making field measurements will be regularly calibrated (where appropriate) as specified in Appendix I.

Data Assessment

The Sample Team Leader will assess data to assure QA/QC objectives are met.

Accuracy/Precision Definitions

No quantitative levels are specified.

Corrective Actions

If variability among multiple readings at a single site is judged excessive, instruments will be recalibrated (if appropriate) and the measurement repeated. If variability remains unacceptably high and instruments fail to properly calibrate, the SM will be notified.

8.0 QUALITY ASSURANCE REPORTS

No separate QA report for this project is anticipated. The final RI report and FS report will contain separate QA sections that summarize data quality information collected during the project.

Appendix A
EXISTING DATA

Appendix A-1
EXISTING SURFACE WATER DATA - LITTLE MENOMONEE RIVER

Sample location	Date	Sampler	Oil mg/l	Phenol mg/l	Hexane extractable mg/l	Comments
Brown Deer Rd.	2-22-68	WDNR		<.04		MP 5.9
Brown Deer Rd.	9-4-68	WDNR		0.30		MP 5.9
Moss Am. Outfall	2-22-68	WDNR		1.70		MP 5.8
Moss Am. Outfall	9-4-68	WDNR		4.00		MP 5.8
Wooden Bridge	2-22-68	WDNR		0.06		MP 5.2
Wooden Bridge	9-4-68	WDNR		0.30		MP 5.2
1	7-20-71	Rexnord	3	<0.01		
2	7-20-71	Rexnord	11	<0.01		
3	7-20-71	Rexnord	4	0.01		
4	7-20-71	Rexnord	3	0.02		
5	7-20-71	Rexnord	0	0.02		
6	7-20-71	Rexnord	2	0.01		
7	7-20-71	Rexnord	0	0.01		
8	7-20-71	Rexnord	1	0.02		
9	7-20-71	Rexnord	2	<0.01		
71-101	10-21-71	USACE		0.028	113	
71-103				0.040	118	

Appendix A-2
EXISTING GROUNDWATER QUALITY DATA

Sample location	Date	Sampler	Methylene Chloride extractables mg/l	Comments
772401	9-20-77	NEIC	4190	
772401	10-19-77	NEIC	99	
772407	10-19-77	NEIC	600	Majority of extractable material resembled highly weathered fuel oil
772413	9-20-77	NEIC	--	Not Detected
772413	10-19-77	NEIC	<2	
772419	10-19-77	NEIC	<2	
772431	9-20-77	NEIC	--	Not Detected
772431	10-19-77	NEIC	<2	
772440	9-20-77	NEIC	20	
772440	10-19-77	NEIC	9	
772452	10-19-77	NEIC	<2	Spring sample, 100 ft SW of 772440

Appendix A-3
EXISTING SEDIMENT SAMPLE DATA - LITTLE MENOMONEE RIVER

Sample location	Date	Sampler	Hexane Extractable g/kg of wet mud	Hexane Extractable g/kg of dry solids	Location
1/2	7-20-71	Rexnord	0.9	0.9	
1	7-20-71	Rexnord	0.2	0.2	
1 1/2	7-20-71	Rexnord	1.7	2.3	
2	7-20-71	Rexnord	25.1	53.5	
3	7-20-71	Rexnord	17.1	30.4	
4	7-20-71	Rexnord	5.4	9.2	
5	7-20-71	Rexnord	3.9	4.6	
6	7-20-71	Rexnord	35.4	77.2	
7	7-20-71	Rexnord	9.0	21.6	
8	7-20-71	Rexnord	2.6	4.2	
9	7-20-71	Rexnord	0.2	0.3	
11	10-16-72	Biotest	--	10.0	730 ft above Calumet Rd.
12	10-16-72	Biotest	--	3.0	730 ft above Calumet Rd.
13	10-16-72	Biotest	--	3.0	730 ft above Calumet Rd.
21	10-16-72	Biotest	--	6.0	530 ft above Calumet Rd.
22	10-16-72	Biotest	--	1.0	530 ft above Calumet Rd.
23	10-16-72	Biotest	--	6.0	530 ft above Calumet Rd.
31	10-16-72	Biotest	--	1.0	430 ft above Calumet Rd.
32	10-16-72	Biotest	--	1.0	430 ft above Calumet Rd.
33	10-16-72	Biotest	--	3.0	430 ft above Calumet Rd.
41	10-16-72	Biotest	--	18.0	330 ft above Calumet Rd.
42	10-16-72	Biotest	--	2.0	330 ft above Calumet Rd.
43	10-16-72	Biotest	--	2.0	330 ft above Calumet Rd.
51	10-16-72	Biotest	--	2.0	230 ft above Calumet Rd.
52	10-16-72	Biotest	--	3.0	230 ft above Calumet Rd.
53	10-16-72	Biotest	--	0.2	230 ft above Calumet Rd.
61	10-16-72	Biotest	--	2.0	130 ft above Calumet Rd.
62	10-16-72	Biotest	--	10.0	130 ft above Calumet Rd.
71	10-16-72	Biotest	--	13.0	30 ft above Calumet Rd.
72	10-16-72	Biotest	--	4.0	30 ft above Calumet Rd.
73	10-16-72	Biotest	--	2.0	30 ft above Calumet Rd.
81	10-16-72	Biotest	--	5.0	400 ft below sta's 71, 72 & 73
82	10-16-72	Biotest	--	23.0	400 ft below sta's 71, 72 & 73
83	10-16-72	Biotest	--	1.0	400 ft below sta's 71, 72 & 73

Appendix A-3

EXISTING SEDIMENT SAMPLE DATA - LITTLE MENOMONEE RIVER

Sample location	Date	Sampler	Hexane Soluble mg/kg	Phenol mg/kg	Percent Volatile Solids
71-102	10-21-71	COE	11050	0.024	17.4
71-104	10-21-71	COE	5470	0.019	32.2
71-105	10-21-71	COE	2910	0.026	11.7
71-106	10-21-71	COE	102180	0.038	33.9
71-107	10-21-71	COE	24080	0.012	16.9

Sample location	Date	Sampler	Oil and Grease Wet Basis mg/kg	Pyrene mg/kg	Fluorene mg/kg
LMR 0.1	6-24-75	USEPA	1330	5.6	--
LMR 2.5	6-24-75	USEPA	3890	31.0	--
LMR 4.0	6-24-75	USEPA	760	6.2	0.2
LMR 4.0	6-24-75	USEPA	4980	200.0	4.9
LMR 5.0	6-24-75	USEPA	7570	330.0	276.0
LMR 5.9	6-24-75	USEPA	730	6.7	--

Sample location	Date	Sampler	Biphenyl mg/kg	Dibenzo-furan mg/kg	Phenan-threne mg/kg
LMR 0.1	6-24-75	USEPA	--	--	--
LMR 2.5	6-24-75	USEPA	1.0	8.4	44.0
LMR 4.0	6-24-75	USEPA	--	--	2.0
LMR 4.0	6-24-75	USEPA	--	2.4	13.0
LMR 5.0	6-24-75	USEPA	42.0	220.0	440.0
LMR 5.9	6-24-75	USEPA	--	--	3.0

Sample location	Date	Sampler	Fluoran-thene mg/kg	Acenaph-thene mg/kg	Comments
LMR 0.1	6-24-75	USEPA	4.5	--	
LMR 2.5	6-24-75	USEPA	23.0	--	
LMR 4.0	6-24-75	USEPA	6.3	0.6	Bottom
LMR 4.0	6-24-75	USEPA	200.0	6.5	Bank
LMR 5.0	6-24-75	USEPA	305.0	300.0	
LMR 5.9	6-24-75	USEPA	6.1	--	

Appendix A-3
EXISTING SEDIMENT SAMPLE DATA - LITTLE MENOMONEE RIVER

Sample	location	Date	Sampler	Methylene Chloride Extractable mg/kg
772401	0-1.5 ft	9-19-77	NEIC	2,500
772401	5-6.5 ft	9-19-77	NEIC	--
772401	10-11.5 ft	9-19-77	NEIC	7,900
772401	15-16.5 ft	9-19-77	NEIC	2,400
772402	surface	9-19-77	NEIC	9,800
772402	62 cm	9-19-77	NEIC	15,400
772402	90 cm	9-19-77	NEIC	1,700
772403	surface	9-19-77	NEIC	9,200
772403	35 cm	9-19-77	NEIC	2,100
772403	50 cm	9-19-77	NEIC	11,100
772404	surface	9-19-77	NEIC	8,500
772404	32 cm	9-19-77	NEIC	4,300
772404	82 cm	9-19-77	NEIC	1,000
772405	surface	9-19-77	NEIC	5,900
772405	35 cm	9-19-77	NEIC	13,100
772405	65 cm	9-19-77	NEIC	4,200
772406	surface	9-19-77	NEIC	9,000
772406	mid-depth	9-19-77	NEIC	31,800
772406	73 cm	9-19-77	NEIC	3,800

Appendix A-3

EXISTING SEDIMENT SAMPLE DATA - LITTLE MENOMONEE RIVER

Sample	location	Date	Sampler	Methylene Chloride Extractable (Dry Weight) mg/kg
772407	0-1.5 ft	9-19-77	NEIC	23,700
772407	5-6.5 ft	9-19-77	NEIC	--
772407	10-11.5 ft	9-19-77	NEIC	--
772408	surface	9-19-77	NEIC	4,500
772408	50 cm	9-19-77	NEIC	4,400
772408	69 cm	9-19-77	NEIC	3,500
772409	surface	9-19-77	NEIC	800
772409	40 cm	9-19-77	NEIC	4,800
772409	69 cm	9-19-77	NEIC	4,300
772410	surface	9-19-77	NEIC	5,300
772410	42 cm	9-19-77	NEIC	3,100
772410	68 cm	9-19-77	NEIC	1,500
772413	5-6.5 ft	9-19-77	NEIC	1,700
772413	10-11.5 ft	9-19-77	NEIC	--
772414	surface	9-19-77	NEIC	71,000
772414	38 cm	9-19-77	NEIC	10,400
772414	55 cm	9-19-77	NEIC	34,300
772415	surface	9-19-77	NEIC	12,900
772415	50 cm	9-19-77	NEIC	--
772415	60 cm	9-19-77	NEIC	--
772416	surface	9-19-77	NEIC	20,000
772416	40 cm	9-19-77	NEIC	5,300
772417	surface	9-19-77	NEIC	1,300
772417	30 cm	9-19-77	NEIC	2,400
772417	60 cm	9-19-77	NEIC	2,200
772419	0-1.5 ft	9-19-77	NEIC	--
772419	5-6.5 ft	9-19-77	NEIC	--
772419	10-11.5 ft	9-19-77	NEIC	--
772420	surface	9-19-77	NEIC	40,200
772420	50 cm	9-19-77	NEIC	19,300
772420	75 cm	9-19-77	NEIC	13,600
772421	surface	9-19-77	NEIC	10,500
772421	47 cm	9-19-77	NEIC	3,800
772421	72 cm	9-19-77	NEIC	2,200
772422	surface	9-19-77	NEIC	4,900
772422	33 cm	9-19-77	NEIC	6,200
772422	73 cm	9-19-77	NEIC	2,200

Appendix A-3
EXISTING SEDIMENT SAMPLE DATA - LITTLE MENOMONEE RIVER

Sample	location	Date	Sampler	Methylene Chloride Extractable (Dry Weight) mg/kg
772426	surface	9-19-77	NEIC	72,900
772426	70 cm	9-19-77	NEIC	7,100
772427	surface	9-19-77	NEIC	279,000
772427	60 cm	9-19-77	NEIC	5,600
772430	surface	9-19-77	NEIC	2,400
772430	52 cm	9-19-77	NEIC	4,100
772430	60 cm	9-19-77	NEIC	--
772431	surface	9-19-77	NEIC	1,900
772431	30 cm	9-19-77	NEIC	2,800
772431	55 cm	9-19-77	NEIC	--
772431	5-6.5 ft	9-19-77	NEIC	--
772431	10-11.5 ft	9-19-77	NEIC	--
772432	surface	9-19-77	NEIC	11,200
772432	36 cm	9-19-77	NEIC	35,800
772432	58 cm	9-19-77	NEIC	--
772433	surface	9-19-77	NEIC	8,800
772433	40 cm	9-19-77	NEIC	--
772433	-- cm	9-19-77	NEIC	--
772434	surface	9-19-77	NEIC	9,100
772434	40 cm	9-19-77	NEIC	1,700
772434	70 cm	9-19-77	NEIC	3,700
772435	surface	9-19-77	NEIC	19,700
772435	40 cm	9-19-77	NEIC	1,000
772435	70 cm	9-19-77	NEIC	1,000
772436	surface	9-19-77	NEIC	10,200
772436	50 cm	9-19-77	NEIC	1,100
772436	80 cm	9-19-77	NEIC	15,600
772437	surface	9-19-77	NEIC	8,600
772437	32 cm	9-19-77	NEIC	11,000
772437	65 cm	9-19-77	NEIC	2,800
772438	surface	9-19-77	NEIC	1,900
772438	52 cm	9-19-77	NEIC	3,900
772438	70 cm	9-19-77	NEIC	3,100
772439	surface	9-19-77	NEIC	900
772439	38 cm	9-19-77	NEIC	25,300
772439	70 cm	9-19-77	NEIC	21,100
772440	10-11.5 ft	9-19-77	NEIC	--

TABLE 1

KERR-MCGEE/MOSS-AMERICAN SITE

SAMPLE LOCATIONS

(April 10, 1984)

SAMPLE NO.	DESCRIPTION
1	Sampled top 6 in. of soil of a bare spot in an area of coarse vegetation; location approximately the same as Well 07 in Reference 1.
2	Composite sample of mounds of black, oily material deposited on surface; location between fence and C & NW tracks, south of Well 01 in Reference 1.
3	Sampled top 12 in. of soil; location along fence line near southernmost access gate, in vicinity of Well 01.
4	Sampled a sediment from drainage ditch on north boundary of site, 85ft. west of discharge to Little Menomonee River.

TABLE 2

SAMPLE ANALYSES

SAMPLE NO.	METHYLENE CHLORIDE EXTRACTABLES, mg/kg (DRY WT)	% EXTRACTABLES
1	98,800 81,000 (90,000 AVE)	9.0
2	300,000 481,000 (390,000 AVE)	39.0
3	< 1,000	< 0.1
4	20,800 22,800 (22,000 AVE)	2.2

NOTES:

1. Procedure described on p. C-3 of Reference 1.
2. Analytical technique was verified by recovering known amounts of creosote added to the samples; the recovery varied from 65 to 109 percent.
3. Creosote free soil was extracted. Values from the analyses were less than 1000 mg/kg.

Appendix B
CLOSE SUPPORT LABORATORY (CSL) METHOD FOR SCREENING ANALYSIS FOR TOTAL CARBONS (TC) IN SOILS AND SEDIMENT

Attachment B-1: Determination of Water (Moisture) Content of Soil

CLOSE SUPPORT LABORATORY (CSL) METHOD FOR SCREENING ANALYSIS FOR TOTAL CARBONS (TC) IN SOILS AND SEDIMENT

1. SCOPE AND APPLICATION

1.1 This quantitative method is utilized for field screening of soils and sediments for total carbon as an indicator of organic constituent contamination at the Moss-American site. It is presented here as a means of rapidly characterizing field samples as part of the Field Sampling Plan.

1.2 Application of this method is limited to the screening analysis for TC in soils and sediment.

1.2.1 The method measures volatile, semi-volatile, and nonvolatile organic constituents present in a sample as a group parameter. Characterization of specific organic contaminants in duplicate or similar composite samples may occur at remote laboratories employing EPA approved testing protocols.

1.2.2 The data produced in the analysis allows the site investigation team to examine the relative degree of contamination associated with other sample constituents as found by the CSL or a remote laboratory. The TC content can be compared between samples spatially related to each other in vertical or horizontal planes and with background.

1.2.3 The method does not distinguish carbon from mineral sources or humus sources in soil from carbon of waste origin. Both calcium carbonate ($CaCO_3$) and ground pine needles, if present, would be measured as TC.

1.3 The method detection limit for the method is estimated to be 0.02 percent. The analytical range is estimated to be from 0.02 to 93 percent (for triphenylmethane). The method is not applicable to liquid samples such as water and

wastewaters having TCs in the parts per million range.

2. SUMMARY OF METHOD

2.1 The method presented here is a manual method adapted for field use from these sources: EPA/CE-81-1, EPA/Corps of Engineers Procedures for Handling and Chemical Analysis of Sediment and Water Samples, TC Procedure for Sediment Samples, May, 1981; American Society of Testing and Materials, ASTM E777-81, Vol. 11.04, 1986 edition; and Commercial Methods of Analysis, Snell and Biffen, p. 282-284, 1964.

2.2 In brief, a sample is ignited in a high temperature furnace in the presence of pure oxygen converting any carbon present to its combustion product, carbon dioxide. The carbon dioxide is absorbed on ascarite, sodium hydroxide impregnated mineral fibers. The carbon dioxide absorbed is determined gravimetrically using an analytical balance. The carbon dioxide weight gain is calculated to the carbon concentration in the sample. The configuration of the TC analysis train is shown in Figure 1.

3. INTERFERENCES

3.1 Carbon dioxide from the atmosphere and the oxygen can be absorbed by the ascarite and weighed. A trap is used to remove carbon dioxide prior to entering the furnace. An airtight system is maintained during the analysis to prevent gain from or loss to the atmosphere.

3.2 Moisture can be absorbed by the ascarite and weighed. Traps are used for drying the oxygen supply to the furnace and the combustion offgas from the furnace prior to ascarite absorption of carbon dioxide.

3.3 Sulfur dioxide from combustion can be absorbed by the ascarite and weighed. A chromic acid trap is used to convert sulfur dioxide to sulfate and absorb it.

3.4 Organics from equipment handling (fingerprints), airborne dust, cleaning residue, etc. can effect the results. Care will be used in cleaning. Forceps and tongs will be used in handling equipment.

3.5 Traces of analysis chemicals on the outer surfaces of glassware can absorb carbon dioxide and/or water. Outer surfaces will be clean and dry.

3.6 Sample drying or excessive handling may result in the loss of volatile organic constituents which contribute to the TC. Whenever possible, sample analysis will be on an "as received" basis minimizing sample handling to obtain representative aliquots to analyze.

3.7 Delayed analysis may result in loss of volatiles, biodegredation, or other physical-chemical changes effecting the TC results. In the event that analysis is delayed to the next day, samples will be refrigerated and a notation given in the Data Analysis Book.

4. SAFETY

4.1 All samples are assumed to be hazardous from their acidity and/or individual constituents within the sample. They may contain known or suspected carcinogens. Samples shall be handled with utmost care using good laboratory techniques in order to avoid harmful exposure. Samples shall be prepared in a fume hood.

4.2 Lab analysts shall wear lab coats, safety glasses, and surgical gloves when preparing and handling chemicals, standards, and samples. Safety equipment, including a fire extinguisher, first aid kit, eye wash, and chemical spill clean-up kit shall be available for use at all times during operation of the CSL.

4.3 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound shall be treated as a potential health hazard. Exposure must be reduced by whatever means available including those discussed above. Analysts shall read and observe guidance from the Material Safety Data Sheets (MSDS) provided by suppliers on initial purchase of reagents. While not as comprehensive as the MSDS, the following describes some important safety information.

4.3.1 Chromic acid is a combination of concentrated sulfuric acid and potassium dichromate. Even when cold, the solution can cause irritation and ulceration. It

is a powerful oxidizer for almost all organic substances. Pathways of exposure are oral and dermal. Potassium dichromate is a corrosive poison. Pathways of exposure of the dry salt are oral, dermal, and airway. Sulfuric acid is a corrosive liquid with a high affinity for water abstracting water from air and organic substances with simultaneous charring. Contact with the skin may result in severe burns and contact with the eyes may result in loss of vision. When diluting, add the acid slowly to water (not the reverse) to avoid boiling and spattering. When hot, the acid decomposes into sulfur trioxide, which is corrosive to the respiratory tract.

4.3.2 Ascarite is sodium hydroxide coated on a mineral fiber. Sodium hydroxide is corrosive to all tissues. Contact with the skin may result in severe burns or contact with the eyes may result in loss of vision. Pathways of exposure are oral, dermal, and airway. Inhalation may cause damage to the respiratory tract. Evidence of dermal contact is the slippery feel of saponified fats from the action of sodium hydroxide on skin material. Sodium hydroxide generates heat on dissolving with the potential for corrosive mists. Ascarite rapidly absorbs water and carbon dioxide from the air; spills will be evidenced by corrosive drops on spill surfaces. The mineral fibers may be carcinogenic. Avoid inhalation!

4.3.3 Magnesium perchlorate is a hygroscopic powder having strong oxidative properties. Pathways of exposure are oral, dermal, and airway.

4.4 Spilled or spent reagents require care in disposal. Ascarite absorber material shall be deposited in the drum marked "CSL Lab Wastes Only--Hazardous". Chromic acid and magnesium perchlorate shall be removed from their absorbers and shall be carefully diluted with water followed by flushing down the sink with large volumes of tap water. Acid spills require acid spill kit application and/or water. Spill

surfaces need a final cleaning with a damp towel followed with a dry one.

4.5 An oxygen cylinder is a potential bomb. The cylinder shall be secured by a chain such that the uncapped cylinder cannot fall over. As a combustion support, oxygen shall not be released to the lab atmosphere at rates exceeding the short term maximum that is required by the test, estimated to be 500 ml/minute.

4.6 The furnace combustion tube and boat with spent sample are at times above temperatures of 1000 degrees C. Severe burns occur at these temperatures. Care shall be taken to avoid direct contact with these hot surfaces. Use heat resistant gloves as a precaution against accidental contact. Handle the boat and spent sample with the furnace tool, forceps, and heat resistant surface (metal or transite plate). Have ice cubes ready for application on minor burns.

5. APPARATUS AND MATERIALS

5.1 Tube Furnace--Lindberg, single heating zone, split furnace controlling temperatures at 1000-1300 degrees C.

5.2 Balance--Sartorius, top-loading, electronic balance, 1500 gm capacity with 0.01 gm sensitivity for sample preparation.

5.3 Balance--Sartorius, analytical electronic balance, 0.0001 gm sensitivity, minimum 110 gm capacity.

5.4 Furnace Combustion Tube--McDanel, one reduced end, 22mm I.D. X 29mm O.D. X 30 cm L, maximum working temperature of 1400 degrees C.

5.5 Combustion Boats--Fisher, heavy gauge nickel boats, minimum 2 in number, 89 mm L X 16 mm W X 9.5 mm D, hole to facilitate removal at one end.

5.6 Combustion Boats--Fisher, ceramic disposable boats and boat covers essentially free of carbon, 95 mm L X 13 mm W X 11 mm D for use with oily samples.

5.7 Oxygen (O_2) Purifying Train--between the O_2 cylinder and the furnace.

5.7.1 Water Absorber--Gas drying cylinder containing indicating Drierite.

5.7.2 Carbon Dioxide (CO_2) Absorber--Drying tube or U-tube containing Ascarite.

5.7.3 Water Absorber--Drying tube containing Aquasorb.

5.8 Offgas Purifying Train--after the furnace.

5.8.1 Acid Trap--Fisher, bubble counter containing no reagent for preventing acid carry back into the furnace.

5.8.2 Water Absorber/Flow Rate Indicator--Fleming absorption bulb containing chromic acid.

5.8.3 Water Absorber--U-tube containing magnesium perchlorate, $Mg(ClO_4)_2$.

5.9 Carbon Dioxide Absorption Tower--Either a Nesbitt bulb for a 160 gm capacity balance, or a Stetser-Norton bulb for a 110 gm capacity balance.

5.10 Miscellaneous Items--Filter wool for fish aquaria filters, one quarter inch amber latex tubing, rubber stoppers, needle valve gas flow controller, forceps, timer, plate (transite or metal), furnace tool (straightened coat hanger with 4 mm right angle bend at one end), glass funnels and rods, pipets, mortar and pestle.

6. CHEMICALS, REAGENTS, AND GASES

6.1 Oxygen--99.99 percent pure, water less than 5 ppm, hydrocarbons less than 1 ppm, carbon monoxide less than 0.2 ppm, and carbon dioxide less than 0.5 ppm, metal-oxide semi-conductor grade or better, complete with two stage oxygen regulator or nitrogen regulator with an adapter.

6.2 Combustion Boat Reagents

6.2.1 Alundum--Reagent grade, 60 mesh, containing less than 0.0015 percent carbon.

6.2.2 Tin--Reagent grade, 20 mesh fine grain, containing less than 0.0015 percent carbon.

6.3 Absorber Reagents

6.3.1 Drierite--Self indicating, 10-20 mesh.

6.3.2 Ascarite--Self indicating, 20-30 mesh.

6.3.3 Aquasorb--Indicating phosphorous pentoxide pre-packed in drying tube.

6.3.4 Magnesium Perchlorate--Reagent grade, anhydrous, salt, granular.

6.3.5 Sulfuric Acid--Reagent grade, concentrated acid at 95-98 percent composition.

6.3.6 Potassium Dichromate--Reagent grade.

6.3.7 Chromic Acid--Made in the lab by adding some potassium dichromate to the concentrated sulfuric acid bottle and mixing in order to obtain a saturated solution having undissolved dichromate crystals in the bottom.

6.4 Carbon Standards

6.4.1 Dextrose--Reagent grade, anhydrous powder, 40.00 percent carbon.

6.4.2 Potassium Hydrogen Phthalate (KHP)--Reagent grade, primary standard, 47.05 percent carbon.

6.4.3 Performance Check--5 percent wt/wt concentration made with 12.5 gm dextrose and 87.5 gm Bentonite, 20-200 mesh, well ground and mixed with a mortar and pestle.

6.4.4 Reference Sample--National Bureau of Standards (NBS), Standard Reference Material (SRM), if available.

7. CALIBRATION

7.1 Calibration is not conducted as with instrumental methods because this method is a manual gravimetric determination.

7.2 System stability and performance checks are discussed under ANALYSIS PREPARATION and QUALITY ASSURANCE.

7.3 Balances will be calibrated using NBS Class S-1 weights.

8. SAMPLE PREPARATION

8.1 Mix the sample in its container with a clean glass rod to obtain homogenous, representative sub-samples. Perform this in the fume hood.

8.2 If necessary, break lumps into uniformly small size using a mortar and pestle located in the fume hood.

8.3 If rocks are present in the sample, remove them using forceps. Weigh the rocks and remaining sample recording the weights in both the Sample Log Book and the Analysis Data Book.

8.4 Details for sample receiving, storage, and disposal are described under a separately prepared standard operating procedure entitled SAMPLE HANDLING.

9. SYSTEM PREPARATION--See Figure 1

9.1 Connect copper tubing from the two-stage oxygen regulator to the needle valve gas flow control near the furnace.

9.2 Assemble the oxygen purifying train in the order listed in 5.7 using amber latex tubing from the needle valve through the train to a glass tube in a rubber stopper which fits the open end of the combustion tube.

9.2.1 Use filter wool at each end of the absorbing tubes to prevent movement of the absorbents, Ascarite or Drierite.

9.3 Assemble the combustion offgas purifying train in the order listed in 5.8 using amber latex tubing from the reduced end of the combustion tube through the train to the carbon dioxide absorber.

9.3.1 Carefully pipet 20 ml of chromic acid into the Fleming absorption bulb.

9.3.2 Use filter wool at each end of the U-tube to prevent movement of the magnesium perchlorate.

9.4 Assemble the carbon dioxide absorption tower

9.4.1 Place filter wool in the bottom 5 mm of the Nesbitt or Stetser-Norton bulb.

9.4.2 Using a glass funnel, add 5 mm depth of magnesium perchlorate on the top of the filter wool.

9.4.3 Mix the Ascarite by shaking in its original container. Using a glass funnel, add Ascarite on top of the magnesium perchlorate. Add to a depth such that the final weight of the absorber will be at least 10 gms less than the capacity of the analytical balance.

9.4.4 Using a glass funnel, add another 5 mm depth of magnesium perchlorate on top of the Ascarite.

9.4.5 Place 5 mm of filter wool on top of the magnesium perchlorate.

10. ANALYSIS PREPARATION

10.1 Turn on furnace to allow it to warm up to 1000-1300 degrees C. Leave the furnace on all the time to allow analysis without waiting for the furnace to warm up. Temperatures in excess of 1400 degrees C may damage the combustion tube.

10.2 Turn on the first stage oxygen cylinder valve to full tank pressure. Adjust the second stage reducing valve to 20 psi. Replace the oxygen cylinder when the tank pressure falls below 25 psi.

10.3 Close the needle valve control and check for leaks in the system from the oxygen cylinder to the needle valve.

10.4 Adjust the overall system oxygen flow rate to approximately 100 ml/minute using the needle valve control; the flow is indicated by a steady stream of bubbles in the chromic acid in the Fleming absorption bulb. Too much oxygen flow (500 ml/min) is indicated by violent bubbling at the chromic acid surface.

10.5 Analyze consecutive blanks consisting of a boat containing alundum in accordance with the procedure detailed in the following section ANALYSIS. System stability is shown by a carbon dioxide absorber weight difference of less than 0.0015 gm, the detection limit for a 2 gm sample.

10.6 Analyze consecutive standards in accordance with the procedure in the following section, ANALYSIS. Initial calibration shall be indicated by two consecutive TOC values within 2 percent of the theoretical value. Alternate dextrose and potassium hydrogen phthalate (KHP) standards every other day. Two percent represents plus or minus 0.80 percent for dextrose and plus or minus 0.90 percent for KHP.

11. ANALYSIS

11.1 Fill a boat with alundum. Use a nickel boat for routine determinations; use a disposable boat with cover for oily samples.

11.2 Use a spatula tip to make groove in the center of alundum along the length of the boat within 5 mm of each end.

11.3 Lay down a fine line of granular tin into the groove in such a manner that the tin particles touch each other. To economize weigh the tin for the first few analyses to determine the minimum amount of tin necessary for the type of boat used.

11.4 Weigh an appropriate size sample into the prepared boat distributing the sample along the length of the fine line of tin. Use the balance taring feature and record the weight to the nearest 0.1 mg (0.0001 gm).

11.4.1 Appropriate size samples should yield less than 0.200 gm of carbon dioxide:

grams sample	percent carbon	grams sample	percent carbon
2.0	0-1	0.25	10-20
1.0	1-2	0.15	20-35
0.75	2-7	0.10	over 35
0.50	7-10		

These sample sizes are recommended for reasons of representativeness, absorption efficiency, and absorber recharge minimization. Carbon dioxide yields greater than 0.200 gm may provide data of equivalent quality.

11.4.2 Samples may be weighed directly into the boat or into a weighing device (glassine,

plastic boat) if a quantitative transfer to the boat can be accomplished.

11.5 Lay down another fine line of granular tin on top of the sample in the same manner and amount as 11.3. It is not necessary to cover the sample with tin. Take care to avoid having the tin contact the side of the boat.

11.6 Crack open the carbon dioxide absorption tower (absorber) for 10 seconds to equilibrate to atmospheric pressure. Close and weigh, recording the weight to the nearest 0.1 mg (0.0001 gm). Place the absorber near the end of the combustion train.

11.7 Using forceps, place the boat in the cool portion of the furnace allowing sufficient clearance for the rubber stopper.

11.8 Insert the rubber stopper into the combustion tube and adjust the system flow rate as in 10.4.

11.9 After 30 seconds, open the carbon dioxide absorber. Connect the absorber to the tubing from the combustion gas purifying train taking care that the connection allows gas flow from the bottom of the absorber and venting to the atmosphere at the top of the absorber.

11.10 Remove the stopper and push the entire boat into the red hot zone of the tube using the furnace tool.

11.11 Insert the stopper, start the timer, and wait for combustion to occur. Generally, but not always, this occurs within 2 or 3 minutes. Evidence of combustion is reduced oxygen bubbles in the chromic acid followed by chromic acid rise in the Fleming bulb caused by the vacuum created by rapid oxygen consumption during combustion.

11.12 It is IMPERATIVE that a POSITIVE OXYGEN FLOW RATE BE MAINTAINED OVER THE ENTIRE COMBUSTION PERIOD estimated to be 2 minutes in duration. From the first moment that reduced flow is observed, add increasing amounts of oxygen by opening the needle control valve while trying to maintain a near constant bubble rate in the chromic acid. But, do not add so much oxygen as to cause violent bubbling.

11.12.1 Too little oxygen flow can result in chromic acid being pulled back into the tubing, safety trap, and, perhaps, the combustion tube itself. This will negate the test results. Additionally, a safety hazard will occur should the acid crack the combustion tube and fill the lab with acid fumes.

11.12.2 Too much oxygen flow can result in breaking a seal in the system or poor carbon dioxide absorption from the channeling in the ascarite.

11.13 When combustion ceases as evidenced by increased bubbling, reduce the oxygen flow to around 100 ml/minute as before.

11.14 Allow 5 minutes for complete carbon dioxide absorption. Use this time to weigh another boat and sample for the next determination.

11.15 Disconnect the carbon dioxide absorber, close its stopper, and place it by the analytical balance allowing 5 minutes for equilibration. Do 11.16 during this period.

11.16 Remove the boat from the combustion tube using the furnace tool to slide it onto a heat resistant plate. CAUTION! SEE SAFETY SECTION 4.6.

11.17 Crack open the carbon dioxide absorber for 10 seconds, close, and weigh as in 11.6.

11.18 Remove most of the combustion residue from the (first) boat using forceps. Traces of residue will not interfere with the next test because they are essentially free of carbon.

11.19 Replenish the alundum in the boat.

11.20 Continue analysis as in 11.2.

12. CALCULATIONS

For each soil sample, a fraction will be analyzed for TC and a fraction will be analyzed for moisture content. Percent moisture will be determined in accordance with ASTM Method D2216-80; please see attachment C-1 for procedure and calculation of moisture content. Once percent moisture has been calculated, analytical results for TC will be reported on a dry weight basis.

12.1 Percent carbon on an as-received or wet basis:

$$\%\ \text{Carbon}_{\text{wet basis}} = \frac{D}{A} \times 27.29$$

where (weights expressed in grams):

A = Sample weight
B = Final absorber weight
C = Initial absorber weight
D = B - C = weight of carbon dioxide

$$27.29 = \frac{12.011\ \text{(molecular weight of carbon)}}{44.011\ \text{(molecular weight of carbon dioxide)}}$$

12.2 Percent carbon on a dry weight basis:

$$\%\ \text{Carbon}_{\text{dry basis}} = \frac{E}{F}$$

where:

E = Percent carbon on a wet weight basis

$$F = \text{Fraction of solids} = \frac{\%\ \text{Solids}}{100}$$

$$= \frac{100 - \%\ \text{Moisture}}{100}$$

13. QUALITY ASSURANCE REQUIREMENTS

13.1 Detection Limit--0.02 percent carbon for 2 gm sample. If using a smaller sample and have less than 0.0015 gm of absorbed carbon dioxide, rerun the analysis with 2 or more gm of sample. If result is below the method detection limit report "BMDL."

13.2 Blanks--absorber weight gain of less than 0.0015 gm.

13.2.1 Initial Blanks--without tin as in 10.5.

13.2.2 Continuing Blanks--with normal proportion of tin performed at a frequency of 1 in 10 or 1 per day if less than 10 samples are analyzed.

13.3 Calibration Standards--plus or minus 2 percent of theoretical value for initial calibration as in 10.6 and final calibration performed at the end of each day's analyses.

13.4 Precision--duplicates of plus or minus 20 percent RPD performed at a frequency of 1 in 10 field samples or 1 per day if less than 10 samples are analyzed.

13.5 Accuracy--spikes of 75-125 percent spike recovery performed at a frequency of 1 in 10 field samples or 1 per day if less than 10 samples are analyzed.

13.6 Accuracy--reference sample of plus or minus 10 percent of NBS certified value performed at a rate of 1 in 20 field samples analyzed (not per day), if the reference material is available.

13.7 Accuracy--performance check sample (6.4.3) of plus or minus 10 percent of the calculated value performed at a rate of one per day.

13.8 If any or all parts of 13.2-13.7 are out of compliance, then the cause will be determined and corrective action taken. Record the out-of-compliance event and remedy in the data log book. Rerun all samples analyzed while the system was out of compliance.

13.8.1 Potential problems and their solutions--Check for system leaks. Recharge chromic acid, magnesium perchlorate, Drierite, Aquasorb, or Ascarite absorbers before their absorption capacities are exceeded. The carbon dioxide absorbent, Ascarite, changes from brown to white; recharge the absorber when the white absorption front reaches within 10 mm of the upper surface.

13.9 Quality assurance calculations:

13.9.1 Relative percent difference, RPD

$$\% \text{ RPD} = \frac{\frac{D1 - D2}{D1 + D2}}{2} \times 100$$

where:

D1 = First duplicate
D2 = Second duplicate

13.9.2 Spike recovery, R

$$\% \text{ R} = \frac{SSR - SR}{SA} \times 100$$

where (results in percent):

SSR = Spiked sample result
SR = Sample result
SA = Spike added = $\frac{\text{spike gm X F X 100}}{\text{spike gm + sample gm}}$

where:

F = 0.4000, fraction of carbon in dextrose,

or

F = 0.4700, fraction of carbon in KHP

14. TYPICAL DAILY ANALYTICAL SEQUENCE

14.1 Assume a 20 sample per day workload:

14.1.1 Initial blank without tin

14.1.2 Initial blank without tin (if needed as in 10.5)

14.1.3 Initial standard--Dextrose or KHP on alternating days

14.1.4 Initial standard as in 14.1.3

14.1.5 Field samples 1 through 10 (maximum)

14.1.6 Duplicate of one of the samples in 14.1.5

14.1.7 Spike of the same sample in 14.1.6

14.1.8 Daily performance check sample

14.1.9 Reference sample (if available and if needed for 1 in 20 requirement)

14.1.10 Continuing blank with tin

14.1.11 Field samples 11 through 20 (maximum)

14.1.12 Duplicate of one of samples in 14.1.11

14.1.13 Spike of the same sample in 14.1.12

14.1.14 Continuing blank with tin

14.1.15 Final standard--same as 14.1.3

Attachment B-1
DETERMINATION OF WATER (MOISTURE) CONTENT OF SOIL

AMERICAN NATIONAL STANDARD

ASTM D 2216 - 80

Standard Method for

LABORATORY DETERMINATION OF WATER (MOISTURE) CONTENT OF SOIL, ROCK, AND SOIL-AGGREGATE MIXTURES[1]

This standard is issued under the fixed designation D 2216; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers the laboratory determination of the water (moisture) content of soil, rock, and soil-aggregate mixtures by weight. For simplicity, the word "material" hereinafter refers to either soil, rock, or soil-aggregate mixtures, whichever is most applicable.

1.2 The water content of a material is defined as the ratio, expressed as a percentage, of the mass of "pore" or "free" water in a given mass of material to the mass of the solid material particles.

1.3 This method does not give true representative results for: materials containing significant amounts of halloysite, montmorillonite, or gypsum minerals; highly organic soils; or, materials in which the pore water contains dissolved solids (such as salt in the case of marine deposits). For a material of the previously mentioned types, a modified method of testing or data calculation may be established to give results consistent with the purpose of the test.

2. Summary of Method

2.1 The practical application in determining the water content of a material is to determine the mass of water removed by drying the moist material (test specimen) to a constant mass in a drying oven controlled at 110 ± 5°C and to use this value as the mass of water in the test specimen. The mass of material remaining after oven-drying is used as the mass of the solid particles.

3. Significance and Use

3.1 For many soil types, the water content is one of the most significant index properties used in establishing a correlation between soil behavior and an index property.

3.2 The water content of a soil is used in almost every equation expressing the phase relationships of air, water, and solids in a given volume of material.

3.3 In fine-grained (cohesive) soils, the consistency of a given soil type depends on its water content. The water content of a soil, along with its liquid and plastic limit, is used to express its relative consistency or liquidity index.

3.4 The term "water" as used in geotechnical engineering, is typically assumed to be "pore" or "free" water and not that which is hydrated to the mineral surfaces. Therefore, the water content of materials containing significant amounts of hydrated water at in-situ temperatures or less than 110°C can be misleading.

3.5 The term "solid particles" as used in geotechnical engineering, is typically assumed to mean naturally occurring mineral particles that are not readily soluble in water. Therefore, the water content of materials containing extraneous matter (such as cement, etc), water-soluble matter (such as salt) and highly organic

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[1] This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity and Density Characteristics of Soils.

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matter typically require special treatment or a qualified definition of water content.

4. Apparatus

4.1 *Drying Oven*, thermostatically-controlled, preferably of the forced-draft type, and maintaining a uniform temperature of 110 ± 5°C throughout the drying chamber.

4.2 *Balances*, having a precision (repeatability) of ±0.01 g for specimens having a mass of 200 g or less, ±0.1 g for specimens having a mass of between 200 and 1000 g, or ± 1 g for specimens having a mass greater than 1000 g.

4.3 *Specimen Containers*—Suitable containers made of material resistant to corrosion and a change in mass upon repeated heating, cooling, and cleaning. Containers with close-fitting lids shall be used for testing specimens having a mass of less than about 200 g; while for specimens having a mass greater than about 200 g, containers without lids may be used (Note 1). One container is needed for each water content determination.

NOTE 1—The purpose of close-fitting lids is to prevent loss of moisture from specimens before initial weighing and to prevent absorption of moisture from the atmosphere following drying and before final weighing.

4.4 *Desiccator*—A desiccator of suitable size (a convenient size is 200 to 250-mm diameter) containing a hydrous silica gel. This equipment is only recommended for use when containers having close-fitting lids are not used. See 7.4.1.

5. Samples

5.1 Keep the samples that are stored prior to testing in noncorrodible airtight containers at a temperature between approximately 3 and 30°C and in an area that prevents direct contact with sunlight.

5.2 The water content determination should be done as soon as practicable after sampling, especially if potentially corrodible containers (such as steel thin-walled tubes, paint cans, etc.) or sample bags are used.

6. Test Specimen

6.1 For water contents being determined in conjunction with another ASTM method, the method of specimen selection specified in that method controls.

6.2 The manner in which the test specimen is selected and its required mass is basically dependent on the purpose (application) of the test, type of material being tested, and the type of sample (specimen from another test, bag, tube, split-barrel, etc.). In all cases, however, a representative portion of the total sample shall be selected. If a layered soil or more than one soil type is encountered, select an average portion or individual portions or both, and note which portion(s) was tested in the report of the results.

6.2.1 For bulk samples, select the test specimen from the material after it has been thoroughly mixed. The mass of moist material selected shall be in accordance with the following table:

Sieve Retaining More Than About 10 % of Sample	Recommended Minimum Mass of Moist Specimen, g
2.0 mm (No. 10) sieve	100 to 200
4.75 mm (No. 4) sieve	300 to 500
19 mm	500 to 1000
38 mm	1500 to 3000
76 mm	5000 to 10 000

6.2.2 For small (jar) samples, select a representative portion in accordance with the following procedure:

6.2.2.1 For cohesionless soils, thoroughly mix the material, then select a test specimen having a mass of moist material in accordance with the table in 6.2.1. See Note 2.

6.2.2.2 For cohesive soils, remove about 3 mm of material from the exposed periphery of the sample and slice it in half (to check if the material is layered) prior to selecting the test specimen. If the soil is layered see 6.2. The mass of moist material selected should not be less than 25 g or should be in accordance with the table in 6.2.1 if coarse-grained particles are noted. (Note 2).

6.3 Using a test specimen smaller than the minimum mass indicated previously requires discretion, though it may be adequate for the purpose of the test. A specimen having a mass less than the previously indicated value shall be noted in the report of the results.

NOTE 2—In many cases, when working with a small sample containing a relatively large coarse-grained particle, it is appropriate not to include this particle in the test specimen. If this occurs, it should be noted in the report of the results.

7. Procedure

7.1 Select representative test specimens in accordance with Section 6.

7.2 Place the moist specimen in a clean, dry container of known mass (Note 3), set the lid securely in position, and determine the mass of the container and moist material using an appropriate balance (4.2). Record these values.

7.3 Remove the lid and place the container with moist material in a drying oven maintained at 110 ± 5°C and dry to a constant mass (Notes 4, 5, and 6).

NOTE 3—To assist in the oven-drying of large test specimens, they should be placed in containers having a large surface area (such as pans) and the material broken up into smaller aggregations.

NOTE 4—The time required to obtain constant mass will vary depending on the type of material, size of specimen, oven type and capacity, and other factors. The influence of these factors generally can be established by good judgment, and experience with the materials being tested and the apparatus being used. In most cases, drying a test specimen over night (about 16 h) is sufficient. In cases where there is doubt concerning the adequacy of drying, drying should be continued until the mass after two successive periods (greater than ½ h) of drying indicate an insignificant change (less than about 0.1 %). Specimens of sand may often be dried to constant mass in a period of about 4 h, when a forced-draft oven is used.

NOTE 5—Oven-drying at 110 ± 5°C does not always result in water content values related to the intended use or the basic definition especially for materials containing gypsum or other minerals having significant amounts of hydrated water or for soil containing a significant amount of organic material. In many cases, and depending on the intended use for these types of materials, it might be more applicable to maintain the drying oven at 60 ± 5°C or use a vacuum desiccator at a vacuum of approximately 133 Pa (10 mm Hg) and at a temperature ranging between 23 and 60°C for drying. If either of these drying methods are used, it should be noted in the report of the results.

NOTE 6—Since some dry materials may absorb moisture from moist specimens, dried specimens should be removed before placing moist specimens in the oven. However, this requirement is not applicable if the previously dried specimens will remain in the drying oven for an additional time period of about 16 h.

7.4 After the material has dried to constant mass remove the container from the oven and replace the lid. Allow the material and container to cool to room temperature or until the container can be handled comfortably with bare hands and the operation of the balance will not be affected by convection currents. Determine the mass of the container and oven-dried material using the same balance as used in 7.2. Record this value.

7.4.1 If the container does not have a lid, weigh the container and material right after their temperatures are such that the operation of the balance will not be affected by convection currents or after cooling in a desiccator.

NOTE 7—Cooling in a desiccator is recommended since it prevents absorption of moisture from the atmosphere during cooling.

8. Calculation

8.1 Calculate the water content of the material as follows:

$$w = [(W_1 - W_2)/(W_2 - W_c)] \times 100 = \frac{W_w}{W_s} \times 100$$

where:

w = water content, %,
W_1 = mass of container and moist specimen, g,
W_2 = mass of container and oven-dried specimen, g,
W_c = mass of container, g,
W_w = mass of water, g, and
W_s = mass of solid particles, g.

9. Report

9.1 The report (data sheet) shall include the following:

9.1.1 Identification of the sample (material) being tested, by boring number, sample number, test number, etc.

9.1.2 Water content of the specimen to the nearest 0.1 % or 1 %, depending on the purpose of the test.

9.1.3 Indication of test specimen having a mass less than the minimum indicated in Section 6.

9.1.4 Indication of test specimen containing more than one soil type (layered, etc).

9.1.5 Indication of the method of drying if different from oven-drying at 110 ± 5°C.

9.1.6 Indication of any material (size and amount) excluded from the test specimen.

10. Precision and Accuracy

10.1 Requirements for the precision and accuracy of this test method have not yet been developed.

Appendix C
GAS CHROMATOGRAPHY WITH FLAME IONIZATION DETECTION (GC/FID) STANDARD OPERATING PROCEDURE

Prepared by CH2M HILL, Inc.
Montogmery, Alabama for the
Determination of PAH's and
Phenolic Acids in Soil Matrix

THE DETERMINATION OF POLYNUCLEAR AROMATIC HYDROCARBONS AND PHENOLIC ACIDS IN SOIL MATRIX

1. Scope and Application

1.1 This method uses capillary gas chromatography with flame ionization detection (GC/FID) to screen soil samples for the presence of selected polynuclear aromatic hydrocarbons (PAHs) and phenolic acids. The following compounds can be determined by this method.

PAH Compounds	CAS No.
Naphthalene	91-20-3
Acenaphthylene	208-96-8
Acenaphthene	83-32-9
Fluorene	86-73-7
Phenanthrene	85-01-8
Anthracene	120-12-7
Fluoranthene	206-44-0
Pyrene	129-00-0
Benzo(a)anthracene	56-55-3
Chrysene	218-01-9
Benzo(b)fluoranthene	205-99-2
Benzo(k)fluoranthene	207-08-9
Benzo(a)pyrene	50-32-8
Indeno(1,2,3-cd)pyrene	193-39-5
Dibenzo(a,h)anthracene	53-70-3
Benzo(g,h,i)perylene	191-24-2
Phenolic Compounds	
Phenol	108-95-2
2-Chlorophenol	95-57-8
2-Nitrophenol	88-75-5
2,4-Dimethylphenol	105-67-9
2,4-Dichlorophenol	120-83-2
4-Chloro-3-methylphenol	59-50-7
2,4,6-Trichlorophenol	88-06-2
2,4-Dinitrophenol	51-28-5
4-Nitrophenol	100-02-7
4,6-Dinitro-2-methylphenol	534-52-1
Pentachlorophenol	87-86-5

1.2 This method is a quick and efficient determination of the compounds listed above. Compound identifications are based upon retention times only. And for this reason, compound identifications should be supported by an additional qualitative technique. EPA Method 625 provides a gas chromatography/mass spectrometer (GC/MS) analysis which may be used to confirm results produced by this screening method.

1.3 The method detection limits (MDLs) for the various compounds have not been determined. The MDL for each compound will certainly be dependent upon the nature of interferences co-extracted from the matrix. For soils not heavily contaminated, most compounds are detectable at the one part per million level or lower.

1.4 The use of this method must be restricted to those analysts experienced with capillary gas chromatography and the use of multiple internal standard analysis.

2. Summary of the Method

2.1 Approximately one gram of soil is weighed into a tared 15-mL screw-cap vial already containing approximately 10 grams of anhydrous sodium sulfate. The soil and drying salt are mixed with a spatula until a sandy texture is achieved. This mixing process helps offer a large soil surface area to the solvent added in the next step. Five mL of methylene chloride containing surrogates is added to the vial. The vial is capped and shaken vigorously for one minute.

2.2 The phenolic acids may be segregated from the total soil extract by partitioning them into basic water. The soil extract is transferred into a 125-mL separatory funnel containing 50 mL of basic water (pH 11-12). The transfer is completed in such a way as to control the amount of organic liquid phase in the separatory funnel (50:10 H_2O:CH_2Cl_2). Phenols are partitioned into the aqueous phase with one equilibration in the separatory funnel. After separation of the liquid phases, the organic phase (a base/neutral fraction) may be retained for further cleanup and PAH analysis or it may be discarded if the PAH analysis is not required.

2.3 Phenolic acids are extracted from the aqueous phase as an acid fraction. To achieve good acid recoveries the aqueous phase is adjusted to pH 2 or lower, and then ten grams of sodium chloride are dissolved. Finally the aqueous phase is extracted twice in series using 50 mL of fresh methylene chloride each time. The combined acid extract is concentrated to low sample volume (approximately 1 mL) in readiness for GC/FID analysis.

2.4 A PAH fraction may be prepared for analysis by claiming the organic liquid phase from the first separatory funnel equilibration at high pH (see 2.2 above). This base/neutral fraction should be subjected to additional silica gel column cleanup before PAH analysis by GC/FID.

2.5 Complex soil samples should have the PAH fraction and the phenolic fraction analyzed separately. For relatively clean soils however, the method does provide instrument conditions for simultaneous analysis of the PAHs and the phenolic acids.

3. Safety

3.1 The hazard of each reagent used in this method is not accurately known. Exposure to the chemicals should be held to the smallest practical level. Safety data sheets for all materials must be made available to the personnel involved in the chemical analysis.

3.2 All parameters in this analysis are designated as priority pollutants by the Environmental Protection Agency. Additionally, any unfamiliar soil sample may offer dangerous native contents beyond the list of target parameters.

4. Apparatus and Materials

4.1 Samples should be collected in the field in accordance with the quality assurance project plan. Sample containers should be glass or similar inert material which will not offer interferences for the analysis.

4.2 Glassware

4.2.1 Screw-cap vial, 15-mL size (4 dram), teflon-lined cap.

4.2.2 Separatory funnel, 125-mL size, Teflon stopcock.

4.2.3 Concentrator tube, Kuderna-Danish, 10-mL size, graduated, ground glass joint.

4.2.4 Evaporative Flask, Kuderna-Danish, 500-mL size, ground glass joints.

4.2.5 Snyder column, Kuderna-Danish, three-ball macro, ground glass joint, floodless type.

4.2.6 Snyder column, Kuderna-Danish, two-ball micro, ground glass joint, floodless type.

4.2.7 Screw-cap autosampler vials, 2-mL size, teflon-lined cap.

4.2.8 Graduated cylinder, 100-mL and 10-mL sizes.

4.2.9 Centrifuge tubes, 40-mL size.

4.3 Centrifuge, table top model. (Damon/IEC Division, IEC HN-SII Centrifuge)

4.4 Boiling Chips, 10/40 mesh, heat at 400 °C for 30 minutes before use to insure freedom from contamination.

4.5 Heated water bath, steam delivery and temperature should be controllable, concentric ring covers for openings.

4.6 Polypropylene filtration column, 6-mL size, 20-um polyethylene frit. Stopcock. (J.T.Baker 7121-6 and 7241-0)

4.7 Analytical Balance, large tare capability desirable.

4.8 Gas Chromatographic System, FID, split/splitless capillary injector and pneumatics, digital electronic integrator and data system for processing chromatographic data.

4.9 Fused silica capillary column, 5% phenyl 95% methylsilicone bonded stationary phase, 20 meters in length, 0.32 mm inside diameter, 0.25 um film thickness. A 60-meter length may be purchased and cut into desirable lengths. (DB-5, J&W Scientific)

5. Reagents

5.1 Blank water - Blank Water is that which does not produce an interference at or above the method detection limit for any target parameter.

5.2 Methylene Chloride and n-Hexane, pesticide quality or equivalent.

5.3 Acetone, technical grade or equivalent. For cleaning glassware.

5.4 Sodium sulfate, granular, anhydrous. Purify by heating in a shallow tray for four hours at 400 °C.

5.5 Silica Gel, 100/200 mesh. Activated at 200 °C for 12 hours in a shallow tray loosely covered with aluminum foil. (Fisher, S679 or equivalent)

5.6 Sodium Hydroxide solution, 10N. Dissolve 40 grams NaOH in reagent water and dilute to 100 mL.

5.7 Hydrochloric Acid, concentrated, ACS reagent grade.

6. Standards

6.1 Concentrated stock standard solutions may be prepared from primary neat materials or may be purchased as primary stock solutions.

6.1.1 A 16-component PAH standard with each component at 2000 ug/mL may be purchased from Supelco: 4-8905.

6.1.2 An 11-component phenolic acids standard with each component at 2000 ug/mL may be purchased from Supelco: 4-8904.

6.1.3 A concentrated stock solution of each surrogate and each matrix spike compound may be prepared from the neat material. Weigh approximately 0.0100 grams to the nearest 0.1 mg and dissolve in the appropriate amount of methylene chloride to achieve a 1.00 mg/ml concentration. If the original compound purity is 95% or better, then the measured mass should be used without correction to calculate the amount of solvent for the standard. A graduated 10-mL pipet may be used to deliver the solvent. Caution: Disposable pipets may not offer the accuracy needed here.

6.1.4 A 5-component internal standard solution may be prepared from the neat materials by weighing the first compound and dissolving in solvent as described above (see 6.1.3). *Most* of the 1-component solution just prepared may be used in place of pure solvent to dissolve the second weighed component. Similarly, *Most* of the 2-component solution just prepared may be used to dissolve the third weighed component. This procedure is repeated two more times before all five components are in a common solution at 1.0 mg/mL each. This approach to preparing a multicomponent solution is especially useful when small amounts are being weighed accurately, and uniform concentrations are desired near the saturation level.

6.2 Working solutions for surrogate spiking, matrix spiking, instrument calibration, and internal standard spiking should all be prepared in methylene chloride solvent.

6.2.1 A working standard should be prepared for spiking surrogates into the sample. The concentrated stocks mentioned earlier (see 6.1.3) are used to prepare the surrogate fortification solution.

Surrogate Compound	Concentration
2,3-Benzofuran	50 ug/mL
2,4,6-Tribromophenol	100 ug/mL
Terphenyl-d14	50 ug/mL

6.2.2 A working standard should be prepared for spiking target compounds into the sample matrix. The the concentrated stocks mentioned earlier (see 6.1.3) are used to prepare the matrix spiking solution.

Matrix Spike Compound	Concentration
Phenol	100 ug/mL
4-Nitrophenol	100 ug/mL
Pentachlorophenol	100 ug/mL
Naphthalene	50 ug/mL
Acenaphthalene	50 ug/mL
Pyrene	50 ug/mL

6.2.3 Instrument calibration standards may be prepared by mixing concentrated stock solutions of the surrogates, PAH's, and phenolic acids. Note: It is good practice to add internal standards to calibration solutions using the same fortifying technique as that used with sample extracts (see 7.4).

7. Instrument Calibration

7.1 Figure 1 is an example chromatogram of the instrument calibration standard. Recommended instrument conditions are included in the legend.

7.2 Instrument calibration and sample analysis must be performed using multiple internal standards. The five internal standard (IS) compounds listed in Table 1 are recommended to establish both relative retention times (RRT) and relative response factors (RRF). Each internal standard appearing in a chromatogram will establish a primary search window for those target compounds nearby in the chromatogram. Relative retention times are calculated using equation 1.

$$RRT = R^{target}/RI^{IS} \qquad \text{Eq. 1}$$

Relative response factors are calculated as follows:

$$\text{Absolute Response Factor} = RF = \text{Amount/Area} \qquad \text{Eq. 2}$$

$$\text{Relative Response Factor} = RRF = RF^{target}/RI^{IS} \qquad \text{Eq. 3}$$

Note: Amount in equation 2 refers to the mass (e.g. ug) of compound mixed into the solution injected.

7.3 Table 2 is a peak listing for the calibration standard in Figure 1. Notice that both RRT's and RRF's are based upon a close internal standard. Target Quantifications must be based upon relative response to a nearby internal standard to minimize the error associated with uncontrollable changes in volatility discrimination.

7.4 A five-point calibration should be employed, including standards at 50 ppb, 150 ppb, 500 ppb, 750 ppb, and 1 ppm levels. The samples extracts will be stored in autosampler vials, therefore, it is recommended that calibration standards be prepared for injection in these vials also. A known mass of each calibration compound is added to an autosampler vial (see 6.2.3 for multicomponent stock), then any additional solvent if needed, and finally 100 uL of internal standard solution (see 6.1.4). Cap and shake the vial for a uniform solution. Label the vial for contents expressed in mass units only (e.g., micrograms of each compound added to the concentration of any compounds in the vial.

7.5 Analyze each level of calibration standard. For each compound, tabulate the RRF at each level (see equation 3). If the RRF over the working range is constant enough (less than 25% relative standard deviation), then the midpoint value may be used for calculations. Alternatively, the calibration data may be used to construct a calibration curve of relative response against relative amounts (the relative amounts in the autosampler vial are the same as relative amounts injected).

7.6 Check the continuing validity of initial calibration at least once daily. The midpoint level of initial calibration must be injected as the continuing calibration check standard. Each of six calibration check compounds (CCC) listed in table 3 must be carefully evaluated for a shift in RRF. Equation 3 must be used to evaluate changes in the RRF since initial calibration.

$$\pm \%D = (RRF^{daily} - RRF^{initial})(100)/RRF^{initial} \qquad \text{Eq. 4}$$

Any percent difference (%D) greater than 50 for the CCCs requires new initial calibration according to paragraphs 7.4 through 7.5 above.

8. Quality Control

8.1 Every laboratory should operate a formal quality control program. Before using this method, laboratory capability must be demonstrated by analyzing spiked samples. Ongoing performance checks may be accomplished by analyzing matrix spike (MS) and matrix spike duplicates (MSD) at a frequency which meets the needs of quality assurance project plans. Method blanks should be extracted with every set of samples.

9. Sample Extraction

9.1 Deliver approximately 10 grams of anhydrous sodium sulfate into a 15-mL screw-cap vial. Tare the vial and contents before weighing approximately 1 gram of soil sample into the same vial. Record the weight of the sample to the nearest 0.01 gram.

9.2 Mix the soil and drying salt with a spatula until a sandy texture is achieved. Insufficient mixing of the drying salt with high moisture samples may result in poor extraction efficiency.

9.3 Fortify the blended sample with 1 mL of the surrogate fortification solution (see 6.2.1). Note: All samples including blanks are spiked with surrogates. MS and MSD aliquots are additionally fortified with 1 mL of the matrix spiking solution (see 6.2.2).

9.4 Add additional methylene chloride solvent until a total of 5 mL are in the vial. Shake the vial vigorously for one minute.

10. Phenolic Acid Cleanup

10.1 Add 50 mL of blank water to a 125-mL separatory funnel. Add 10 drops of 10N NaOH. Stopper the funnel and mix before checking the pH. Use wide range pH paper to insure adjustment to pH 11-12.

10.2 Transfer the methylene chloride extract from step 9.4 to the separatory funnel. Complete the transfer by adding 7 fresh mL of methylene chloride to the screw-cap vial. Shake vigorously for approximately 10 seconds. Transfer this solvent to the separatory funnel. Note: Approximately 1 mL of solvent is not recoverable from the drying salt and soil for each transfer. Ideally 10 mL of total solvent is transferred to the separatory funnel.

10.3 Shake the contents of the separatory funnel vigorously for 1 minute. Caution: Before vigor is used, invert and vent the excess pressure a sufficient number of times.

10.4 Allow the two liquid phases to separate. Emulsions may make it necessary to use centrifugation to separate the phases. Drain the lower phase (the base/neutral fraction) and retain for further cleanup and PAH analysis.

10.5 Add approximately 5 mL of fresh methylene chloride to the separatory funnel. Gently invert the funnel allowing the organic phase to rinse the walls of the vessel. The organic wash and any solid phase appearing at the interface should be discarded to waste through the stopcock.

10.6 Add approximately 2 mL of Concentrated Hydrochloric Acid to adjust the water in the separatory funnel to pH 2 or lower. Check the pH adjustment with wide range pH paper.

10.7 Add approximately 10 grams of NaCl to the separatory funnel and dissolve by shaking.

10.8 Add 50 mL of fresh methylene chloride to the funnel and equilibrate as before (see 10.3 above). Drain the lower phase (the acid fraction) into a Kadurna-Danish (K-D) evaporative flask equipped with a 10-mL graduated concentrator tube.

10.9 Repeat step 10.8.

10.10 Add a couple of boiling chips to the K-D flask before attaching a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K-D apparatus on a hot water bath (60 °C or warmer)

so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in approximately 5 minutes. When the apparent volume of liquid reaches 1 ml, promptly remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 2 to 5 ml of methylene chloride. A 5-ml syringe is recommended for this operation.

10.11 Add a fresh boiling chip and attach a two-ball micro Snyder column to the concentrator tube. Prewet the Snyder column by adding about 0.5 ml of methylene chloride to the top of the column. Place the K-D on a hot water bath (60°C or warmer) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in approximately 5 minutes. When the volume reaches <0.5 ml, quickly remove the K-D apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Adjust the final volume to 0.5 ml.

10.12 Use a disposable pipet to transfer the contents of the concentrator tube into a 2-ml screw-cap autosampler vial. Complete the transfer by rinsing the joint of the Snyder column and the inner walls of the concentrator tube with <0.5 ml of methylene chloride. Add this rinse to the autosampler vial and cap. Label the vial. The final extract volume is adjusted to 1 ml.

11. PAH Cleanup

11.1 Retrieve the base/neutral fraction from step 10.4, add a couple of fresh boiling chips, and attach a clean 2-ball micro Snyder column. Concentrate the extract as described in step 10.11.

11.2 While the K-D apparatus cools, adjust the temperature of the water bath to 90°C or warmer. Add a fresh boiling chip and approximately 5 ml of n-hexane to the concentrator tube. Re-attach the micro Snyder column and carefully restart the evaporation. Note: For previous evaporations the temperature of the Snyder column has been cool to the touch. As this evaporation proceeds, a noticeable increase in temperature of the Snyder column indicates successful solvent exchange. Promptly remove the K-D apparatus when the apparent volume of hexane is 0.5 ml. Allow the K-D to cool and the Snyder column to drain at least 10 minutes before claiming the base/neutral extract.

11.3 Weigh 2.5 gram (+/- 0.1 gram) of dry activated silica gel into a 50-ml beaker. Create a slurry by covering the silica gel with approximately 10 ml of methylene chloride. Attach a

stopcock to a disposable 6-mL size filtration column. Pour the slurry into the filtration column with the stopcock open. Complete the transfer of the silica gel by swirling small aliquots of methylene chloride in the beaker and pouring into the column. Tap the side of the column several times with a pencil to settle the bed. Cap the silica gel with approximately 1 cm of anhydrous sodium sulfate. (Caution: Do not allow the silica gel medium to become dry.)

11.4 Wash the silica gel bed with an additional 5 mL of methylene chloride. Condition the bed by washing with 5 mL of hexane. Use the stopcock to control the solvent reservoir above the bed.

11.5 Position a 10-mL graduated cylinder to collect the silica gel column effluent. Transfer the base/neutral hexane extract from 11.2 onto the silica gel bed. Open the stopcock and allow the sample to flow into the bed. Close the stopcock. Complete the transfer of the sample by rinsing the joint of the micro Snyder column and the walls of the concentrator tube with two successive 0.5 mL aliquots of hexane. Transfer both of these hexane rinses to the silica gel column. Open the stopcock and allow the transfer rinses to flow into the bed. Continue adding hexane to the bed until 7 mL of the effluent are collected. Close the stopcock and discard the effluent in the graduated cylinder.

11.6 Prepare new mobile phase by mixing equal volumes of methylene chloride and hexane. Label the container 50% methylene chloride in hexane.

11.7 Position a clean 10-mL graduated concentrator tube to collect the PAH fraction as it is eluted from the silica gel bed. Add 50% methylene chloride in hexane to the bed reservoir and open the stopcock. Continue adding the mixed solvent to the bed until 10 mL have been collected. Turnoff the stopcock and discard the disposable column. Note: The stopcock may be cleaned and reused.

11.8 Add a boiling chip to the concentrator tube, attach a clean 2-ball Snyder column, and move the PAH fraction to the hot water bath for solvent exchange and concentration.

11.9 Adjust the hot water bath to 60 °C or warmer. Prime the Snyder column with approximately 0.5 mL of methylene chloride. Begin the concentration as described in 10.11, but remove the K-D from the heat momentarily at an apparent solvent volume of 2 mL. Pour approximately 5 mL of methylene chloride through the Snyder column, and then resume concentration at the hot water bath. Caution: The binary azeotrope formed between methylene chloride and hexane boils at a very low temperature. This final stage of concentration will proceed rapidly. Hold the Snyder column with the finger tips (do not support the apparatus mechanically). This

procedure will permit monitoring the temperature of the Snyder surface by touch. At this stage of concentration a proper solvent exchange into methylene chloride will not produce a hot Snyder surface. If the Snyder temperature becomes hot to the touch, then additional methylene chloride must be added to the vessel to insure a proper solvent exchange.

11.10 The concentration is complete when the volume of solvent is <0.5 ml. Remove the K-D from the hot water bath and allow the Snyder to drain and cool for at least 10 minutes. Adjust the final volume to 0.5 ml.

11.11 Transfer the PAH fraction into a screw-cap autosampler vial. Complete the transfer by rinsing the joint of the Snyder and the inner walls of the concentrator tube with <0.5 ml of methylene chloride. Add this rinse to the autosampler vial and cap. The final extract volume is then adjusted to 1 ml. Label the vial.

12. Gas Chromatography

12.1 The gas chromatographic (GC) conditions listed in Figure 1 are recommended. Some GC systems may be able to achieve worthy improvement in compound separations by using multiple temperature ramps for the column oven. Caution must be exercised, however, not to improve resolution at the expense of column capacity or some other desirable chromatographic feature.

12.2 Continuing calibration checks require a daily injection of the midpoint calibration standard (see 7.6). If all CCC requirements are met, then updates may be made for both RRT's and RRF's to allow for small fluctuations in instrument response as well as shifts in retention time due to capillary column maintenance.
Note: It is a common practice to routinely clean injection port liners and cut small portions from the inlet end of a capillary column. This practice helps reduce chromatographic active sites.

12.3 Qualitative Analysis

12.3.1 Peak identification is based upon relative retention time comparison to calibration data. Library retention times are established on a daily basis. Internal standards serve as time references for all chromatographic acquisitions. A target compound should be identified in a sample only if the chromatographic peak matches the predicted retention time within 0.05 minutes.

$$\text{Predicted RT} = (RRT^{library})(RT_{18}{}^{sample}) \qquad \text{Eq.5}$$

12.3.2 Equation 5 should serve only as a recommended treatment for peak identification. The experienced analyst must consider other chromatographic features such as peak shape, resolution, and distance from the time reference.

12.4 Quantitative Analysis.

12.4.1 Sample extracts should be prepared for analysis by placing 100 uL of internal standard solution (see 6.1.4) into the autosampler vial and mixing. Internal standard levels are high to minimize the effects of compound coelution.

12.4.2 The concentration of an identified target compound (e.g. compound X) may be calculated using equation 6.

$$PPM_x^{sample} = \frac{(ug_{IS}^{sample})(Area_x^{sample})(RRF^x)}{(Area_{IS}^{sample})(\text{grams extracted})} \qquad \text{Eq.6}$$

It should be noted that the concentration calculated is based upon the wet weight of the soil sample.

TABLE 1

LISTING OF INTERNAL STANDARD COMPOUNDS

Internal Standard	Compound
Internal Standard #1	1,4-Dichlorobenzene-d4
Internal Standard #2	2-Fluorobiphenyl
Internal Standard #3	n-Hexadecane
Internal Standard #4	2,4,6-Tribromobiphenyl
Internal Standard #5	Perylene-d12

TABLE 2

PEAK LISTING FOR FIGURE 1 CALIBRATION STANDARD

Compound	RT (MIN.)	RRT	Peak Area	Amt.*	RRF
Phenol	4.594	0.918	430241	100	0.769
2-Chlorophenol	4.690	0.937	334306	100	0.990
2,3-Benzofuran (surr.)	4.807	0.960	233053	50	0.710
I.S. #1	5.005	1.000	330858	100	1.000
2-Nitrophenol	6.864	0.666	244794	100	2.191
2,4-Dimethylphenol	7.133	0.692	416758	100	1.287
2,4-Dichlorophenol	7.403	0.718	238624	100	2.248
Naphthalene	7.665	0.774	310573	50	0.864
4-Chloro-3-methylphenol	9.198	0.893	305774	100	1.754
2,4,6-Trichlorophenol	10.087	0.979	184451	100	2.908
I.S. #2	10.304	1.000	536391	100	1.000
Acenaphthylene	11.395	0.856	272447	50	0.940
Acenaphthene	11.862	0.891	291254	50	0.879
2,4-Dinitrophenol	12.063	0.906	100976	100	5.071
4-Nitrophenol	12.415	0.932	165345	100	3.097
Fluorene	13.098	0.983	263934	50	0.970
I.S. #3	13.319	1.000	512076	100	1.000
4,6-Dinitro-2-methylphenol	13.434	1.009	143587	100	3.566
2,4,6-Tribromophenol (surr.)	13.727	1.031	46517	100	11.008
Pentachlorophenol	15.121	0.832	81907	100	2.191
Phenanthrene	15.392	0.847	243175	50	0.369
Anthracene	15.503	0.853	198134	50	0.453
I.S. #4	18.171	1.000	179468	100	1.000
Fluoranthene	18.322	1.008	210783	50	0.426
Pyrene	18.837	1.037	207789	50	0.432
Terphenyl-d14 (surr.)	19.484	1.072	200730	50	0.447
Benzo(a)anthracene	21.867	0.867	177233	50	0.960
Chrysene	21.967	0.871	194432	50	0.875
Benzo(b)fluoranthene	24.408	0.968	177141	50	0.960
Benzo(k)fluoranthene	24.466	0.970	174051	50	0.977
Benzo(a)pyrene	25.077	0.994	157454	50	1.080
I.S. #5	25.221	1.000	340257	100	1.000
Indeno(1,2,3-cd)pyrene	27.387	1.086	152731	50	1.114
Dibenzo(a,h)anthracene	27.475	1.089	146545	50	1.161
Benzo(g,h,i)perylene	27.936	1.108	156756	50	1.085

* This numerical value does not necessarily refer to concentration nor to amount injected, but it always refers to the mass of compound dissolved in an undefined volume of solvent.

TABLE 3

LISTING OF CALIBRATION CHECK COMPOUNDS

Phenolic Acids	PAHs
Phenol	Fluorene
2,4,6-Trichlorophenol	Fluoranthene
2,4-Dinitrophenol	Benzo(a)pyrene

1. Phenol
2. 2-Chlorophenol
3. 2,3-Benzofuran
4. I.S. #1
5. 2-Nitrophenol
6. 2,4-Dimethylphenol
7. 2,4-Dichlorophenol
8. Naphthalene
9. 4-Chloro-3-methylphenol
10. 2,4,6-Trichlorophenol
11. I.S. #2
12. Acenaphthylene
13. Acenaphthene
14. 2,4-Dinitrophenol
15. 4-Nitrophenol
16. Fluorene
17. I.S. #3
18. 4,6-Dinitro-2-methylphenol
19. 2,4,6-Tribromophenol
20. Pentachlorophenol
21. Phenanthrene
22. Anthracene
23. I.S. #4
24. Fluoranthene
25. Pyrene
26. Terphenyl-d14
27. Benzo(a)anthracene
28. Chrysene
29. Benzo(b)fluoranthene
30. Benzo(k)fluoranthene
31. Benzo(a)pyrene
32. I.S. #5
33. Indeno(1,2,3-cd)pyrene
34. Dibenzo(a,h)anthracene
35. Benzo(g,h,i)perylene

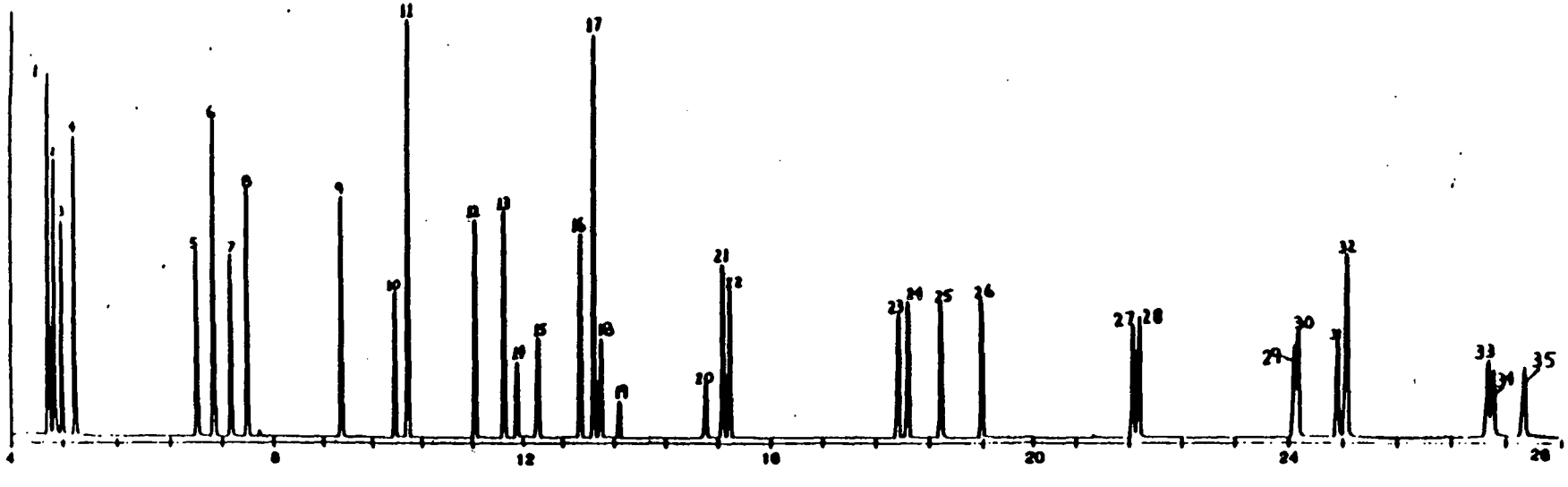

Figure 1. GC/FID chromatogram of the instrument calibration standard. DB-5 fused silica capillary column, 21m x 0.32mm ID, 0.25um film. Column Zone: 45 °C for 1 minute, then 10 °C/minute to 300 °C and hold. Splitless injection. Split opened at 0.95 minutes. Injector zone: 250 °C. Detector zone: 320 °C. Helium carrier inlet pressure set at 12 psig. Electrometer range set at 10^{-11} amps/mV. Sample: 1.5 uL of methylene chloride containing 150 ng of each internal standard and phenolic acid and containing 75 ng of each PAH.

CASE NARRATIVE

INTRODUCTION

In April 1987, the CH2M HILL Montgomery laboratory presented a new soil screening method for selected polyaromatic hydrocarbons (PAHs) and phenolic acid compounds. This document provides a brief narrative of the method development and testing.

SUMMARY

Priority pollutant PAHs and phenolic acids were the target compounds. The analytical approach was capillary gas chromatography with flame ionization detection (GC/FID). The method was tested with five carefully selected soil samples from the Moss America site. The five soils offered varying amoumts of contamination. For comparison, the soils were analyzed by the new method and by validated methodology (US EPA, Method 8270 with 3550 extraction)[1]. Method 3550 is extraction by serial sonifications. Method 8270 is analysis of sample extracts by capillary gas chromatography with mass spectrometry (GC/MS).

METHOD DEVELOPMENT

The method development may be divided into four subtasks.

1. Evaluate capillary columns for best resolution of target parameters.
2. Select internal standards and surrogates.
3. Evaluate linearity of the instrument.
4. Develope a fast cleanup procedure for the target compounds.

Each subtask was performed using target compounds dissolved in solvent.

Subtask 1. Three capillary columns were investigated. All three are available from J & W Scientific and were 0.32 mm inside diameter with a 0.25 micrometer filmthickness: a DB-5, a DB-17, and a DB-1701. A twenty-meter length of the DB-5 column offered sufficient resolution of the compounds of interest with a fast analysis time. The DB-1701 and the DB-17 were more polar columns and did not offer superior performance.

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[1] Office of Solid Waste and Emergency Response, SW-846, Test Methods for Evaluating Solid Waste, Third Edition, November 1986.

Subtask 2. Five internal standards were selected to monitor the quality of each injection. The compounds of interest cover such a large boiling range that multiple internal standards were required. For the alert analyst, multiple internal standards also reduce the errors associated with compound coelution. If the primary internal standard appears to suffer from coelution, then an alternate internal standard may be used for the calculation. Three surrogates were selected to monitor the quality of each sample preparation and analysis. Two surrogates were chosen for the PAH fraction since this fraction is more likely to suffer interferences than the acid fraction.

Subtask 3. Linearity of the GC/FID system was evaluated by injecting six levels of calibration standards (see table 1). Relative response factors (RRFs) were calculated for each compound at each level injected. The PAHs were injected at 100, 75, 50, 25, 10, and 5 ug/mL for RRF #1, ...RRF #6 respectively. Similar repartitions were injected for the phenolic acids except that concentrations were doubled. Notice that two compounds (2,4-Dinitrophenol and 2-methyl-4,6-dinitrophenol) failed the initial calibration criteria prescribed by the method. However, by droping the lowest level calibration point, these two compounds will have acceptable values for %RSD (see table 2). Typical 3-level calibration is illustrated in table 3.

Subtask 4. To design the cleanup procedures, two additional compounds were added to the calibration mixture: n-Dodecane and n-Tridecane. Acid cleanup procedures were available in the literature (US EPA, Method 3650)[1]. No significant modifications were prescribed for this acid cleanup. Several commercially available extraction columns were tested for use in the PAH cleanup procedure. The largest column size available was only a 1-gram bed. Silica Gel, Alumina, and Phenyl columns were tested. Both capacity and activity were determined to be low for the needs of this cleanup design. A 2.5-gram bed of silica gel packed as a slurry into into a disposable filtration column at the laboratory fulfilled the needs of the PAH cleanup design.

METHOD TESTING

Eighteen soil samples from the Moss America site were submitted for gross screening, and five of these samples were used to test the new method. Sam 1 was selected for matrix spiking because it was the least contaminated of the soils.

The five samples were analyzed by both GC/FID and GC/MS. For additional data, the soils were also analyzed by GC/FID without applying sample cleanup procedures. Only two samples were anlayzed by GC/MS without applying cleanup procedures. Without cleanup, most sample extracts damaged the chromatographic systems.

TABLE 1

GC/FID INITIAL CALIBRATION APRIL 8, 1987

COMPOUND	MEAN RRF	%RSD	RRF #1	RRF #2	RRF #3	RRF #4	RRF #5	RRF #6
PHENOL	0.8066	7.9	0.7606	0.7610	0.7690	0.7838	0.8451	0.9203
2-CHLOROPHENOL	1.0292	6.6	0.9788	0.9817	0.9897	1.0048	1.0712	1.1487
2,3-BENZOFURAN SURR.	0.7388	5.0	0.7115	0.7142	0.7098	0.7326	0.7623	0.8022
I.S. #1	1.0000	0.0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2-NITROPHENOL	2.3788	14.4	2.1105	2.1191	2.1912	2.2902	2.5753	2.9867
2,4-DIMETHYLPHENOL	1.3421	8.0	1.2860	1.2721	1.2871	1.2824	1.3798	1.5451
2,4-DICHLOROPHENOL	2.3597	8.8	2.2224	2.2080	2.2479	2.2828	2.4584	2.7389
NAPHTHALENE	0.8737	2.8	0.8626	0.8575	0.8636	0.8533	0.8879	0.9171
4-CL-3-ME PHENOL	1.8741	12.1	1.6904	1.7129	1.7542	1.8260	1.9760	2.2851
2,4,6-TRICL PHENOL	3.1244	11.7	2.8784	2.8432	2.9080	3.0297	3.2935	3.7938
I.S. #2	1.0000	0.0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
ACENAPHTHYLENE	0.9765	6.3	0.9472	0.9293	0.9398	0.9530	0.9959	1.0940
ACENAPHTHENE	0.9097	5.4	0.8914	0.8748	0.8791	0.8860	0.9225	1.0043
2,4-DINITROPHENOL	6.3542	27.2	5.1632	5.2463	5.0713	6.4458	6.5936	9.6050
4-NITROPHENOL	3.5270	15.6	3.0999	3.2703	3.0970	3.6843	3.4563	4.5542
FLUORENE	1.0373	10.2	0.9643	0.9662	0.9701	1.0231	1.0607	1.2394
I.S. #3	1.0000	0.0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2-ME-4,6-DINO PHENOL	4.3256	24.8	3.5225	3.6663	3.5663	4.4320	4.4174	6.3490
TBP-SURR.	12.6944	18.7	10.4318	11.1604	11.0084	13.2603	13.5139	16.7915
PENTACHLOROPHENOL	2.4137	10.9	2.6330	2.2545	2.1911	2.1712	2.4171	2.8151
PHENANTHRENE	0.3786	4.5	0.3937	0.3707	0.3690	0.3542	0.3843	0.3999
ANTHRACENE	0.4584	5.1	0.4692	0.4356	0.4529	0.4334	0.4632	0.4960
I.S. #4	1.0000	0.0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
FLUORANTHENE	0.4472	6.6	0.4639	0.4429	0.4257	0.4285	0.4235	0.4988
PYRENE	0.4615	6.7	0.4719	0.4513	0.4319	0.4461	0.4495	0.5185
TERPHENYL-d14 SURR.	0.4944	9.9	0.4778	0.4790	0.4470	0.4733	0.5020	0.5875
BENZO(A)ANTHRACENE	0.9627	3.4	0.9289	0.9267	0.9600	0.9641	1.0102	0.9866
CHRYSENE	0.8882	3.0	0.8708	0.8591	0.8750	0.8820	0.9180	0.9243
BENZO(B)FLUORANTHENE	1.0004	7.8	0.9441	0.9373	0.9604	0.9676	1.0620	1.1307
BENZO(K)FLUORANTHENE	1.0026	4.0	0.9785	0.9783	0.9775	0.9975	1.0035	1.0806
BENZO(A)PYRENE	1.1437	10.9	1.0682	1.0684	1.0805	1.0972	1.1593	1.3886
I.S. #5	1.0000	0.0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
INDENO(123-CD)PYRENE	1.1857	11.2	1.0482	1.1090	1.1139	1.1736	1.2520	1.4174
DIBENZO(AH)ANTHRA.	1.2575	11.2	1.2241	1.1535	1.1609	1.1902	1.2888	1.5278
BENZO(GHI)PERYLENE	1.1513	8.4	1.0859	1.0898	1.0853	1.1340	1.1794	1.3311

TABLE 2

GC/FID INITIAL CALIBRATION APRIL 8, 1987

COMPOUND	MEAN RRF	%RSD	RRF #1	RRF #2	RRF #3	RRF #4	RRF #5	RRF #6
PHENOL	0.8066	7.9	0.7606	0.7610	0.7690	0.7838	0.8451	0.9203
2-CHLOROPHENOL	1.0292	6.6	0.9788	0.9817	0.9897	1.0048	1.0712	1.1487
2,3-BENZOFURAN SURR.	0.7388	5.0	0.7115	0.7142	0.7098	0.7326	0.7623	0.8022
I.S. #1	1.0000	0.0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2-NITROPHENOL	2.3788	14.4	2.1105	2.1191	2.1912	2.2902	2.5753	2.9867
2,4-DIMETHYLPHENOL	1.3421	8.0	1.2860	1.2721	1.2871	1.2824	1.3798	1.5451
2,4-DICHLOROPHENOL	2.3597	8.8	2.2224	2.2080	2.2479	2.2828	2.4584	2.7389
NAPHTHALENE	0.8737	2.8	0.8626	0.8575	0.8636	0.8533	0.8879	0.9171
4-CL-3-ME PHENOL	1.8741	12.1	1.6904	1.7129	1.7542	1.8260	1.9760	2.2851
2,4,6-TRICL PHENOL	3.1244	11.7	2.8784	2.8432	2.9080	3.0297	3.2935	3.7938
I.S. #2	1.0000	0.0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
ACENAPHTHYLENE	0.9765	6.3	0.9472	0.9293	0.9398	0.9530	0.9959	1.0940
ACENAPHTHENE	0.9097	5.4	0.8914	0.8748	0.8791	0.8860	0.9225	1.0043
2,4-DINITROPHENOL	5.7040	13.1	5.1632	5.2463	5.0713	6.4458	6.5936	
4-NITROPHENOL	3.5270	15.6	3.0999	3.2703	3.0970	3.6843	3.4563	4.5542
FLUORENE	1.0373	10.2	0.9643	0.9662	0.9701	1.0231	1.0607	1.2394
I.S. #3	1.0000	0.0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2-ME-4,6-DINO PHENOL	3.9209	11.8	3.5225	3.6663	3.5663	4.4320	4.4174	
TBP-SURR.	12.6944	18.7	10.4318	11.1604	11.0084	13.2603	13.5139	16.7915
PENTACHLOROPHENOL	2.4137	10.9	2.6330	2.2545	2.1911	2.1712	2.4171	2.8151
PHENANTHRENE	0.3786	4.5	0.3937	0.3707	0.3690	0.3542	0.3843	0.3999
ANTHRACENE	0.4584	5.1	0.4692	0.4356	0.4529	0.4334	0.4632	0.4960
I.S. #4	1.0000	0.0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
FLUORANTHENE	0.4472	6.6	0.4639	0.4429	0.4257	0.4285	0.4235	0.4988
PYRENE	0.4615	6.7	0.4719	0.4513	0.4319	0.4461	0.4495	0.5185
TERPHENYL-d14 SURR.	0.4944	9.9	0.4778	0.4790	0.4470	0.4733	0.5020	0.5875
BENZO(A)ANTHRACENE	0.9627	3.4	0.9289	0.9267	0.9600	0.9641	1.0102	0.9866
CHRYSENE	0.8882	3.0	0.8708	0.8591	0.8750	0.8820	0.9180	0.9243
BENZO(B)FLUORANTHENE	1.0004	7.8	0.9441	0.9373	0.9604	0.9676	1.0620	1.1307
BENZO(K)FLUORANTHENE	1.0026	4.0	0.9785	0.9783	0.9775	0.9975	1.0035	1.0806
BENZO(A)PYRENE	1.1437	10.9	1.0682	1.0684	1.0805	1.0972	1.1593	1.3886
I.S. #5	1.0000	0.0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
INDENO(123-CD)PYRENE	1.1857	11.2	1.0482	1.1090	1.1139	1.1736	1.2520	1.4174
DIBENZO(AH)ANTHRA.	1.2575	11.2	1.2241	1.1535	1.1609	1.1902	1.2888	1.5278
BENZO(GHI)PERYLENE	1.1513	8.4	1.0859	1.0898	1.0853	1.1340	1.1794	1.3336

TABLE 3

GC/FID INITIAL CALIBRATION APRIL 8, 1987

COMPOUND	MEAN RRF	%RSD	RRF #1	RRF #2	RRF #3	RRF #4	RRF #5	RRF #6
PHENOL	0.7916	5.9	0.7606		0.7690		0.8451	
2-CHLOROPHENOL	1.0132	5.0	0.9788		0.9897		1.0712	
2,3-BENZOFURAN SURR.	0.7279	4.1	0.7115		0.7098		0.7623	
I.S. #1	1.0000	0.0	1.0000		1.0000		1.0000	
2-NITROPHENOL	2.2923	10.8	2.1105		2.1912		2.5753	
2,4-DIMETHYLPHENOL	1.3176	4.1	1.2860		1.2871		1.3798	
2,4-DICHLOROPHENOL	2.3096	5.6	2.2224		2.2479		2.4584	
NAPHTHALENE	0.8713	1.6	0.8626		0.8636		0.8879	
4-CL-3-ME PHENOL	1.8069	8.3	1.6904		1.7542		1.9760	
2,4,6-TRICL PHENOL	3.0266	7.7	2.8784		2.9080		3.2935	
I.S. #2	1.0000	0.0	1.0000		1.0000		1.0000	
ACENAPHTHYLENE	0.9610	3.2	0.9472		0.9398		0.9959	
ACENAPHTHENE	0.8976	2.5	0.8914		0.8791		0.9225	
2,4-DINITROPHENOL	5.6094	15.2	5.1632		5.0713		6.5936	
4-NITROPHENOL	3.2177	6.4	3.0999		3.0970		3.4563	
FLUORENE	0.9984	5.4	0.9643		0.9701		1.0607	
I.S. #3	1.0000	0.0	1.0000		1.0000		1.0000	
2-ME-4,6-DINO PHENOL	3.8354	13.2	3.5225		3.5663		4.4174	
TBP-SURR.	11.6514	14.1	10.4318		11.0084		13.5139	
PENTACHLOROPHENOL	2.4137	9.2	2.6330		2.1911		2.4171	
PHENANTHRENE	0.3823	3.3	0.3937		0.3690		0.3843	
ANTHRACENE	0.4618	1.8	0.4692		0.4529		0.4632	
I.S. #4	1.0000	0.0	1.0000		1.0000		1.0000	
FLUORANTHENE	0.4377	5.2	0.4639		0.4257		0.4235	
PYRENE	0.4511	4.4	0.4719		0.4319		0.4495	
TERPHENYL-d14 SURR.	0.4756	5.8	0.4778		0.4470		0.5020	
BENZO(A)ANTHRACENE	0.9664	4.2	0.9289		0.9600		1.0102	
CHRYSENE	0.8879	2.9	0.8708		0.8750		0.9180	
BENZO(B)FLUORANTHENE	0.9888	6.5	0.9441		0.9604		1.0620	
BENZO(K)FLUORANTHENE	0.9865	1.5	0.9785		0.9775		1.0035	
BENZO(A)PYRENE	1.1026	4.5	1.0682		1.0805		1.1593	
I.S. #5	1.0000	0.0	1.0000		1.0000		1.0000	
INDENO(123-CD)PYRENE	1.1380	9.1	1.0482		1.1139		1.2520	
DIBENZO(AH)ANTHRA.	1.2246	5.2	1.2241		1.1609		1.2888	
BENZO(GHI)PERYLENE	1.1169	4.8	1.0859		1.0853		1.1794	

ANALYTICAL REPORT
PAH/PHENOLS by GC/FID

Client: CH2M HILL/GLO
Sample Identification: Laboratory Method Blank

Lab No: 040878S1
Date Received: N/A

Date Extracted: 04/08/87
Date Analyzed: 04/09/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	< 1	Phenol	< 2
Acenaphthylene	< 1	2-Chlorophenol	< 2
Acenaphthene	0.9	2-Nitrophenol	< 2
Fluorene	< 1	2,4-Dimethylphenol	< 2
Phenanthrene	0.5	2,4-Dichlorophenol	< 2
Anthracene	0.4	4-Chloro-3-methylphenol	< 2
Fluoranthene	0.2	2,4,6-Trichlorophenol	< 2
Pyrene	0.2	2,4-Dinitrophenol	< 5
Benzo(a)anthracene	< 1	4-Nitrophenol	< 5
Chrysene	< 1	4,6-Dinitro-2-methylphenol	< 5
Benzo(b)fluoranthene	< 1	Pentachlorophenol	< 5
Benzo(k)fluoranthene	0.5		
Benzo(a)pyrene	< 1		
Indeno(1,2,3-cd)pyrene	< 1		
Dibenzo(a,h)anthracene	< 1		
Benzo(g,h,i)perylene	< 1		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 64 %	2,4,6-Tribromophenol = 52 %
Terphenyl-d14 = 82 %	

Comments:

Reviewed by: J. Smiley

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/FID

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 1, Grab, 2/1/87, 1030

Lab No: Sam 1
Date Received: 02/10/87

Date Extracted: 04/08/87
Date Analyzed: 04/09/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	< 1	Phenol	< 2
Acenaphthylene	< 1	2-Chlorophenol	< 2
Acenaphthene	0.8	2-Nitrophenol	< 2
Fluorene	< 1	2,4-Dimethylphenol	< 2
Phenanthrene	0.3	2,4-Dichlorophenol	< 2
Anthracene	0.3	4-Chloro-3-methylphenol	< 2
Fluoranthene	< 1	2,4,6-Trichlorophenol	< 2
Pyrene	< 1	2,4-Dinitrophenol	< 5
Benzo(a)anthracene	< 1	4-Nitrophenol	< 5
Chrysene	< 1	4,6-Dinitro-2-methylphenol	< 5
Benzo(b)fluoranthene	< 1	Pentachlorophenol	< 5
Benzo(k)fluoranthene	0.4		
Benzo(a)pyrene	< 1		
Indeno(1,2,3-cd)pyrene	< 1		
Dibenzo(a,h)anthracene	< 1		
Benzo(g,h,i)perylene	< 1		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 68 %	2,4,6-Tribromophenol = 47 %
Terphenyl-d14 = 87 %	

Comments:

Reviewed by: J. Smiley

M A T R I X S P I K E R E S U L T S

Laboratory No.: S1PAH-S.WK1 Fraction: PAH by GC/FID Matrix: Soil

Sample Description: Matrix spike and matrix spike duplicate into soil sample labeled Moss American, Sample 1, Grab, 2/1/87, 1030.

Compound	Concentration Spiked (PPB)	Sample Result (PPB)	Spike Result (PPB)	Spike Percent Recovery	Duplicate Spike Result (PPB)	Duplicate Percent Recovery	RPD
NAPHTHALENE	30	0	21	70	22	73	5
ACENAPHTHYLENE	30	0	23	77	23	77	0
ACENAPHTHENE	30	0.8	23	74	25	81	8
FLUORENE	30	0	23	77	22	73	4
PHENANTHRENE	30	0.3	24	79	23	76	4
ANTHRACENE	30	0.3	27	89	25	82	8
FLUORANTHENE	30	0	23	77	22	73	4
PYRENE	30	0	23	77	23	77	0
BENZO(A)ANTHRACENE	30	0	24	80	24	80	0
CHRYSENE	30	0	24	80	23	77	4
BENZO(B)FLUORANTHENE	30	0	23	77	23	77	0
BENZO(K)FLUORANTHENE	30	0.4	23	75	22	72	4
BENZO(A)PYRENE	30	0	23	77	23	77	0
INDENO(123-CD)PYRENE	30	0	23	77	23	77	0
DIBENZO(AH)ANTHRA.	30	0	22	73	20	67	10
BENZO(GHI)PERYLENE	30	0	22	73	21	70	5
2,3-BENZOFURAN *	30	---	19	63	20	67	5
TERPHENYL-d14 *	30	---	22	73	22	73	0

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* Surrogate

$$\text{Percent Recovery} = \frac{\text{Spike Result} - \text{Sample Result}}{\text{Concentration Spiked}} \times 100$$

$$\text{RPD} = \frac{\text{Spike Result} - \text{Duplicate Spike Result}}{\text{Spike Result} + \text{Duplicate Spike Result}} \times 200$$

Comments:

CH2M HILL
ENVIRONMENTAL LI

M A T R I X S P I K E R E S U L T S

Laboratory No.: S1AC-S.WK1 Fraction: Phenolic Acids by GC/FID Matrix: Soil

Sample Description: Matrix spike and matrix spike duplicate into soil sample labeled Moss American, Sample 1, Grab, 2/1/87, 1030.

Compound	Concentration Spiked (PPB)	Sample Result (PPB)	Spike Result (PPB)	Spike Percent Recovery	Duplicate Spike Result (PPB)	Duplicate Percent Recovery	RPD
PHENOL	60	0	48	80	49	82	2
2-CHLOROPHENOL	60	0	49	82	48	80	2
2-NITROPHENOL	60	0	52	87	49	82	6
2,4-DIMETHYLPHENOL	60	0	47	78	44	73	7
2,4-DICHLOROPHENOL	60	0	51	85	49	82	4
4-CL-3-ME PHENOL	60	0	51	85	50	83	2
2,4,6-TRICL PHENOL	60	0	51	85	50	83	2
2,4-DINITROPHENOL	60	0	27	45	33	55	20
4-NITROPHENOL	60	0	29	48	42	70	37
2-ME-4,6-DINO PHENOL	60	0	38	63	45	75	17
PENTACHLOROPHENOL	60	0	56	93	55	92	2
2,4,6-TRIBROMOPHENOL *	60	---	43	72	45	75	5

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* Surrogate

$$\text{Percent Recovery} = \frac{\text{Spike Result} - \text{Sample Result}}{\text{Concentration Spiked}} \times 100$$

$$\text{RPD} = \frac{\text{Spike Result} - \text{Duplicate Spike Result}}{\text{Spike Result} + \text{Duplicate Spike Result}} \times 200$$

Comments:

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/FID

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 5, Grab, 2/1/87, 1130

Lab No: Sam 5
Date Received: 02/10/87

Date Extracted: 04/08/87
Date Analyzed: 04/09/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	0.2	Phenol	< 2
Acenaphthylene	0.2	2-Chlorophenol	< 2
Acenaphthene	1.9	2-Nitrophenol	< 2
Fluorene	< 1	2,4-Dimethylphenol	< 2
Phenanthrene	1.1	2,4-Dichlorophenol	< 2
Anthracene	0.5	4-Chloro-3-methylphenol	< 2
Fluoranthene	1.8	2,4,6-Trichlorophenol	< 2
Pyrene	1.7	2,4-Dinitrophenol	< 5
Benzo(a)anthracene	2.0	4-Nitrophenol	< 5
Chrysene	1.6	4,6-Dinitro-2-methylphenol	< 5
Benzo(b)fluoranthene	2.3	Pentachlorophenol	< 5
Benzo(k)fluoranthene	1.3		
Benzo(a)pyrene	2.1		
Indeno(1,2,3-cd)pyrene	1.7		
Dibenzo(a,h)anthracene	0.6		
Benzo(g,h,i)perylene	1.9		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 68 %	2,4,6-Tribromophenol = 36 %
Terphenyl-d14 = 70 %	

Comments: PAH and Phenol fractions were analyzed separately.

Reviewed by: J. Smiley

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/FID

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 15, Grab, 2/1/87, 1400

Lab No: Sam 15
Date Received: 02/10/87
Date Extracted: 04/08/87
Date Analyzed: 04/09/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	0.4	Phenol	< 2
Acenaphthylene	< 1	2-Chlorophenol	< 2
Acenaphthene	1.5	2-Nitrophenol	< 2
Fluorene	0.2	2,4-Dimethylphenol	< 2
Phenanthrene	2.9	2,4-Dichlorophenol	< 2
Anthracene	1.2	4-Chloro-3-methylphenol	< 2
Fluoranthene	3.9	2,4,6-Trichlorophenol	< 2
Pyrene	3.2	2,4-Dinitrophenol	< 5
Benzo(a)anthracene	3.6	4-Nitrophenol	< 5
Chrysene	1.9	4,6-Dinitro-2-methylphenol	< 5
Benzo(b)fluoranthene	6.0	Pentachlorophenol	< 5
Benzo(k)fluoranthene	2.4		
Benzo(a)pyrene	6.2		
Indeno(1,2,3-cd)pyrene	3.6		
Dibenzo(a,h)anthracene	2.1		
Benzo(g,h,i)perylene	4.1		

SURROGATE RECOVERIES		SURROGATE RECOVERY	
2,3-Benzofuran	63 %	2,4,6-Tribromophenol	41 %
Terphenyl-d14	70 %		

Comments: PAH and Phenol fractions were analyzed separately.

Reviewed by: J. Smiley

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/FID

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 17, Grab, 2/1/87, 1430

Lab No: Sam 17
Date Received: 02/10/87

Date Extracted: 04/08/87
Date Analyzed: 04/09/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	1.0	Phenol	< 2
Acenaphthylene	< 1	2-Chlorophenol	< 2
Acenaphthene	0.8	2-Nitrophenol	< 2
Fluorene	0.2	2,4-Dimethylphenol	< [illegible]
Phenanthrene	4.5	2,4-Dichlorophenol	< [illegible]
Anthracene	1.3	4-Chloro-3-methylphenol	< 2
Fluoranthene	10	2,4,6-Trichlorophenol	< 2
Pyrene	22	2,4-Dinitrophenol	< 5
Benzo(a)anthracene	8.0	4-Nitrophenol	< 5
Chrysene	7.4	4,6-Dinitro-2-methylphenol	< 5
Benzo(b)fluoranthene	16	Pentachlorophenol	< 5
Benzo(k)fluoranthene	8.0		
Benzo(a)pyrene	9.6		
Indeno(1,2,3-cd)pyrene	6.7		
Dibenzo(a,h)anthracene	3.4		
Benzo(g,h,i)perylene	9.3		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 70 %	2,4,6-Tribromophenol = 53 %
Terphenyl-d14 = 72 %	

Comments: PAH and Phenol fractions were analyzed separately.

Reviewed by: J. Smiley

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/FID

Client: CH2M HILL/GLO
Sample Identification: Laboratory Method Blank

Lab No: 04087BS2
Date Received: N/A

Date Extracted: 04/08/87
Date Analyzed: 04/09/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	< 1	Phenol	< 2
Acenaphthylene	< 1	2-Chlorophenol	< 2
Acenaphthene	0.9	2-Nitrophenol	< 2
Fluorene	< 1	2,4-Dimethylphenol	< 2
Phenanthrene	0.5	2,4-Dichlorophenol	< 2
Anthracene	0.4	4-Chloro-3-methylphenol	< 2
Fluoranthene	0.2	2,4,6-Trichlorophenol	< 2
Pyrene	0.2	2,4-Dinitrophenol	< 5
Benzo(a)anthracene	< 1	4-Nitrophenol	< 5
Chrysene	< 1	4,6-Dinitro-2-methylphenol	< 5
Benzo(b)fluoranthene	< 1	Pentachlorophenol	< 5
Benzo(k)fluoranthene	0.5		
Benzo(a)pyrene	< 1		
Indeno(1,2,3-cd)pyrene	< 1		
Dibenzo(a,h)anthracene	< 1		
Benzo(g,h,i)perylene	< 1		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 59 %	2,4,6-Tribromophenol = 60 %
Terphenyl-d14 = 70 %	

Comments: No cleanup procedures were applied to this sample.

Reviewed by: J. Smiley

ANALYTICAL REPORT
PAH/PHENOLS by GC/FID

Client: CH2M HILL/GLO
Sample Identification: Laboratory Method Blank

Lab No: 02187BS1
Date Received: N/A

Date Extracted: 02/18/87
Date Analyzed: 03/07/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	< 1	Phenol	< 2
Acenaphthylene	< 1	2-Chlorophenol	< 2
Acenaphthene	1.6	2-Nitrophenol	< 2
Fluorene	< 1	2,4-Dimethylphenol	< 2
Phenanthrene	< 1	2,4-Dichlorophenol	< 2
Anthracene	< 1	4-Chloro-3-methylphenol	< 2
Fluoranthene	< 1	2,4,6-Trichlorophenol	< 2
Pyrene	< 1	2,4-Dinitrophenol	< 5
Benzo(a)anthracene	< 1	4-Nitrophenol	< 5
Chrysene	< 1	4,6-Dinitro-2-methylphenol	< 5
Benzo(b)fluoranthene	< 1	Pentachlorophenol	< 5
Benzo(k)fluoranthene	< 1		
Benzo(a)pyrene	< 1		
Indeno(1,2,3-cd)pyrene	< 1		
Dibenzo(a,h)anthracene	< 1		
Benzo(g,h,i)perylene	< 1		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 76 %	2,4,6-Tribromophenol = 69 %
Terphenyl-d14 = 53 %	

Comments: No cleanup procedures were applied to this sample.

Reviewed by: J. Smiley

ANALYTICAL REPORT
PAH/PHENOLS by GC/FID

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 1, Grab, 2/1/87, 1030

Lab No: Sam 1
Date Received: 02/10/87

Date Extracted: 04/08/87
Date Analyzed: 04/09/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	< 1	Phenol	< 2
Acenaphthylene	< 1	2-Chlorophenol	< 2
Acenaphthene	0.6	2-Nitrophenol	< 2
Fluorene	< 1	2,4-Dimethylphenol	< 2
Phenanthrene	0.6	2,4-Dichlorophenol	< 2
Anthracene	< 1	4-Chloro-3-methylphenol	< 2
Fluoranthene	< 1	2,4,6-Trichlorophenol	< 2
Pyrene	< 1	2,4-Dinitrophenol	2
Benzo(a)anthracene	< 1	4-Nitrophenol	< 5
Chrysene	< 1	4,6-Dinitro-2-methylphenol	< 5
Benzo(b)fluoranthene	< 1	Pentachlorophenol	< 5
Benzo(k)fluoranthene	< 1		
Benzo(a)pyrene	< 1		
Indeno(1,2,3-cd)pyrene	< 1		
Dibenzo(a,h)anthracene	< 1		
Benzo(g,h,i)perylene	< 1		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 68 %	2,4,6-Tribromophenol = 50 %
Terphenyl-d14 = 60 %	

Comments: No cleanup procedures were applied to this sample.

Reviewed by: J. Smiley

M A T R I X S P I K E R E S U L T S

Laboratory No.: SIP-WO-S.WK1 Fraction: PAH by GC/FID Matrix: Soil

Sample Description: Matrix spike and matrix spike duplicate into soil sample labeled Moss American, Sample 1, Grab, 2/1/87, 1030.

Compound	Concentration Spiked (PPB)	Sample Result (PPB)	Spike Result (PPB)	Spike Percent Recovery	Duplicate Spike Result (PPB)	Duplicate Percent Recovery	RPD
NAPHTHALENE	30	0	22	73	25	83	13
ACENAPHTHYLENE	30	0	23	77	27	90	16
ACENAPHTHENE	30	0.6	23	75	27	88	16
FLUORENE	30	0	22	73	27	90	20
PHENANTHRENE	30	0.6	25	81	28	91	11
ANTHRACENE	30	0	26	87	29	97	11
FLUORANTHENE	30	0	20	67	24	80	18
PYRENE	30	0	19	63	24	80	23
BENZO(A)ANTHRACENE	30	0	22	73	27	90	20
CHRYSENE	30	0	22	73	27	90	20
BENZO(B)FLUORANTHENE	30	0	22	73	26	87	17
BENZO(K)FLUORANTHENE	30	0	21	70	25	83	17
BENZO(A)PYRENE	30	0	23	77	26	87	12
INDENO(123-CD)PYRENE	30	0	22	73	26	87	17
DIBENZO(AH)ANTHRA.	30	0	21	70	25	83	17
BENZO(GHI)PERYLENE	30	0	21	70	25	83	17
2,3-BENZOFURAN *	30	---	21	70	24	80	13
TERPHENYL-d14 *	30	---	18	60	23	77	24

* Surrogate

$$\text{Percent Recovery} = \frac{\text{Spike Result} - \text{Sample Result}}{\text{Concentration Spiked}} \times 100$$

$$\text{RPD} = \frac{\text{Spike Result} - \text{Duplicate Spike Result}}{\text{Spike Result} + \text{Duplicate Spike Result}} \times 200$$

Comments: No cleanup procedures were applied to this sample.

CH2M HILL
ENVIRONMENTAL

M A T R I X S P I K E R E S U L T S

Laboratory No.: SIP-WO-S.WK1 Fraction: PAH by GC/FID Matrix: Soil

Sample Description: Matrix spike and matrix spike duplicate into soil sample labeled Moss American, Sample 1, Grab, 2/1/87, 1030.

Compound	Concentration Spiked (PPB)	Sample Result (PPB)	Spike Result (PPB)	Spike Percent Recovery	Duplicate Spike Result (PPB)	Duplicate Percent Recovery	RPD
NAPHTHALENE	30	0	22	73	25	83	13
ACENAPHTHYLENE	30	0	23	77	27	90	16
ACENAPHTHENE	30	0.6	23	75	27	88	16
FLUORENE	30	0	22	73	27	90	20
PHENANTHRENE	30	0.6	25	81	28	91	11
ANTHRACENE	30	0	26	87	29	97	11
FLUORANTHENE	30	0	20	67	24	80	18
PYRENE	30	0	19	63	24	80	23
BENZO(A)ANTHRACENE	30	0	22	73	27	90	20
CHRYSENE	30	0	22	73	27	90	20
BENZO(B)FLUORANTHENE	30	0	22	73	26	87	17
BENZO(K)FLUORANTHENE	30	0	21	70	25	83	17
BENZO(A)PYRENE	30	0	23	77	26	87	12
INDENO(123-CD)PYRENE	30	0	22	73	26	87	17
DIBENZO(AH)ANTHRA.	30	0	21	70	25	83	17
BENZO(GHI)PERYLENE	30	0	21	70	25	83	17
2,3-BENZOFURAN *	30	---	21	70	24	80	13
TERPHENYL-d14 *	30	---	18	60	23	77	24

* Surrogate

$$\text{Percent Recovery} = \frac{\text{Spike Result} - \text{Sample Result}}{\text{Concentration Spiked}} \times 100$$

$$\text{RPD} = \frac{\text{Spike Result} - \text{Duplicate Spike Result}}{\text{Spike Result} + \text{Duplicate Spike Result}} \times 200$$

Comments: No cleanup procedures were applied to this sample.

CH2M HILL
ENVIRONMENTAL

M A T R I X S P I K E R E S U L T S

Laboratory No.: S1A-WO-S.WK1 Fraction: Phenolic Acids by GC/FID Matrix: Soil

Sample Description: Matrix spike and matrix spike duplicate into soil sample labeled Moss American, Sample 1, Grab, 2/1/87, 1030.

Compound	Concentration Spiked (PPB)	Sample Result (PPB)	Spike Result (PPB)	Spike Percent Recovery	Duplicate Spike Result (PPB)	Duplicate Percent Recovery	RPD
PHENOL	60	0	45	75	52	87	14
2-CHLOROPHENOL	60	0	46	77	52	87	12
2-NITROPHENOL	60	0	48	80	54	90	12
2,4-DIMETHYLPHENOL	60	0	49	82	56	93	13
2,4-DICHLOROPHENOL	60	0	48	80	54	90	12
4-CL-3-ME PHENOL	60	0	46	77	56	93	20
2,4,6-TRICL PHENOL	60	0	47	78	56	93	17
2,4-DINITROPHENOL	60	0	29	48	49	82	51
4-NITROPHENOL	60	0	26	43	46	77	56
2-ME-4,6-DINO PHENOL	60	0	36	60	53	88	38
PENTACHLOROPHENOL	60	0	53	88	66	110	22
2,4,6-TRIBROMOPHENOL *	60	---	40	67	52	87	26

* Surrogate

$$\text{Percent Recovery} = \frac{\text{Spike Result} - \text{Sample Result}}{\text{Concentration Spiked}} \times 100$$

$$\text{RPD} = \frac{\text{Spike Result} - \text{Duplicate Spike Result}}{\text{Spike Result} + \text{Duplicate Spike Result}} \times 200$$

Comments: No cleanup procedures were applied to this sample.

CH2M HILL
ENVIRONMENTAL LAB

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/FID

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 5, Grab, 2/1/87, 1130

Lab No: Sam 5
Date Received: 02/10/87

Date Extracted: 02/18/87
Date Analyzed: 03/07/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	< 1	Phenol	1
Acenaphthylene	0.6	2-Chlorophenol	< 2
Acenaphthene	0.6	2-Nitrophenol	< 2
Fluorene	0.9	2,4-Dimethylphenol	< 2
Phenanthrene	1.5	2,4-Dichlorophenol	< 2
Anthracene	1.4	4-Chloro-3-methylphenol	2
Fluoranthene	3.3	2,4,6-Trichlorophenol	< 2
Pyrene	4.7	2,4-Dinitrophenol	< 5
Benzo(a)anthracene	2.9	4-Nitrophenol	< 5
Chrysene	3.5	4,6-Dinitro-2-methylphenol	< 5
Benzo(b)fluoranthene	< 1	Pentachlorophenol	< 5
Benzo(k)fluoranthene	4.5		
Benzo(a)pyrene	17		
Indeno(1,2,3-cd)pyrene	4.7		
Dibenzo(a,h)anthracene	< 1		
Benzo(g,h,i)perylene	6.3		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 96 % Terphenyl-d14 = 112 %	2,4,6-Tribromophenol = 120 %

Comments: No cleanup procedures were applied to this sample.

Reviewed by: J. Smiley

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/FID

Client: CH2M HILL/BLD
Sample Identification: Soil - Moss American, Sample 7, Grab, 2/1/87, 1200

Lab No: Sam 7
Date Received: 02/10/87

Date Extracted: 02/18/87
Date Analyzed: 03/08/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	51	Phenol	< 100
Acenaphthylene	55	2-Chlorophenol	< 100
Acenaphthene	880	2-Nitrophenol	< 100
Fluorene	640	2,4-Dimethylphenol	< 100
Phenanthrene	2100	2,4-Dichlorophenol	< 100
Anthracene	760	4-Chloro-3-methylphenol	< 100
Fluoranthene	3000	2,4,6-Trichlorophenol	< 100
Pyrene	2600	2,4-Dinitrophenol	< 250
Benzo(a)anthracene	190	4-Nitrophenol	1600
Chrysene	690	4,6-Dinitro-2-methylphenol	< 250
Benzo(b)fluoranthene	< 50	Pentachlorophenol	1300
Benzo(k)fluoranthene	40		
Benzo(a)pyrene	< 50		
Indeno(1,2,3-cd)pyrene	< 50		
Dibenzo(a,h)anthracene	< 50		
Benzo(g,h,i)perylene	< 50		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = * %	2,4,6-Tribromophenol = * %
Terphenyl-d14 = * %	

Comments: No cleanup procedures were applied to this sample.
* Surrogate recoveries were not determined due to the largedilution needed for analysis.

Reviewed by: Jo Smiley

[redacted]

ANALYTICAL REPORT
PAH/PHENOLS by GC/FID

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 15, Grab, 2/1/87, 1400

Lab No: Saa 15
Date Received: 02/10/87

Date Extracted: 02/18/87
Date Analyzed: 03/19/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	< 1	Phenol	< 2
Acenaphthylene	0.6	2-Chlorophenol	< 2
Acenaphthene	< 1	2-Nitrophenol	< 2
Fluorene	< 1	2,4-Dimethylphenol	< 2
Phenanthrene	2.0	2,4-Dichlorophenol	< 2
Anthracene	1.5	4-Chloro-3-methylphenol	< 2
Fluoranthene	4.6	2,4,6-Trichlorophenol	< 2
Pyrene	6.3	2,4-Dinitrophenol	< 5
Benzo(a)anthracene	< 1	4-Nitrophenol	< 5
Chrysene	8.8	4,6-Dinitro-2-methylphenol	< 5
Benzo(b)fluoranthene	< 1	Pentachlorophenol	4
Benzo(k)fluoranthene	8.7		
Benzo(a)pyrene	2.9		
Indeno(1,2,3-cd)pyrene	3.1		
Dibenzo(a,h)anthracene	1.2		
Benzo(g,h,i)perylene	4.6		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 92 %	2,4,6-Tribromophenol = 77 %
Terphenyl-d14 = 93 %	

Comments: No cleanup procedures were applied to this sample.

Reviewed by: Jo Smiley

[redacted]

ANALYTICAL REPORT
PAH/PHENOLS by GC/FID

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 17, Grab, 2/1/87, 1430

Lab No: Sam 17
Date Received: 02/10/87
Date Extracted: 02/18/87
Date Analyzed: 03/08/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	0.8	Phenol	< 2
Acenaphthylene	1.8	2-Chlorophenol	< 2
Acenaphthene	1.0	2-Nitrophenol	< 2
Fluorene	0.6	2,4-Dimethylphenol	< 2
Phenanthrene	5.0	2,4-Dichlorophenol	< 2
Anthracene	3.7	4-Chloro-3-methylphenol	< 2
Fluoranthene	10	2,4,6-Trichlorophenol	< 2
Pyrene	40	2,4-Dinitrophenol	< 5
Benzo(a)anthracene	9.0	4-Nitrophenol	4
Chrysene	14	4,6-Dinitro-2-methylphenol	< 5
Benzo(b)fluoranthene	< 1	Pentachlorophenol	2
Benzo(k)fluoranthene	35		
Benzo(a)pyrene	14		
Indeno(1,2,3-cd)pyrene	< 1		
Dibenzo(a,h)anthracene	10		
Benzo(g,h,i)perylene	12		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 96 %	2,4,6-Tribromophenol = 112 %
Terphenyl-d14 = 74 %	

Comments: No cleanup procedures were applied to this sample.

Reviewed by: [signature]

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/MS

Client: CH2M HILL/GLO
Sample Identification: Laboratory Method Blank

Lab No: 04037BS2
Date Received: N/A

Date Extracted: 04/03/87
Date Analyzed: 04/05/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	< 0.4	Phenol	< 0.4
Acenaphthylene	< 0.4	2-Chlorophenol	< 0.4
Acenaphthene	< 0.4	2-Nitrophenol	< 0.4
Fluorene	< 0.4	2,4-Dimethylphenol	< 0.4
Phenanthrene	< 0.4	2,4-Dichlorophenol	< 0.4
Anthracene	< 0.4	4-Chloro-3-methylphenol	< 0.4
Fluoranthene	< 0.4	2,4,6-Trichlorophenol	< 0.4
Pyrene	< 0.4	2,4-Dinitrophenol	< 2.0
Benzo(a)anthracene	< 0.4	4-Nitrophenol	< 2.0
Chrysene	< 0.4	4,6-Dinitro-2-methylphenol	< 2.0
Benzo(b)fluoranthene	< 0.4	Pentachlorophenol	< 0.4
Benzo(k)fluoranthene	< 0.4		
Benzo(a)pyrene	< 0.4		
Indeno(1,2,3-cd)pyrene	< 0.4		
Dibenzo(a,h)anthracene	< 0.4		
Benzo(g,h,i)perylene	< 0.4		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 70 %	2,4,6-Tribromophenol = 71 %
Terphenyl-d14 = 78 %	

Comments:

Reviewed by: Ward Dickens

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/MS

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 1, Grab, 2/1/87, 1030

Lab No: Sam 1
Date Received: 02/10/87

Date Extracted: 04/03/87
Date Analyzed: 04/05/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	< 0.4	Phenol	< 0.4
Acenaphthylene	< 0.4	2-Chlorophenol	< 0.4
Acenaphthene	< 0.4	2-Nitrophenol	< 0.4
Fluorene	< 0.4	2,4-Dimethylphenol	< 0.4
Phenanthrene	< 0.4	2,4-Dichlorophenol	< 0.4
Anthracene	< 0.4	4-Chloro-3-methylphenol	< 0.4
Fluoranthene	< 0.4	2,4,6-Trichlorophenol	< 0.4
Pyrene	< 0.4	2,4-Dinitrophenol	< 2.0
Benzo(a)anthracene	< 0.4	4-Nitrophenol	< 2.0
Chrysene	< 0.4	4,6-Dinitro-2-methylphenol	< 2.0
Benzo(b)fluoranthene	< 0.4	Pentachlorophenol	< 0.4
Benzo(k)fluoranthene	< 0.4		
Benzo(a)pyrene	< 0.4		
Indeno(1,2,3-cd)pyrene	< 0.4		
Dibenzo(a,h)anthracene	< 0.4		
Benzo(g,h,i)perylene	< 0.4		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 71 %	2,4,6-Tribromophenol = 80 %
Terphenyl-d14 = 92 %	

Comments:

Reviewed by: Ward Dickens

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/MS

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 5, Grab, 2/1/87, 1130

Lab No: Sam 5
Date Received: 02/10/87
Date Extracted: 04/03/87
Date Analyzed: 04/05/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	< 0.4	Phenol	< 0.4
Acenaphthylene	< 0.4	2-Chlorophenol	< 0.4
Acenaphthene	< 0.4	2-Nitrophenol	< 0.4
Fluorene	< 0.4	2,4-Dimethylphenol	< 0.4
Phenanthrene	0.7	2,4-Dichlorophenol	< 0.4
Anthracene	0.5	4-Chloro-3-methylphenol	< 0.4
Fluoranthene	1.7	2,4,6-Trichlorophenol	< 0.4
Pyrene	1.8	2,4-Dinitrophenol	< 2.0
Benzo(a)anthracene	0.5	4-Nitrophenol	< 2.0
Chrysene	1.3	4,6-Dinitro-2-methylphenol	< 2.0
Benzo(b)fluoranthene	2.9	Pentachlorophenol	< 0.4
Benzo(k)fluoranthene	1.2		
Benzo(a)pyrene	1.0		
Indeno(1,2,3-cd)pyrene	1.8		
Dibenzo(a,h)anthracene	< 0.4		
Benzo(g,h,i)perylene	2.1		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 66 %	2,4,6-Tribromophenol = 80 %
Terphenyl-d14 = 77 %	

Comments: PAH and Phenol fractions were analyzed separately.

Reviewed by: Ward Dickens

ANALYTICAL REPORT
PAH/PHENOLS by GC/MS

Client: CH2M HILL/BLD
Sample Identification: Soil - Moss American, Sample 7, Grab, 2/1/87, 1200

Lab No: Sam 7
Date Received: 02/10/87

Date Extracted: 04/03/87
Date Analyzed: 04/05/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	34	Phenol	< 0.8
Acenaphthylene	< 4.0	2-Chlorophenol	< 0.8
Acenaphthene	440	2-Nitrophenol	< 0.8
Fluorene	330	2,4-Dimethylphenol	< 0.8
Phenanthrene	1100	2,4-Dichlorophenol	< 0.8
Anthracene	330	4-Chloro-3-methylphenol	< 0.8
Fluoranthene	1800	2,4,6-Trichlorophenol	< 0.8
Pyrene	1400	2,4-Dinitrophenol	< 4.0
Benzo(a)anthracene	260	4-Nitrophenol	< 4.0
Chrysene	330	4,6-Dinitro-2-methylphenol	< 4.0
Benzo(b)fluoranthene	210	Pentachlorophenol	< 0.8
Benzo(k)fluoranthene	96		
Benzo(a)pyrene	120		
Indeno(1,2,3-cd)pyrene	48		
Dibenzo(a,h)anthracene	14		
Benzo(g,h,i)perylene	51		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 51 %	2,4,6-Tribromophenol = 77 %
Terphenyl-d14 = 75 %	

Comments: PAH and Phenolic fractions were analyzed separately.

Reviewed by: Ward Dukes

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/MS

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 15, Grab, 2/1/87, 1400

Lab No: Sam 15
Date Received: 02/10/87

Date Extracted: 04/03/87
Date Analyzed: 04/05/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	< 0.4	Phenol	< 0.4
Acenaphthylene	< 0.4	2-Chlorophenol	< 0.4
Acenaphthene	< 0.4	2-Nitrophenol	< 0.4
Fluorene	< 0.4	2,4-Dimethylphenol	< 0.4
Phenanthrene	0.9	2,4-Dichlorophenol	< 0.4
Anthracene	0.4	4-Chloro-3-methylphenol	< 0.4
Fluoranthene	2.3	2,4,6-Trichlorophenol	< 0.4
Pyrene	2.1	2,4-Dinitrophenol	< 2.0
Benzo(a)anthracene	0.4	4-Nitrophenol	< 2.0
Chrysene	1.5	4,6-Dinitro-2-methylphenol	< 2.0
Benzo(b)fluoranthene	3.0	Pentachlorophenol	< 0.4
Benzo(k)fluoranthene	1.1		
Benzo(a)pyrene	0.9		
Indeno(1,2,3-cd)pyrene	1.5		
Dibenzo(a,h)anthracene	< 0.4		
Benzo(g,h,i)perylene	2.1		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 50 %	2,4,6-Tribromophenol = 77 %
Terphenyl-d14 = 73 %	

Comments: PAH and Phenol fractions were analyzed separately.

Reviewed by: Wood Stickney

ANALYTICAL REPORT
PAH/PHENOLS by GC/MS

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 17, Grab, 2/1/87, 1430

Lab No: Sam 17
Date Received: 02/10/87

Date Extracted: 04/03/87
Date Analyzed: 04/05/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	0.5	Phenol	< 0.4
Acenaphthylene	< 0.4	2-Chlorophenol	< 0.4
Acenaphthene	< 0.4	2-Nitrophenol	< 0.4
Fluorene	< 0.4	2,4-Diethylphenol	< 0.4
Phenanthrene	1.6	2,4-Dichlorophenol	< 0.4
Anthracene	0.9	4-Chloro-3-methylphenol	< 0.4
Fluoranthene	5.1	2,4,6-Trichlorophenol	< 0.4
Pyrene	17.0	2,4-Dinitrophenol	< 2.0
Benzo(a)anthracene	1.9	4-Nitrophenol	< 2.0
Chrysene	3.9	4,6-Dinitro-2-methylphenol	< 2.0
Benzo(b)fluoranthene	5.8	Pentachlorophenol	< 0.4
Benzo(k)fluoranthene	3.6		
Benzo(a)pyrene	3.6		
Indeno(1,2,3-cd)pyrene	0.5		
Dibenzo(a,h)anthracene	< 0.4		
Benzo(g,h,i)perylene	< 0.4		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 32 %	2,4,6-Tribromophenol = 82 %
Terphenyl-d14 = 44 %	

Comments: PAH and Phenol fractions were analyzed separately.

Reviewed by: Ward Stickles

M A T R I X S P I K E R E S U L T S

Laboratory No.: S1PAH-S.WK1 Fraction: PAH by GC/MS Matrix: Soil

Sample Description: Matrix spike and matrix spike duplicate into soil sample labeled Moss American, Sample 1, Grab, 2/1/87, 1030.

Compound	Concentration Spiked (PPB)	Sample Result (PPB)	Spike Result (PPB)	Spike Percent Recovery	Duplicate Spike Result (PPB)	Duplicate Percent Recovery	RPD
NAPHTHALENE	50	0	39	78	45	90	14
ACENAPHTHYLENE	50	0	40	80	44	88	10
ACENAPHTHENE	50	0	40	80	43	86	7
FLUORENE	50	0	41	82	42	84	2
PHENANTHRENE	50	0	42	84	46	92	9
ANTHRACENE	50	0	40	80	44	88	10
FLUORANTHENE	50	0	44	88	46	92	4
PYRENE	50	0	45	90	47	94	4
BENZO(A)ANTHRACENE	50	0	43	86	42	84	2
CHRYSENE	50	0	42	84	42	84	0
BENZO(B)FLUORANTHENE	50	0	42	84	45	90	7
BENZO(K)FLUORANTHENE	50	0	46	92	45	90	2
BENZO(A)PYRENE	50	0	44	88	45	90	2
INDENO(123-CD)PYRENE	50	0	41	82	47	94	14
DIBENZO(AH)ANTHRA.	50	0	41	82	47	94	14
BENZO(GHI)PERYLENE	50	0	40	80	46	92	14
2,3-BENZOFURAN *	50	---	33	66	38	76	14
TERPHENYL-d14 *	50	---	43	86	46	92	7

* Surrogate

$$\text{Percent Recovery} = \frac{\text{Spike Result} - \text{Sample Result}}{\text{Concentration Spiked}} \times 100$$

$$\text{RPD} = \frac{\text{Spike Result} - \text{Duplicate Spike Result}}{\text{Spike Result} + \text{Duplicate Spike Result}} \times 200$$

Comments:

CH2M HILL
ENVIRONMENTAL LABORATORY

M A T R I X S P I K E R E S U L T S

Laboratory No.: S1AC-S.WK1 Fraction: Phenolic Acids by GC/MS Matrix: Soil

Sample Description: Matrix spike and matrix spike duplicate into soil sample labeled Moss American, Sample 1, Grab, 2/1/87, 1030.

Compound	Concentration Spiked (PPB)	Sample Result (PPB)	Spike Result (PPB)	Spike Percent Recovery	Duplicate Spike Result (PPB)	Duplicate Percent Recovery	RPD
PHENOL	100	0	79	79	78	78	1
2-CHLOROPHENOL	100	0	76	76	74	74	3
2-NITROPHENOL	100	0	80	80	85	85	6
2,4-DIMETHYLPHENOL	100	0	74	74	78	78	5
2,4-DICHLOROPHENOL	100	0	81	81	85	85	5
4-CL-3-ME PHENOL	100	0	84	84	89	89	6
2,4,6-TRICL PHENOL	100	0	82	82	86	86	5
2,4-DINITROPHENOL	100	0	73	73	71	71	3
4-NITROPHENOL	100	0	82	82	85	85	4
2-ME-4,6-DINO PHENOL	100	0	82	82	85	85	4
PENTACHLOROPHENOL	100	0	85	85	91	91	7
2,4,6-TRIBROMOPHENOL *	100	---	87	87	88	88	1

---

* Surrogate

$$\text{Percent Recovery} = \frac{\text{Spike Result} - \text{Sample Result}}{\text{Concentration Spiked}} \times 100$$

$$\text{RPD} = \frac{\text{Spike Result} - \text{Duplicate Spike Result}}{\text{Spike Result} + \text{Duplicate Spike Result}} \times 200$$

Comments:

CH2M HILL
ENVIRONMENTAL LABORATORY

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/MS

Client: CH2M HILL/GLO
Sample Identification: Laboratory Method Blank

Lab No: 04037BS1
Date Received: N/A

Date Extracted: 04/03/87
Date Analyzed: 04/05/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	< 0.4	Phenol	< 0.4
Acenaphthylene	< 0.4	2-Chlorophenol	< 0.4
Acenaphthene	< 0.4	2-Nitrophenol	< 0.4
Fluorene	< 0.4	2,4-Dimethylphenol	< 0.4
Phenanthrene	< 0.4	2,4-Dichlorophenol	< 0.4
Anthracene	< 0.4	4-Chloro-3-methylphenol	< 0.4
Fluoranthene	< 0.4	2,4,6-Trichlorophenol	< 0.4
Pyrene	< 0.4	2,4-Dinitrophenol	< 2.0
Benzo(a)anthracene	< 0.4	4-Nitrophenol	< 2.0
Chrysene	< 0.4	4,6-Dinitro-2-methylphenol	< 2.0
Benzo(b)fluoranthene	< 0.4	Pentachlorophenol	< 0.4
Benzo(k)fluoranthene	< 0.4		
Benzo(a)pyrene	< 0.4		
Indeno(1,2,3-cd)pyrene	< 0.4		
Dibenzo(a,h)anthracene	< 0.4		
Benzo(g,h,i)perylene	< 0.4		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 92 %	2,4,6-Tribromophenol = 85 %
Terphenyl-d14 = 87 %	

Comments: No cleanup procedures were applied to this sample.

Reviewed by: Wendi Dickson

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/MS

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 1, Grab, 2/1/87, 1030

Lab No: Sam 1
Date Received: 02/10/87

Date Extracted: 04/03/87
Date Analyzed: 04/05/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	< 0.4	Phenol	< 0.4
Acenaphthylene	< 0.4	2-Chlorophenol	< 0.4
Acenaphthene	< 0.4	2-Nitrophenol	< 0.4
Fluorene	< 0.4	2,4-Dimethylphenol	< 0.4
Phenanthrene	< 0.4	2,4-Dichlorophenol	< 0.4
Anthracene	< 0.4	4-Chloro-3-methylphenol	< 0.4
Fluoranthene	< 0.4	2,4,6-Trichlorophenol	< 0.4
Pyrene	< 0.4	2,4-Dinitrophenol	< 2.0
Benzo(a)anthracene	< 0.4	4-Nitrophenol	< 2.0
Chrysene	< 0.4	4,6-Dinitro-2-methylphenol	< 2.0
Benzo(b)fluoranthene	< 0.4	Pentachlorophenol	< 0.4
Benzo(k)fluoranthene	< 0.4		
Benzo(a)pyrene	< 0.4		
Indeno(1,2,3-cd)pyrene	< 0.4		
Dibenzo(a,h)anthracene	< 0.4		
Benzo(g,h,i)perylene	< 0.4		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 66 %	2,4,6-Tribromophenol = 78 %
Terphenyl-d14 = 85 %	

Comments: No cleanup procedures were applied to this sample.

Reviewed by: Ward Dickens

CH2M HILL

ANALYTICAL REPORT
PAH/PHENOLS by GC/MS

Client: CH2M HILL/GLO
Sample Identification: Soil - Moss American, Sample 7, Grab, 2/1/87, 1200

Lab No: Sam 7
Date Received: 02/10/87

Date Extracted: 04/03/87
Date Analyzed: 04/05/87

PAH COMPOUNDS	CONCENTRATION (PPM)	PHENOLIC ACID COMPOUNDS	CONCENTRATION (PPM)
Naphthalene	70	Phenol	< 10
Acenaphthylene	45	2-Chlorophenol	< 10
Acenaphthene	600	2-Nitrophenol	< 10
Fluorene	420	2,4-Diaethylphenol	< 10
Phenanthrene	1300	2,4-Dichlorophenol	< 10
Anthracene	340	4-Chloro-3-aethylphenol	< 10
Fluoranthene	2200	2,4,6-Trichlorophenol	< 10
Pyrene	1600	2,4-Dinitrophenol	< 50
Benzo(a)anthracene	440	4-Nitrophenol	< 50
Chrysene	550	4,6-Dinitro-2-aethylphenol	< 50
Benzo(b)fluoranthene	350	Pentachlorophenol	< 10
Benzo(k)fluoranthene	170		
Benzo(a)pyrene	210		
Indeno(1,2,3-cd)pyrene	110		
Dibenzo(a,h)anthracene	< 10		
Benzo(g,h,i)perylene	95		

SURROGATE RECOVERIES	SURROGATE RECOVERY
2,3-Benzofuran = 48 %	2,4,6-Tribromophenol = * %
Terphenyl-d14 = 29 %	

Comments: No cleanup procedures were applied to this sample.
* Surrogate not recovered.

Reviewed by: Ward Dickson

M A T R I X S P I K E R E S U L T S

Laboratory No.: S1PAH-S.WK1 Fraction: PAH by GC/MS Matrix: Soil

Sample Description: Matrix spike and matrix spike duplicate into soil sample labeled Moss American, Sample 1, Grab, 2/1/87, 1030.

Compound	Concentration Spiked (PPB)	Sample Result (PPB)	Spike Result (PPB)	Spike Percent Recovery	Duplicate Spike Result (PPB)	Duplicate Percent Recovery	RPD
NAPHTHALENE	50	0	40	80	46	92	14
ACENAPHTHYLENE	50	0	41	82	44	88	7
ACENAPHTHENE	50	0	41	82	42	84	2
FLUORENE	50	0	41	82	42	84	2
PHENANTHRENE	50	0	41	82	43	86	5
ANTHRACENE	50	0	38	76	40	80	5
FLUORANTHENE	50	0	44	88	44	88	0
PYRENE	50	0	45	90	45	90	0
BENZO(A)ANTHRACENE	50	0	42	84	38	76	10
CHRYSENE	50	0	40	80	38	76	5
BENZO(B)FLUORANTHENE	50	0	47	94	46	92	2
BENZO(K)FLUORANTHENE	50	0	45	90	47	94	4
BENZO(A)PYRENE	50	0	46	92	46	92	0
INDENO(123-CD)PYRENE	50	0	57	114	52	104	9
DIBENZO(AH)ANTHRA.	50	0	59	118	55	110	7
BENZO(GHI)PERYLENE	50	0	55	110	51	102	8
2,3-BENZOFURAN *	50	---	35	70	41	82	16
TERPHENYL-d14 *	50	---	46	92	44	88	4

* Surrogate

$$\text{Percent Recovery} = \frac{\text{Spike Result} - \text{Sample Result}}{\text{Concentration Spiked}} \times 100$$

$$\text{RPD} = \frac{\text{Spike Result} - \text{Duplicate Spike Result}}{\text{Spike Result} + \text{Duplicate Spike Result}} \times 200$$

Comments: No cleanup procedures were applied to this sample.

CH2M HILL
ENVIRONMENTAL LABORATORY

M A T R I X S P I K E R E S U L T S

Laboratory No.: S1AC-S.WK1 Fraction: Phenolic Acids by GC/MS Matrix: Soil

Sample Description: Matrix spike and matrix spike duplicate into soil sample labeled Moss American, Sample 1, Grab, 2/1/87, 1030.

Compound	Concentration Spiked (PPB)	Sample Result (PPB)	Spike Result (PPB)	Spike Percent Recovery	Duplicate Spike Result (PPB)	Duplicate Percent Recovery	RPD
PHENOL	100	0	73	73	80	80	9
2-CHLOROPHENOL	100	0	70	70	79	79	12
2-NITROPHENOL	100	0	80	80	88	88	10
2,4-DIMETHYLPHENOL	100	0	74	74	82	82	10
2,4-DICHLOROPHENOL	100	0	79	79	87	87	10
4-CL-3-ME PHENOL	100	0	87	87	90	90	3
2,4,6-TRICL PHENOL	100	0	84	84	87	87	4
2,4-DINITROPHENOL	100	0	75	75	74	74	1
4-NITROPHENOL	100	0	90	90	84	84	7
2-ME-4,6-DINO PHENOL	100	0	86	86	80	80	7
PENTACHLOROPHENOL	100	0	84	84	90	90	7
2,4,6-TRIBROMOPHENOL *	100	---	91	91	86	86	6

---

* Surrogate

$$\text{Percent Recovery} = \frac{\text{Spike Result} - \text{Sample Result}}{\text{Concentration Spiked}} \times 100$$

$$\text{RPD} = \frac{\text{Spike Result} - \text{Duplicate Spike Result}}{\text{Spike Result} + \text{Duplicate Spike Result}} \times 200$$

Comments: No cleanup procedures were applied to this sample.

CH2M HILL
ENVIRONMENTAL LABORATORY

Appendix D
SAMPLING PLAN
FOR THE
PHASE I REMEDIAL INVESTIGATION
AT THE MOSS-AMERICAN SITE

CONTENTS

Figures

Tables

GLT595/31

SAMPLING PLAN
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
MOSS-AMERICAN SITE MILWAUKEE, WISCONSIN

1.0 OBJECTIVE

This Sampling Plan (SP) has been prepared to provide the strategy for data gathering during the Phase I remedial investigation at the Moss-American site. This SP has been designed to address the data uses and data quality objectives (DQO's) developed in the QAPP. The objectives of this SP are to:

- Summarize the methods used to select the sample locations
- Define the number of samples to be collected
- Describe the procedures used for sample collection, preservation, packaging, and transport
- Define the necessary documentation for sample custody and recordkeeping

In summary, this Sampling Plan presents the procedures to be used for soil boring and installing monitoring wells for performing hydrologic studies, and for obtaining samples of groundwater, surface water, sediment, and soil at the Moss-American site and in the Little Menomonee River.

The following sampling activities will occur during this Phase I remedial investigation. An estimated 15 shallow, 5 medium, and 3 deep monitoring wells will be installed. Subsurface soil samples will also be collected during well installation. Groundwater samples will be taken from all 23 monitoring wells. Approximately 250 surface soil samples will be taken during the initial screening using a split spoon sampler. Approximately 257 sediment samples from the Little Menomonee River will be taken during the initial screening using a hand corer. Eight surface water samples will be taken from locations onsite and in the river. In addition, several field blanks and replicate samples will be collected from each media to meet QA/QC standards.

2.0 SAMPLE LOCATIONS AND DESIGNATION

2.1 SAMPLE LOCATIONS

Surface Soil Sampling

A large number of soil samples are necessary to estimate the existence and extent of contaminant migration. The initial soil screening strategy (as defined in Task G1 of the Work Plan) to identify representative sample locations is an alternate grid-point method. This involves sampling from within large grid spacing, analyzing the samples using two screening methods, and then reducing the spacing for additional sample locations, to define limits of contamination more accurately. The sample locations consist of using a 200-foot interval initially, which is then reduced to 100 feet, if necessary, based on analyses of the 200-foot grid samples. Figure D-1 shows the grid spacing at 100-foot intervals. These grid points will be staked during the site and river survey program (Task FM).

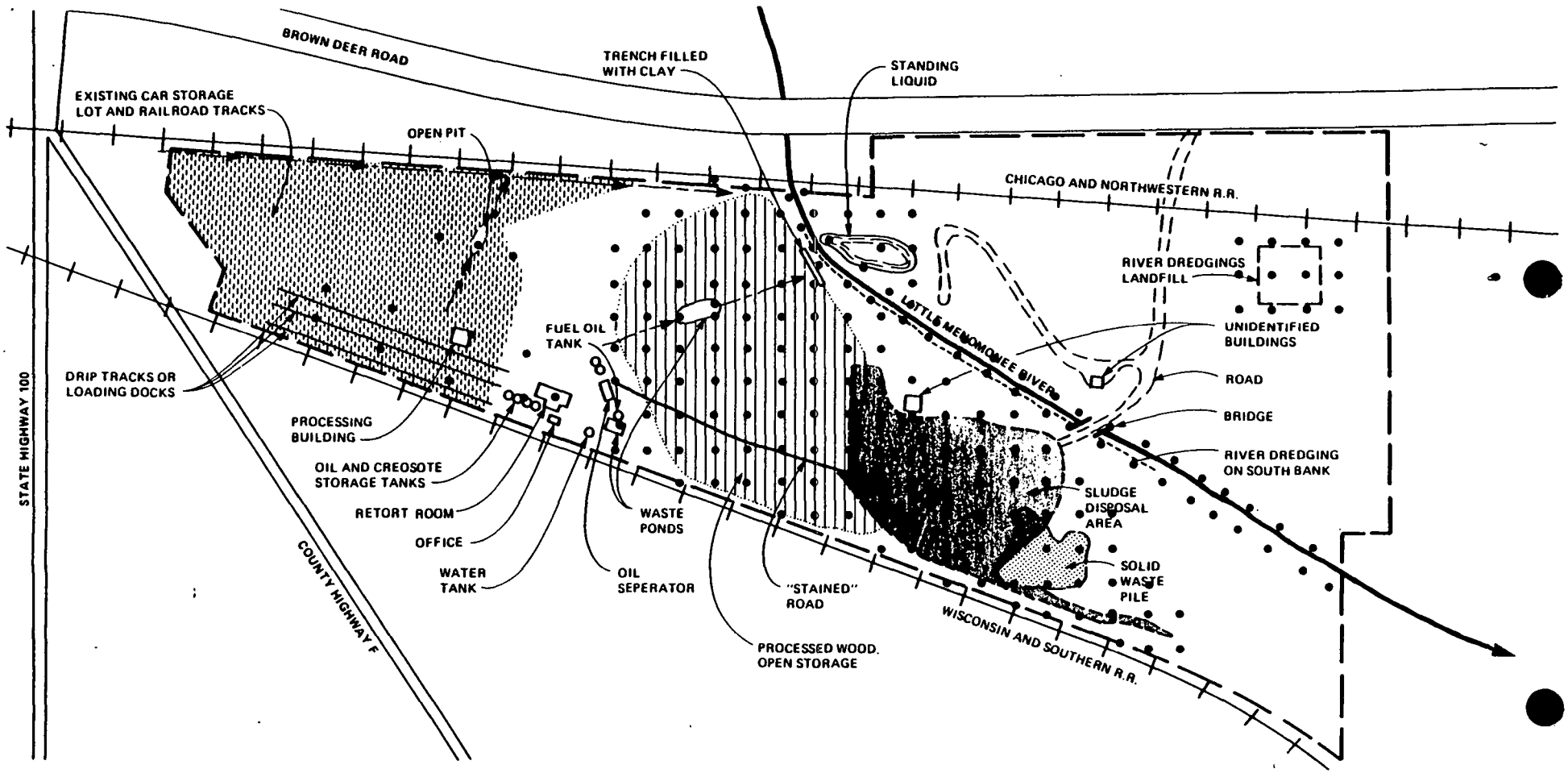
An estimated 250 total surface soil sample locations will be identified. Initial soil screening includes 190 sample locations within areas of apparent disposal, spillage, and processing. In addition, 46 sample locations will be selected to test for the presence of contamination in unknown or unsuspected processing areas. Seven additional samples will be taken from offsite locations to establish background levels. Seven other sampling locations will be identified during the site surveying to measure levels in the active railroad beds.

Subsurface Soil Sampling

Split-spoon samples will be obtained at all of the proposed monitoring well locations during drilling operations. Continuous split-spoon samples will be collected from the ground surface to a depth of 20 feet (or the bottom of the well). Split-spoon samples will be obtained at 5-foot intervals thereafter.

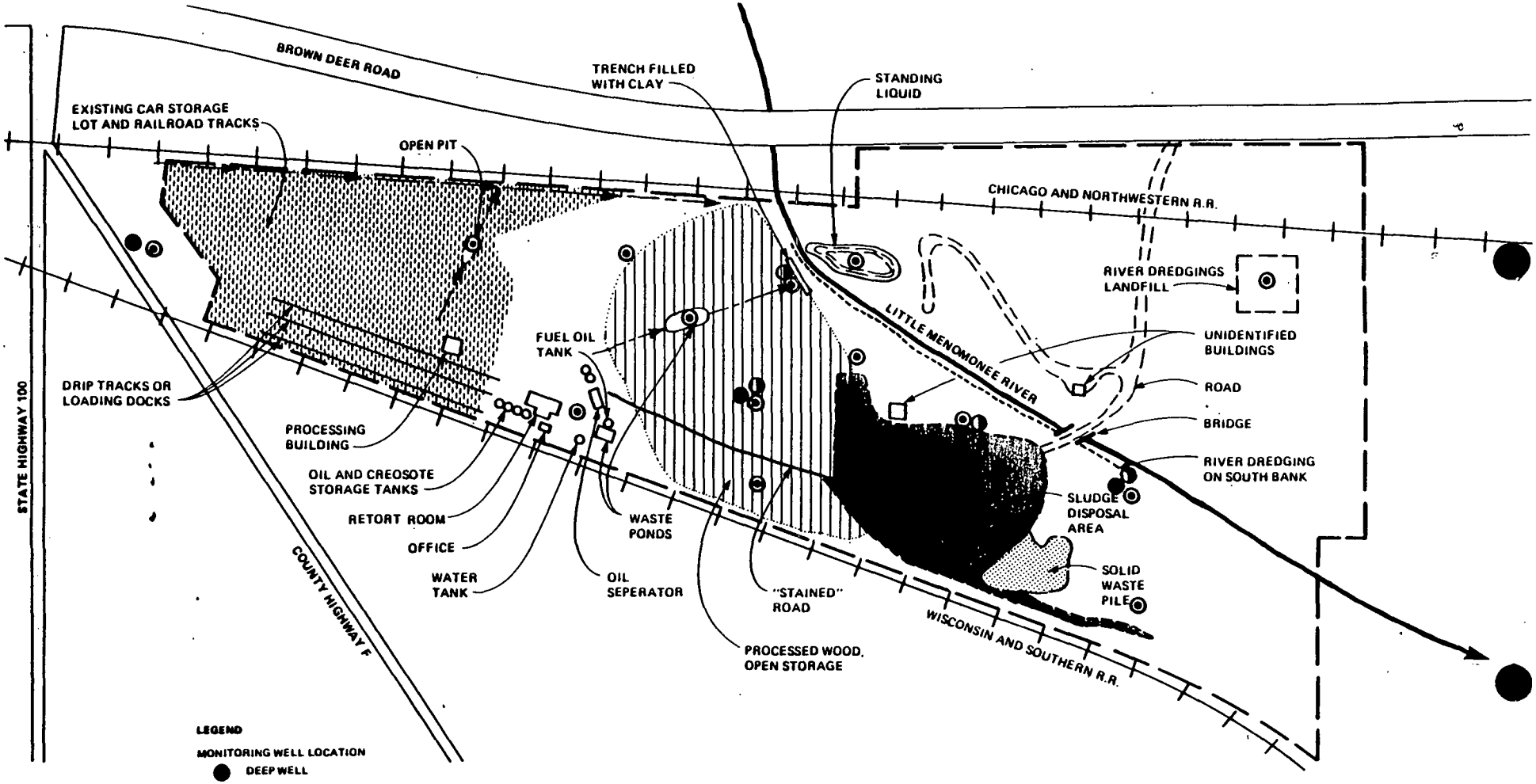
Groundwater Sampling

Groundwater samples will be collected from the 23 monitoring wells (15 shallow, 5 intermediate, and 3 deep) which will be installed onsite. Figure D-2 shows the approximate location of the monitoring wells to be sampled. Final monitoring well locations will be determined based on results of the initial soil screening analyses. Some of the wells will be grouped in clusters to provide vertical gradient and contaminant level data at the same points. The three deep wells will be



NOTE: This is a composite map of features that at one time had been on the site. Many of these have since been dismantled or covered.

FIGURE D-1
PROPOSED SURFACE SOIL SAMPLE LOCATIONS
MOSS AMERICAN



NOTE: This is a composite map of features that at one time had been on the site. Many of these have since been dismantled or covered.

FIGURE D-2
PROPSED GROUNDWATER MONITORING WELLS
MOSS AMERICAN

distributed to provide upgradient water quality data as well as more regional groundwater gradient conditions. Intermediate depth wells will be installed to intercept potential contaminant migration in areas of heaviest soil contamination based on the results of the initial surface soil screening analyses. The shallow monitoring wells will be distributed to provide broad based data points for contaminated as well as clean areas, and to assist in quantification of contaminated groundwater. All well screens will be placed in sand or gravel seams, if encountered.

Surface Water Sampling

Eight surface water grab samples will be collected from the following planned locations:

- In the drainage ditch on the north side of the site (1) where the ditch enters the site; and (2) just before the ditch discharges to the Little Menomonee River.

- In the Little Menomonee River (3) upstream of the site; (4) just below the confluence with the drainage ditch; (5) where the little Menomonee exits the site; (6,7) at downstream locations to be selected after the river survey is complete; and; (8) just before the confluence with the Menomonee River.

The eight surface water sampling locations will be staked and flagged during the site and river surveying (Task FM), so that the locations can be reestablished if more sampling is required. Distance and direction to a permanent feature will also be recorded in the event that the stake is destroyed.

Sediment Sampling

The initial sediment screening consists of sampling the 5-mile length of the Little Menomonee River downstream of the site along profiled cross sections on the river. These profiles will be surveyed and marked during the site and river surveying, conducted prior to sampling. It is estimated that 26 such sections, 1,200 feet apart, will be required to characterize and quantify river sedimentation (Figure D-3).

Six sediment samples will be taken at each cross section: three at the 0-1 foot interval, and three at the 1-2 foot interval, resulting in a total of 156 total sediment samples from the 26 cross sections.

An additional 76 sediment sampling locations are planned at 300 foot intervals. Another 25 sediment sampling locations will be selected to include sampling of river dredgings and floodplain deposits and background locations upstream. These 25 sediment sampling locations will be determined following the field surveying.

In the initial sediment screening, every other cross section (13 sections with 6 samples each) will be sampled and screened using TC methods to determine oil and creosote constituent levels. Timely turnaround of analytical results will allow for adjustment of the expected concentration and location of the other 13 sediment sampling sections to better define the areas of the river containing contaminated sediments. Similarly, the remaining 76 sediment sampling locations based on 300-foot intervals may be adjusted depending on the results of the initial screening.

2.2 SAMPLE DESIGNATION

A CH2M HILL sample numbering system will be used to identify each sample for chemical or physical analysis, including replicate samples and field blanks. A Sample Management Office (SMO) number and a Central Region Lab (CRL) number will also be assigned to each sample at the same time. Refer to the User's Guide to the Contract Laboratory Program (July 1984) for an explanation of the SMO numbers. A listing of sample identification numbers will be maintained in the logbook by the Sample Team Leader. Each CH2M HILL sample number will consist of three components as described below:

Project Identification

A two-letter designation will be used to identify the site where the sample was collected. For this project it will be MA for Moss-American.

Sample Location

Each sample will be identified by an alpha-code corresponding to the sample type, followed by a three-digit sample location number. The alpha-codes are as follows:

MW--monitoring well, groundwater
SW--surface water
SS--surface soil
SB--soil boring
SD--river sediment
FB--field blank

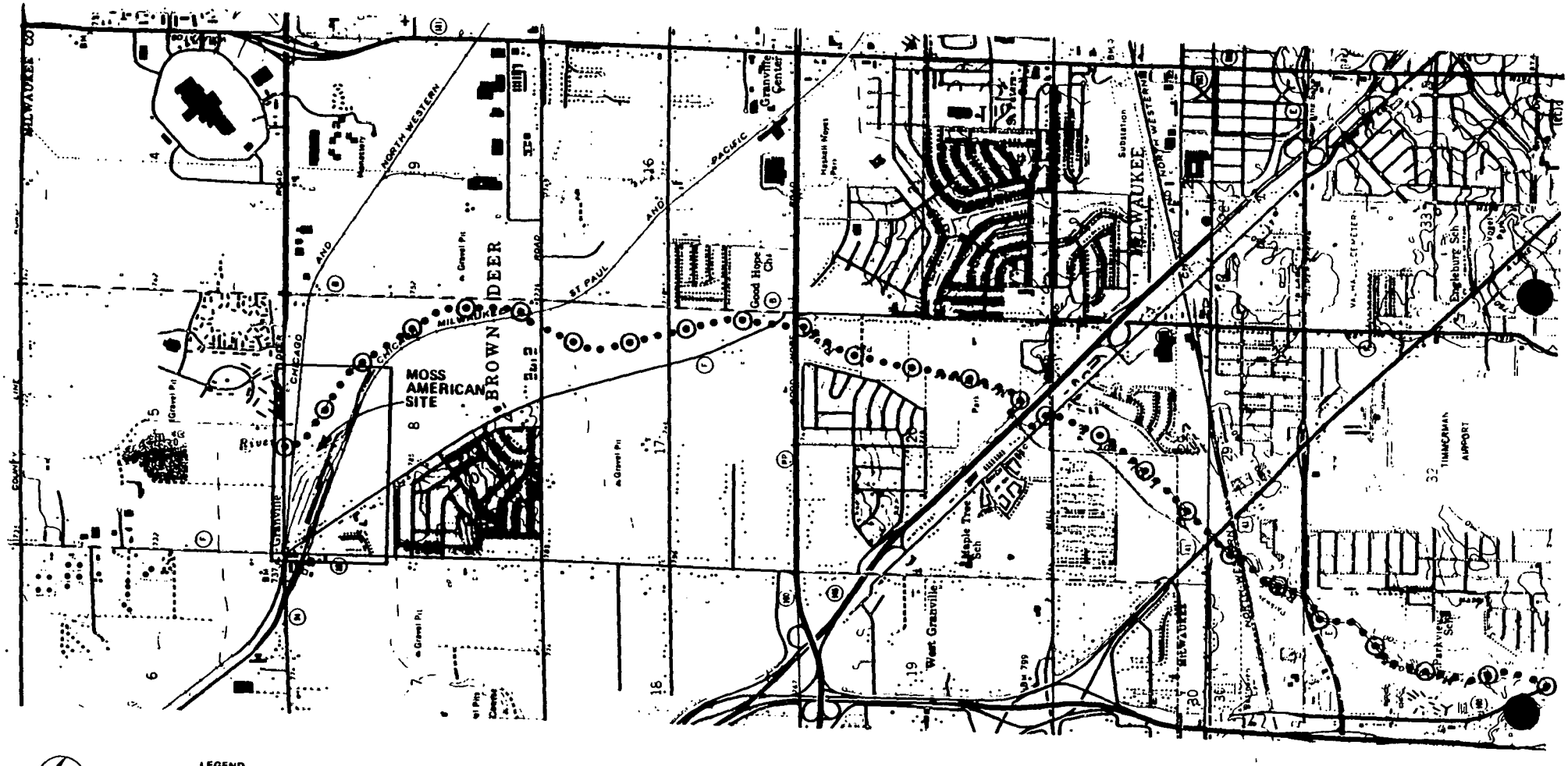


FIGURE D-3
SEDIMENT SAMPLE LOCATIONS
MOSS AMERICAN

BB--bottle blank
RP--replicate

Field blanks will have an FB followed by the alpha code for the type of blank (i.e., a surface water blank will be FBSW). Similarly, field replicates will be RP followed by the appropriate alpha code.

Sample Identifier

All samples will have a two-digit number as the last component of the sample identifier. The sampling events will start with 01 and progress upward.

Sample Number Examples

MA-MW1-02

Moss-American--groundwater sample 2 from monitoring well MW01

MA-SW2-01

Moss-American--surface water sample 1 from location SW02

MA-SS021-01

Moss-American--surface soil sample 1 from location SS021, depth to be noted in logbook

MA-SB008-04

Moss-American--soil boring sample 4 (split-spoon 4) from boring 8, depth to be noted in logbook

3.0 SAMPLING EQUIPMENT AND PROCEDURES

3.1 MONITORING WELL INSTALLATION

Twenty-three groundwater monitoring wells, (15 shallow, 5 intermediate, and 3 deep), will be installed at the locations shown on Figure D-3. The shallow well screens will be placed in the zone of fill, peat, and alluvial deposits at a depth of 15 feet. The intermediate depth well screens will be placed in the upper part of the till, in areas of heaviest soil contamination at a depth of 30 to 35 feet. The deep well screens will be placed deeper in the till at depths of 55 to 60 feet. Wells will be designated 'S' for shallow, 'I' for intermediate, and 'D' for deep. Screen-settings

will be adjusted to fit the apparent most permeable materials in each zone to be monitored.

All drilling and well installations will be supervised and logged by a hydrogeologist. Procedures will include:

- The steam cleaning of all drilling equipment, tools, and materials before commencement of drilling and between boreholes.
- Advancing of all well boreholes using hollow-stem augers or spun-in casing methods with a drop hammer to drive standard split-barrel samplers.
- Collection of soil samples at each location. Where there are nests of wells only the deep borehole will be sampled. Continuous split-spoon samples will be taken from the ground surface to a depth of 20 feet. Samples will be obtained at 5-foot intervals thereafter.

All monitoring wells will be constructed with 2-inch ID stainless steel well screens and risers. The threads may require Teflon taping if they do not appear to be water tight. The wells will be installed with 5-foot manufactured 0.010-inch slotted screens. All construction material will be steam cleaned before construction of the well.

All screened zones will be sand packed to two feet above the top of the well screen. The annular space between the well and the borehole will be backfilled with gravel or flint sand to 2 feet above the top of the well screen, followed by a 2-foot bentonite seal and a cement-bentonite grout mix to 3 feet below the surface. A locking protective stand pipe will be installed over the well stand pipe that is embedded in a concrete pad. At the edge of the pad, three protective posts will be installed.

3.2 AQUIFER TESTING

Hydraulic conductivity (K) will be determined at each monitoring well by "slug" test methods as described in Appendix I. Each slug test is conducted by lowering a weight of known volume into the well, thus causing a rise in the water level. The water level is then recorded against time as the water level drops to its static level which was recorded prior to the start of the test. If the recovery is fast, then an automatic water level recording device is needed, otherwise water levels and times will be recorded manually.

3.3 SAMPLE COLLECTION

Subsurface Soil Samples

Each soil sample collected from the boreholes will be scanned with an HNu or OVA to determine the presence of volatile organics in the soils (and groundwater if samples are saturated). The detection of volatile organics in the samples at known depth will optimize the placement of well screens.

Groundwater Samples

Prior to purging each well for sampling, a static water level measurement will be taken using a fiberglass tape with a steel sounding device attached to the end. The sounding device makes a popping noise at the water table. The tape will then be used to measure the total depth of the well to verify well identification. The depths to water will be measured with respect to the top of the well casing.

Each well to be sampled will be purged immediately prior to sampling using either a stainless steel or Teflon bailer, a submersible positive displacement pump (Johnson Keck), or a peristaltic pump. Discharge water will be collected and measured so that a minimum of five well volumes are removed prior to sample collection. The purpose of this purging is to ensure that the well has been flushed of standing water and contains fresh water from the aquifer. If pumps are used, the bottom 5 feet of hose will be Teflon so the hose will not contaminate the well or well water.

After the well has been purged, the samples will be collected using a stainless steel or Teflon bottom loading bailer. Approximately one-half volume of well water will be removed with a stainless steel bailer prior to the retention of the sample in sample containers. The bailers will be raised and lowered on a thin stainless steel cable.

All sampling equipment will be cleaned between wells by scrubbing with a trisodium phosphate (TSP) decontamination fluid followed by a reagent grade methanol rinse and finally a triple distilled water rinse. The TSP decontamination fluid will be tap water with approximately 2.5 percent TSP dissolved (by weight). Sampling equipment will be triple rinsed with distilled water poured directly from the distilled water containers to eliminate acetone contamination. The pump and/or bailers will be laid out on clean plastic to air dry before reuse.

Surface Water Samples

Samples will be collected using a stainless steel laboratory beaker and transferred to the appropriate sample container. One set of samples collected for metals analysis will be filtered in the field. All sampling equipment will be decontaminated between sampling locations using the procedures outlined for the groundwater sampling equipment.

Surface Soil Samples

The initial surface soil samples, including replicate and field blank samples, will be obtained using a trailer-mounted drill rig and split-spoon sampler. Continuous split-spoon samples will be obtained to a depth of 4 feet. Samples will be visually described and scanned with an OVA and HNu. Sampling equipment will be decontaminated between sample locations using the procedures outlined for the groundwater sampling equipment.

Samples obtained from beneath the parking areas of the railroad's automobile storage facility may require coring and patching, to allow penetration by the split-spoon sampler and to prevent contamination of underlying soils with asphalt.

Sediment Samples

Sediment samples will be collected using a hand corer. If sediment properties make this impossible, samples may be collected using a scoop, ponar grab, or other suitable method. Sample locations will be flagged on one bank of the river. A sketch will be made of important river features at the sampling location such as distance from a permanent structure, bends in the river, shape of the channel, water depth, current, etc. Field personnel will start downstream and work upstream to avoid stirring up sediment that could contaminate unsampled areas.

Following analysis of the samples collected for the initial sediment screening (Task S1) an additional 16 confirmatory samples will be collected using the same procedures as mentioned above.

4.0 SAMPLE HANDLING AND ANALYSIS

4.1 PARAMETERS

Table D-1 summarizes the estimated number of samples, replicates, and field blanks to be collected during the Phase I

Table D-1
SUMMARY OF THE SAMPLING AND ANALYSIS PROGRAM AT MOSS AMERICAN

SAMPLE MATRIX	FIELD MEASUREMENTS	LABORATORY PARAMETERS [1]	QQQ ANALYTICAL LEVEL [2]	SAMPLES NO.	SAMPLES FREQ.	SAMPLES TOTAL	FIELD REPLICATES [3] NO.	FIELD REPLICATES [3] FREQ.	FIELD REPLICATES [3] TOTAL	FIELD BLANKS [3] NO.	FIELD BLANKS [3] FREQ.	FIELD BLANKS [3] TOTAL	MATRIX SPIKE NO.	MATRIX SPIKE FREQ.	MATRIX SPIKE TOTAL	MATRIX TOTAL [4]
GROUNDWATER																
	pH Specific Conductance Temperature Static Water Levels	VOC's consistent with RAS Protocol Unfiltered Samples	IV	23	1	23	2	1	2	2	1	2	2	1	2	27
		Acid, Base/Neutral Extract. RAS Protocol Unfiltered Samples	IV	23	1	23	2	1	2	2	1	2	2	1	2	27
		Pesticides/PCB's RAS Protocol Unfiltered Samples	IV	23	1	23	2	1	2	2	1	2	2	1	2	27
		Metals RAS Protocol Filtered Samples	IV	23	1	23	2	1	2	2	1	2	2	1	2	27
		Metals RAS Protocol Unfiltered Samples	IV	23	1	23	2	1	2	2	1	2	2	1	2	27
		Cyanide RAS Protocol Unfiltered Samples	IV	23	1	23	2	1	2	2	1	2	2	1	2	27
		Biochemical Oxygen Demand SAS (Std Method 507) Unfiltered Samples	V	23	1	23	2	1	2	2	1	2	2	1	2	27
		Chemical Oxygen Demand SAS (EPA 410.1) Unfiltered Samples	V	23	1	23	2	1	2	2	1	2	2	1	2	27
		Total Organic Carbon SAS (EPA 415.1) Unfiltered Samples	V	23	1	23	2	1	2	2	1	2	2	1	2	27
		Sulfate SAS (EPA 375.2 or 4) Unfiltered Samples	V	23	1	23	2	1	2	2	1	2	2	1	2	27
		Total Dissolved Solids SAS (Std Method 209B) Unfiltered Samples	V	23	1	23	2	1	2	2	1	2	2	1	2	27
		Total Suspended Solids SAS (EPA 160.2) Unfiltered Samples	V	23	1	23	2	1	2	2	1	2	2	1	2	27
		Alkalinity/Acidity SAS (EPA 310.1/EPA 305.1) Unfiltered Samples	V	23	1	23	2	1	2	2	1	2	2	1	2	27
		Total Phenol SAS (EPA 420.1 or Std Method 510 A & B)	V	23	1	23	2	1	2	2	1	2	2	1	2	27
SURFACE WATER																
	pH Specific Conductance Temperature	VOC s consistent with RAS Protocol Unfiltered Samples	IV	8	1	8	1	1	1	1	1	1	1	1	1	10
		Acid, Base/Neutral Extract. RAS Protocol Unfiltered Samples	IV	8	1	8	1	1	1	1	1	1	1	1	1	10
		Pesticides/PCB s RAS Protocol Unfiltered Samples	IV	8	1	8	1	1	1	1	1	1	1	1	1	10
		Metals RAS Protocol Filtered Samples	IV	8	1	8	1	1	1	1	1	1	1	1	1	10

Table D-1
SUMMARY OF THE SAMPLING AND ANALYSIS PROGRAM AT MOSS AMERICAN

SAMPLE MATRIX	FIELD MEASUREMENTS	LABORATORY PARAMETERS [1]	DQO ANALYTICAL LEVEL [2]	SAMPLES NO.	SAMPLES FREQ.	SAMPLES TOTAL	FIELD REPLICATES [3] NO.	FIELD REPLICATES [3] FREQ.	FIELD REPLICATES [3] TOTAL	FIELD BLANKS [3] NO.	FIELD BLANKS [3] FREQ.	FIELD BLANKS [3] TOTAL	MATRIX SPIKE NO.	MATRIX SPIKE FREQ.	MATRIX SPIKE TOTAL	MATRIX TOTAL [4]
SURFACE WATER (cont.)																
		Metals RAS Protocol Unfiltered Samples	IV	8	1	8	1	1	1	1	1	1	1	1	1	10
		Cyanide RAS Protocol Unfiltered Samples	IV	8	1	8	1	1	1	1	1	1	1	1	1	10
		Biochemical Oxygen Demand SAS (Std Method 507) Unfiltered Samples	V	8	1	8	1	1	1	1	1	1	1	1	1	10
		Chemical Oxygen Demand SAS (EPA 410.1) Unfiltered Samples	V	8	1	8	1	1	1	1	1	1	1	1	1	10
		Total Organic Carbon SAS (EPA 415.1) Unfiltered Samples	V	8	1	8	1	1	1	1	1	1	1	1	1	10
		Total Dissolved Solids SAS (Std Method 209B) Unfiltered Samples	V	8	1	8	1	1	1	1	1	1	1	1	1	10
		Total Suspended Solids SAS (EPA 160.2) Unfiltered Samples	V	8	1	8	1	1	1	1	1	1	1	1	1	10
		Alkalinity/Acidity SAS (EPA 310.1/EPA 305.1) Unfiltered Samples	V	8	1	8	1	1	1	1	1	1	1	1	1	10
		Total Phenol SAS (EPA 420.1 or Std Method 510 A & B)	V	8	1	8	1	1	1	1	1	1	1	1	1	10
SURFACE SOIL																
	HNu Screening	TOC screening SOP (CH2M HILL/CSL)	II	250	1	250	13	1	13	13	1	13	13	1	13	276
	OVA Screening															
		GC/FID Analysis SOP (CH2M HILL/MGM)	III	60	1	60	3	1	3	3	1	3	3	1	3	66
		VOC's consistent with RAS Protocol	IV	16	1	16	2	1	2	2	1	2	1	1	1	20
		Acid, Base/Neutral Extract. RAS Protocol	IV	16	1	16	2	1	2	2	1	2	1	1	1	20
		Pesticides/PCB's RAS Protocol	IV	16	1	16	2	1	2	2	1	2	1	1	1	20
		Metals RAS Protocol	IV	16	1	16	2	1	2	2	1	2	1	1	1	20
		Cyanide RAS Protocol	IV	16	1	16	2	1	2	2	1	2	1	1	1	20
		Carbon SAS (ASTM D-3178)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Hydrogen SAS (ASTM D-3178)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Sulfur SAS (ASTM D-3177-82)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Oxygen SAS (by difference)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Nitrogen SAS (ASTM [illegible])	V	16	1	16	2	1	2	2	1	2	1	1	1	20

Table D-1
SUMMARY OF THE SAMPLING AND ANALYSIS PROGRAM AT MOSS AMERICAN

SAMPLE MATRIX	FIELD MEASUREMENTS	LABORATORY PARAMETERS [1]	DQO ANALYTICAL LEVEL [2]	SAMPLES NO.	SAMPLES FREQ.	SAMPLES TOTAL	FIELD REPLICATES [3] NO.	FIELD REPLICATES FREQ.	FIELD REPLICATES TOTAL	FIELD BLANKS [3] NO.	FIELD BLANKS FREQ.	FIELD BLANKS TOTAL	MATRIX SPIKE NO.	MATRIX SPIKE FREQ.	MATRIX SPIKE TOTAL	MATRIX TOTAL [4]
SURFACE SOIL (cont.)		Moisture Content SAS (ASTM D-3173)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Ash Content SAS (ASTM D-3174)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Volatile Matter SAS (ASTM D-3175)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Fixed Carbon SAS (ASTM D-3172)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Total Organic Carbon SAS (Dohrmann)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Water Soluble Chlorides SAS (EPA 62-3.5)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Dioxin (several isomers) SAS Anal. Chem. 1980,52, 2045-2054)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Heating Value SAS (ASTM D-2015-77)	V	16	1	16	2	1	2	0	0	0	0	0	0	18
		Flash Point SAS (Test Method for Evaluating Solid Waste SW846)	V	16	1	16	2	1	2	0	0	0	0	0	0	18
		pH SAS	V	16	1	16	2	1	2	0	0	0	0	0	0	18
SEDIMENT	HNu Screening	TOC Screening SOP (CH2M HILL/CSL)	II	257	1	257	13	1	13	13	1	13	13	1	13	283
	OVA Screening	GC/FID Analysis SOP (CH2M HILL/MGM)	III	60	1	60	3	1	3	3	1	3	3	1	3	66
		VOC's consistent with RAS Protocol	IV	16	1	16	2	1	2	2	1	2	1	1	1	20
		Acid, Base/Neutral Extract. RAS Protocol	IV	16	1	16	2	1	2	2	1	2	1	1	1	20
		Pesticides/PCB's RAS Protocol	IV	16	1	16	2	1	2	2	1	2	1	1	1	20
		Metals RAS Protocol	IV	16	1	16	2	1	2	2	1	2	1	1	1	20
		Cyanide RAS Protocol	IV	16	1	16	2	1	2	2	1	2	1	1	1	20
		Carbon SAS (ASTM D-3178)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Hydrogen SAS (ASTM D-3178)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Sulfur SAS (ASTM D-3177-82)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Oxygen SAS (by difference)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Nitrogen SAS (ASTM 3179)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Moisture Content SAS (ASTM D-3173)	V	16	1	16	2	1	2	2	1	2	1	1	1	20

Table D-1
SUMMARY OF THE SAMPLING AND ANALYSIS PROGRAM AT MOSS AMERICAN

SAMPLE MATRIX	FIELD MEASUREMENTS	LABORATORY PARAMETERS [1]	QAO ANALYTICAL LEVEL [2]	SAMPLES NO.	SAMPLES FREQ.	SAMPLES TOTAL	FIELD REPLICATES [3] NO.	FIELD REPLICATES FREQ.	FIELD REPLICATES TOTAL	FIELD BLANKS [3] NO.	FIELD BLANKS FREQ.	FIELD BLANKS TOTAL	MATRIX SPIKE NO.	MATRIX SPIKE FREQ.	MATRIX SPIKE TOTAL	MATRIX TOTAL [4]
SEDIMENT (cont.)		Ash Content SAS (ASTM D-3174)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Volatile Matter SAS (ASTM D-3175)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Fixed Carbon SAS (ASTM D-3172)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Total Organic Carbon SAS (Dohrmann)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Water Soluble Chlorides SAS (EPA 62-3.5)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Dioxin (several isomers) SAS Anal. Chem. 1980,52, 2045-2054)	V	16	1	16	2	1	2	2	1	2	1	1	1	20
		Heating Value SAS (ASTM D-2015-77)	V	16	1	16	2	1	2	0	0	0	0	0	0	16
		Flash Point SAS (Test Method for Evaluating Solid Waste SW846)	V	16	1	16	2	1	2	0	0	0	0	0	0	18
		pH SAS	V	16	1	16	2	1	2	0	0	0	0	0	0	18
SUBSURFACE SOIL																
	HNu Screening	TOC Screening SOP (CH2M HILL/CSL)	II	130	1	130	5	1	5	5	1	5	5	1	5	140
	OVA Screening															
		VOC's consistent with RAS Protocol	IV	30	1	30	2	1	2	2	1	2	2	1	2	34
		Acid, Base/Neutral Extract. RAS Protocol	IV	30	1	30	2	1	2	2	1	2	2	1	2	34
		Pesticides/PCB's RAS Protocol	IV	30	1	30	2	1	2	2	1	2	2	1	2	34
		Metals RAS Protocol	IV	30	1	30	2	1	2	2	1	2	2	1	2	34
		Cyanide RAS Protocol	IV	30	1	30	2	1	2	2	1	2	2	1	2	34
		Carbon SAS (ASTM D-3178)	V	30	1	30	2	1	2	2	1	2	2	1	2	34
		Hydrogen SAS (ASTM D-3178)	V	30	1	30	2	1	2	2	1	2	2	1	2	34
		Sulfur SAS (ASTM D-3177-82)	V	30	1	30	2	1	2	2	1	2	2	1	2	34
		Oxygen SAS (by difference)	V	30	1	30	2	1	2	2	1	2	2	1	2	34
		Nitrogen SAS (ASTM 3179)	V	30	1	30	2	1	2	2	1	2	2	1	2	34
		Moisture Content SAS (ASTM D-3173)	V	30	1	30	2	1	2	2	1	2	2	1	2	34
		Ash Content SAS (ASTM D-3174)	V	30	1	30	2	1	2	2	1	2	2	1	2	34
		Volatile Matter SAS (ASTM D-3175)	V	30	1	30	2	1	2	2	1	2	2	1	2	34

Table D-1
SUMMARY OF THE SAMPLING AND ANALYSIS PROGRAM AT MOSS AMERICAN

SAMPLE MATRIX	FIELD MEASUREMENTS	LABORATORY PARAMETERS [1]	DQO ANALYTICAL LEVEL [2]	SAMPLES NO.	SAMPLES FREQ.	SAMPLES TOTAL	FIELD REPLICATES [3] NO.	FIELD REPLICATES FREQ.	FIELD REPLICATES TOTAL	FIELD BLANKS [3] NO.	FIELD BLANKS FREQ.	FIELD BLANKS TOTAL	MATRIX SPIKE NO.	MATRIX SPIKE FREQ.	MATRIX SPIKE TOTAL	MATRIX [4] TOTAL
SURFACE SOIL (cont.)		Fixed Carbon SAS (ASTM D-3172)	V	30	1	30	2	1	2	2	1	2	2	1	2	34
		Total Organic Carbon SAS (Dohrmann)	V	30	1	30	2	1	2	2	1	2	2	1	2	34
		Water Soluble Chlorides SAS (EPA 62-3.5)	V	30	1	30	2	1	2	2	1	2	2	1	2	34
		Dioxin (several isomers) SAS Anal. Chem. 1980,52, 2045-2054)	V	30	1	30	2	1	2	2	1	2	2	1	2	34
		Heating Value SAS (ASTM D-2015-77)	V	30	1	30	2	1	2	0	0	0	0	0	0	32
		Flash Point SAS (Test Method for Evaluating Solid Waste SW846)	V	30	1	30	2	1	2	0	0	0	0	0	0	32
		pH SAS	V	30	1	30	2	1	2	0	0	0	0	0	0	32
		Particle-Size Analysis SAS (ASTM D-422-63)	V	10	1	10	1	1	1	0	0	0	0	0	0	11
		Atterburg Limits SAS (ASTM D-4318-84)	V	10	1	10	1	1	1	0	0	0	0	0	0	11

Notes:

1
See Appendix B of the QAPP for a complete list of Routine Analytical Services (RAS) parameters.

2
Analytical levels are defined in "Data Quality Objectives for Remedial Response Activities, Volume I" EPA 540/G-87/003A.

3
This is an estimated number of field replicates and field blank samples. The actual number collected or prepared is a function of the number of samples collected per day. At a minimum one replicate and one blank will be prepared each day. In addition to the field blanks, one bottle blank will be prepared for the surface water VOC samples and one for the groundwater VOC samples.

4
Matrix spikes and matrix spike duplicates are not included in the matrix total number of samples. These numbers represent samples on which the laboratory must perform contract required QC analyses. Twice the normal sample volume must be collected for all aqueous volatile organic RAS samples and three times the normal sample volume must be collected for all extractable organic samples including pesticides and PCB's for samples on which the matrix spike and matrix spike duplicte analyses will be performed. Additional sample volume will not be necessary to perform the matrix spike analyses on the remaining aqueous SAS parameters or for the soil and sediment analyses.

Table D-2 (Page 1 of 3)
SAMPLE CONTAINERS, PRESERVATION, SHIPPING
AND PACKAGING REQUIREMENTS

Analysis	Container per Sample	Preservation	Holding Time	Shipping	Packaging
SOIL AND SEDIMENT					
Low Concentration (RAS)					
Acid, base/neutral, pesticide/PCB's extractables	One 8-oz. wide mouth glass jar. Fill 3/4 full.	None	10 days for extraction 40 days for analysis	Overnight carrier	In baggies and Foam Liner No. 3.
Volatiles	Two 120-ml wide mouth glass vials. Fill completely, no headspace.	None	10 days	Overnight carrier	In baggies and Foam Liner No. 3.
Metals Mercury Cyanide	One 8-oz. wide mouth glass jar. Fill 3/4 full.	None	6 months 28 days 14 days	Overnight carrier	In baggies and Foam Liner No. 3.
Medium Concentration (RAS)					
Acid, base/neutral, pesticides/PCB's extractables	One 8-oz. wide mouth glass jar. Fill 3/4 full.	None	10 days	Overnight carrier with attached shipper's certificate for restricted articles.	In cans and surrounded by vermiculite.
Volatiles	Two 120-ml wide mouth glass jars. Fill completely, no headspace.	None	10 days	Overnight carrier with attached shipper's certificate for restricted articles.	In cans and surrounded by vermiculite.
Metals Mercury Cyanide	One 8-oz. wide mouth glass jar.	None	6 months 28 days 14 days	Overnight carrier with attached shipper's certificate for restricted articles.	In cans and surrounded by vermiculite.
High Concentration (SAS)					
All organic parameters	One 8-oz. wide mouth glass jar. Fill 1/2 to 3/4 full.	None	Not Established	Overnight carrier with attached shipper's certificate for restricted articles.	In cans and surrounded by vermiculite.
Metals and Cyanide	One 8-oz. wide mouth glass jar.	None	Not Established	Overnight carrier with attached shipper's certificate for restricted articles.	In cans and surrounded by vermiculite.
Special Analytical Services (SAS)					
GC/FID screening	One 4-oz. glass jar.	None	Not Established	Overnight carrier	In baggies and surrounded by vermiculite.
Proximate and ultimate analysis, heating value, flash point	Two 32-oz. glass jars.	None	Not Established	Overnight carrier	In baggies and surrounded by vermiculite.
Dioxin	One 8-oz. glass jar.	None	Not Established	Overnight carrier	In baggies and surrounded by vermiculite.

Analysis	Container per Sample	Preservation	Holding Time	Shipping	Packaging
GROUNDWATER AND SURFACE WATER					
Low Concentration (RAS)					
Acid and base/neutral extractables, pesticides/ PCB's	Two 1/2-gallon glass amber bottles (teflon-lined caps). Fill bottle to neck.	Must be iced to 4°C	5 days for extraction 40 days for analysis	Overnight carrier	No. 1 foam liner or vermiculite.
Volatiles	Two 40-ml volatile organic analyses/VOA vials. Fill completely, no air bubbles.	Must be iced to 4°C	7 days	Overnight carrier	In baggies and surrounded by vermiculite.
Metals, Filtered Mercury, Filtered	One 1-liter high density polyethylene bottle. Fill to shoulder of bottle.	Filtered samples HNO_3 to pH <2	6 months 28 days	Overnight carrier	No. 2 foam liner or vermiculite.
Metals, Total Mercury, Total	One 1-liter high density polyethylene bottle. Fill to shoulder of bottle.	Unfiltered samples HNO_3 to pH <2	6 months 28 days	Overnight carrier	No. 2 foam liner or vermiculite.
Cyanide	One 1-liter high density polyethylene bottle. Fill to shoulder of bottle.	Unfiltered sample	14 days	Overnight carrier	No. 2 foam liner or vermiculite.
Medium Concentration (RAS)					
Acid and base/neutral extractables, Pesticides/ PCB's	Four 32-oz. wide mouth glass jars. Fill 3/4 full.	None	5 days for extraction 40 days for analysis	Overnight carrier	In cans and surrounded by vermiculite.
Volatiles	Two 40-ml volatile organic analyses (VOA) vials. Fill completely, no headspace.	None	7 days	Overnight carrier with attached shipper's certificate for restricted articles.	In cans and surrounded by vermiculite.
Metals, Filtered	One 16-oz. wide mouth glass jar. Fill 3/4 full.	None	6 months	Overnight carrier with attached shipper's certificate for restricted articles.	In cans and surrounded by vermiculite.
Mercury, Filtered			28 days		
Metals, Total	One 16-oz. wide mouth glass jar. Fill 3/4 full.	None	6 months	Overnight carrier with attached shipper's certificate for restricted articles.	In cans and surrounded by vermiculite.
Mercury, Total					
Cyanide	One 16-oz. wide mouth glass jar. Fill 3/4 full.	None	14 days	Overnight carrier with attached shipper's certificate for restricted articles.	In cans and surrounded by vermiculite.

Analysis	Container per Sample	Preservation	Holding Time	Shipping	Packaging
High Concentration (SAS)					
All organic parameters	One 8-oz. wide mouth glass jar. Fill 1/2 to 3/4 full.	None	Not Established	Overnight carrier with attached shipper's certification for restricted articles.	In cans and surrounded by vermiculite.
Metals and Cyanide	One 8-oz. wide mouth glass jar. Fill 1/2 to 3/4 full.	None	Not Established	Overnight carrier with attached shipper's certification for restricted articles.	In cans and surrounded by vermiculite.
Special Analytical Service (SAS)					
BOD, TSS, TDS, alkalinity/ acidity, sulfates	Two 1-liter polyethylene bottle. Fill to shoulder of bottle.	Cool to 4°C	48 hours for BOD, 7 days for TSS and TDS, 14 days for alkalinity/acidity, and 28 days for sulfates	Overnight carrier	No. 2 foam liner or vermiculite.
TOC, COD, ammonia	One 1-liter polyethylene bottle. Fill to shoulder of bottle.	H_2SO_4 to pH <2 Cool to 4°C	28 days	Overnight carrier	No. 2 foam liner or vermiculite.
Phenol	One 1-liter polyethylene bottle. Fill to shoulder of bottle.	10-ml of ($CaSO_4$ + H_3PO_4) preservative	7 days	Overnight carrier	No. 2 foam liner or vermiculite.

GLT595/37

RI; the analyses to be conducted on the various samples collected; and the analytical level as defined in "Data Quality Objectives for Remedial Response Activities" (EPA 540/G-871003A). The actual number of field replicate samples collected and field blanks prepared is a function of the number of samples collected per day. At a minimum, one replicate and one blank will be prepared each day. Matrix spike (and matrix spike duplicate) samples represent the number of samples for which the laboratory must perform contract required QC analysis. Additional sample volume will not be necessary to perform matrix spike analysis on soil and sediment samples for Routine Analytical Services (RAS) and Special Analytical Services (SAS) parameters performed by the Contract Laboratory Program (CLP). However, two times the normal sample volume for aqueous volatile organic samples and three times the normal sample volume for aqueous acid and base/neutral extractables, pesticides, and PCB's, must be collected for samples on which the matrix spike and matrix spike duplicate analyses will be performed. The number of matrix spike samples collected will be at least 1 per 20 samples collected. Additional sample volume will not be necessary to perform matrix spike analyses on the aqueous SAS parameters. Because of the organization of the CLP it is not possible to prepare a VOA trip blank. To acquire the same information a bottle blank will be prepared for VOA's before going into the field. The water will be HPLC grade water which has passed QC requirement by the manufacturer. One bottle blank will be prepared for the surface water VOC samples and one for the groundwater VOC samples.

The CLP RAS will be used to analyze samples of low or medium levels of concentration. Determination of the level of concentration will be made in the field at the time of sampling based on GC/FID analyses, HNu or OVA readings, and visual inspection of the samples. As a guideline, low level samples would possibly include those collected offsite, around the perimeter of the site, and in unsuspected former process or disposal areas. Medium levels may include samples collected onsite in areas of moderate dilution of the contaminants. Medium level samples are estimated to contain concentrations of individual compounds up to 15 percent. Samples suspected to contain greater than 15 percent concentration of any individual chemical contaminant are considered High Hazard (HH) samples and require analyses based on SAS protocols.

Groundwater Samples

Approximately 27 groundwater samples will be collected. This includes one sample from each of the 23 wells, plus approximately 2 replicate samples, and 2 field blanks. The

number of replicates field blanks prepared, however, is a function of the number of samples collected per day. Filtered and unfiltered replicates will be taken at 2 of the wells showing the most significant amount of organic vapors as indicated by the HNu. Field blanks will consist of deionized water. The blank and replicate samples will be filtered and preserved in the same manner as the other groundwater samples. The field blank will be bottled using the sampling equipment as a test of the cleaning procedures.

Portions of the 27 groundwater samples collected will be sent to the CLP for RAS analyses of organic (acid and base/neutral extractables, volatiles, PCB's/pesticides) parameters and inorganic (metals and cyanide) parameters. Filtered and unfiltered samples will be sent for metals analysis. In addition, portions of these 27 groundwater samples will be analyzed by the CLP according to SAS requests for: BOD_5, TOC, COD, sulfates, TDS, TSS, alkalinity/acidity, total phenols. The SAS protocols for these analyses are given in Appendix G. Field measurements for groundwater samples to be reported in the logbook include: pH, temperature, and specific conductance (along with static water level measurements).

Surface Water Samples

Eight surface water samples, plus one replicate and one field blank, will be collected and submitted to the CLP. These samples will be analyzed for the same RAS and SAS parameters as listed for the groundwater samples.

Initial Screening of Surface Soil and Sediment Samples

The initial screening of 250 surface soil samples and 257 sediment samples will be performed using total carbon (TC) analyses according to the procedures developed by the CH2M HILL (Appendix B). The TC data will be used for the purpose of defining the zone(s) of contamination. The U.S. EPA Close Support Laboratory (CSL) will perform the TOC analysis at the Moss-American site. The purpose of using the mobile laboratory for in-field TC screening is to expedite the analyses. Proposed surface soil and sediment sample locations may be modified based on the results of the screening. CH2M HILL will be responsible for the management and operation of the CSL.

An estimated 12 replicate soil samples and 12 field blanks will also be collected during the initial screening. The actual number of field blanks prepared and replicates collected, will be a function of the number of samples collected

per day. At a minimum, one field blank will be prepared each day. The field blank will consist of commercial clean sand. Similarly, an estimated 12 sediment replicate samples and 12 sediment field blanks will be collected.

The unanalyzed portion samples obtained during the field activities will be retained onsite for further analysis using more sophisticated procedures. The unused portions of the samples will be containerized, labeled, and stored in a refrigerator located in a secure area of the site.

Following evaluation of the TC results, a total of 120 surface soil and sediment samples will be shipped to the CH2M HILL Montgomery laboratory for gas chromatography with flame ionization detection (GC/FID) analysis according to the statement of procedures (SOP) given in Appendix C. The GC/FID data will be used for the purpose of screening samples or analytically looking for compounds that are indicators of contamination at the Moss-American site. The GC/FID screening results will yield relative concentrations to be used in determining samples to be selected for qualitative analyses by EPA CLP protocols.

Confirmatory Surface Soil Samples

Following the collection and analysis of the initial surface soil samples, 16 confirmatory surface soil samples (plus replicate samples and field blanks) will be selected and submitted to the CLP. Six of these samples will be from noncontaminated or background sample locations, based on GC/FID analysis. The other 10 samples will be collected from areas representative of the most contaminated soil. Depending on the GC/FID results, these 10 samples may be handled as high hazard samples requiring analysis using SAS protocols instead of RAS protocols. According to the User's Guide to the CLP, high hazard samples are suspected to contain greater than 15 percent concentration of any individual chemical constituent. The following parameters will be analyzed using RAS (possibly HH-SAS) analyses: volatile organic compounds, acid and base/neutral extractables, pesticides/PCB's, metals, and cyanide. The field blanks will be made from commercial clean sand. Blanks of any kind do not apply to the heating value, flash point or pH.

A portion of these 16 samples will also be analyzed according to the SAS protocols given in Appendixes G & H for the following parameters: total organic carbon (TOC), moisture content, ash content, volatile matter, fixed carbon, flash point, pH, dioxin (all isomers), carbon, hydrogen, sulfur, nitrogen, oxygen and heating value. Field measurements for

all soil samples to be reported in the logbook include the HNu and OVA readings.

Confirmatory Sediment Samples

Similarly, following the initial screening of sediment samples using the GS/FID, 16 confirmatory sediment samples (plus field blanks and replicate samples) will be submitted to the CLP for analysis of the same parameters as defined for the surface soil samples. The field blanks will be prepared from commercial clean sand.

Subsurface Soil Samples

During the installation of the monitoring wells, continuous split-spoon samples will be collected from the ground surface to a depth of 20 feet (or the bottom of the well). Split-spoon samples will be obtained at 5-foot intervals thereafter. Approximately 130 subsurface soil samples will be screened using the TC method and the CSL. The unanalyzed portions of these samples will be containerized, labeled, and stored onsite for further analysis. Approximately 30 subsurface soil samples will be selected for detailed analysis using the CLP following the evaluation of the TC results.

The thirty subsurface samples will be analyzed by the CLP RAS for: VOC's, acid and base/neutral extractables, pesticides/PCB's, metals, and cyanide. These samples may be analyzed as high hazard samples depending on visual inspection, HNu and OVA readings, and TC screening results. A portion of these 30 samples (plus replicate samples and field blanks) will also be analyzed using the SAS protocols for TOC, moisture content, pH, heating value, water soluble chlorides, ash content, flash point, fixed carbon, volatile matter, carbon, hydrogen, sulfur, nitrogen, oxygen and dioxin (all isomers).

In addition, 10 subsurface samples collected (plus one replicate and one field blank) will be selected for hydrogeological testing of soil samples. This will include particle-size analysis and Atterberg limits according the the ASTM methods described in Appendix H. Either the CLP or a soil testing laboratory will be chosen to perform these analyses depending on CLP availability and the relative concentrations measured during the TOC screening. Field blanks will be prepared from commercial clean sand. Blanks of any kind do not apply to the pH, heating value, flash point, particle size or Atterberg limits.

4.2 SAMPLE PREPARATION, PACKAGING, AND SHIPPING

Table D-2 summarizes the sample containerization and preservation requirements based on the User's Guide to the CLP (1984) and 40 CFR Part 136 (Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act). This table also summarizes the sample packaging and shipping procedures based on EPA specifications, as well as Department of Transportation (DOT) regulations (40 CFR Part 172). The procedures vary depending on the sample concentration (low, medium, or high) and the matrix (water or soil/sediment).

All samples will be shipped within 48 hours of collection or before 50 percent of the holding time has elapsed. Shipping containers must be insulated, durable, and watertight. Bagged samples (to prevent vermiculite contamination of samples) are to be cushioned within the shipping container with vermiculite packing material (Zonolite). All containers regardless of size/type will be placed inside sealed plastic bag before packing in vermiculite/zonolite.

Preparation of Groundwater Samples for Inorganic Analysis

The groundwater samples sent to the CLP for inorganic analyses (metals and cyanide) shall include unfiltered and filtered samples. Filtering of the samples for metals analysis will occur in the field as soon as possible after collection. The sample will be filtered through 0.45 micron filter paper using a pressure filtration device. All filtered portions of the sample will be preserved, by using nitric acid to achieve a pH of less than 2, immediately after filtration. The unfiltered samples for metals analysis shall also be preserved using nitric acid to a pH of less than 2. The portion of the sample collected for cyanide analyses will not be filtered, and will be preserved using NaOH to achieve a pH of 12 or greater.

4.3 SCHEDULING

The schedule for the soil, sediment, groundwater, and surface water sampling along with the other Phase I RI Tasks is given in the Work Plan for the Moss-American site. Site and river surveying and mapping activities will be performed to mark sampling locations. Surface water samples will be collected prior to sediment samples to avoid collecting contaminants possibly released by disturbing the sediment. Two sample crews are anticipated for concurrent surface soil and sediment sampling. Groundwater sampling will begin once the drilling activities have been completed.

4.4 SAMPLE DOCUMENTATION

The CLP requires the use of several documentation forms to identify and track each sample from the point of collection through final data reporting. Standard paperwork including sample tags, traffic reports, chain-of-custody forms, and custody seals used for CLP sample tracking and records will be filled out according to the instructions given in Attachment D-1.

All pertinent information about the samples will be logged in the site logbook maintained by the Team Leader. This information will include sample time, location, tag numbers, designation, and sampling personnel. New readings, weather conditions, and field modifications or decisions will also be recorded. The logbook will be filled in ink. Photographs will be taken at sampling locations to clarify the written descriptions in the logbooks. Photograph numbers with the time, date, location, and task description will also be noted in the logbook.

5.0 INVESTIGATION WASTE MANAGEMENT

During drilling activities, cuttings and wash water that give significant HNu or OVA readings and cuttings from the fill material will be retained in Department of Transportation (DOT) approved 55-gallon drums. The full 55-gallon drums will be labeled and stored onsite in a secured area (fenced) for later disposal, if deemed necessary by EPA. All protective clothing and sampling-related wastes will also be disposed of in DOT approved 55-gallon drums.

Appendix E

E-1: INSTRUCTIONS FOR FILLING OUT SAMPLE DOCUMENTATION
E-2: SAMPLE CUSTODY PROCEDURES

Appendix E-1
INSTRUCTIONS FOR FILLING OUT SAMPLE DOCUMENTATION

INSTRUCTIONS FOR FILLING OUT SAMPLE DOCUMENTATION

All samples collected at Superfund sites for laboratory analysis must follow established documentation protocol. Adherence to this protocol provides a network of valuable information documenting sample identification and tracking as well as chain-of-custody.

General Documentation Procedures

Organization and concentration are the keys to completing the required documents efficiently and without error. Make certain that a suitable work area has been set aside with ample table and floor space available for the processing of forms and the packaging of samples. This is especially important for large projects.

Forms, tags, etc. can be filled out in any order; however, past experience has shown that this paperwork can be completed most efficiently and accurately if the sample identification matrix (Figure 1) is completed before or in conjunction with the completion of the rest of the documentation.

Subsequent sections discuss the proper completion of each document. Use these pages as a reference while following this suggested plan of attack:

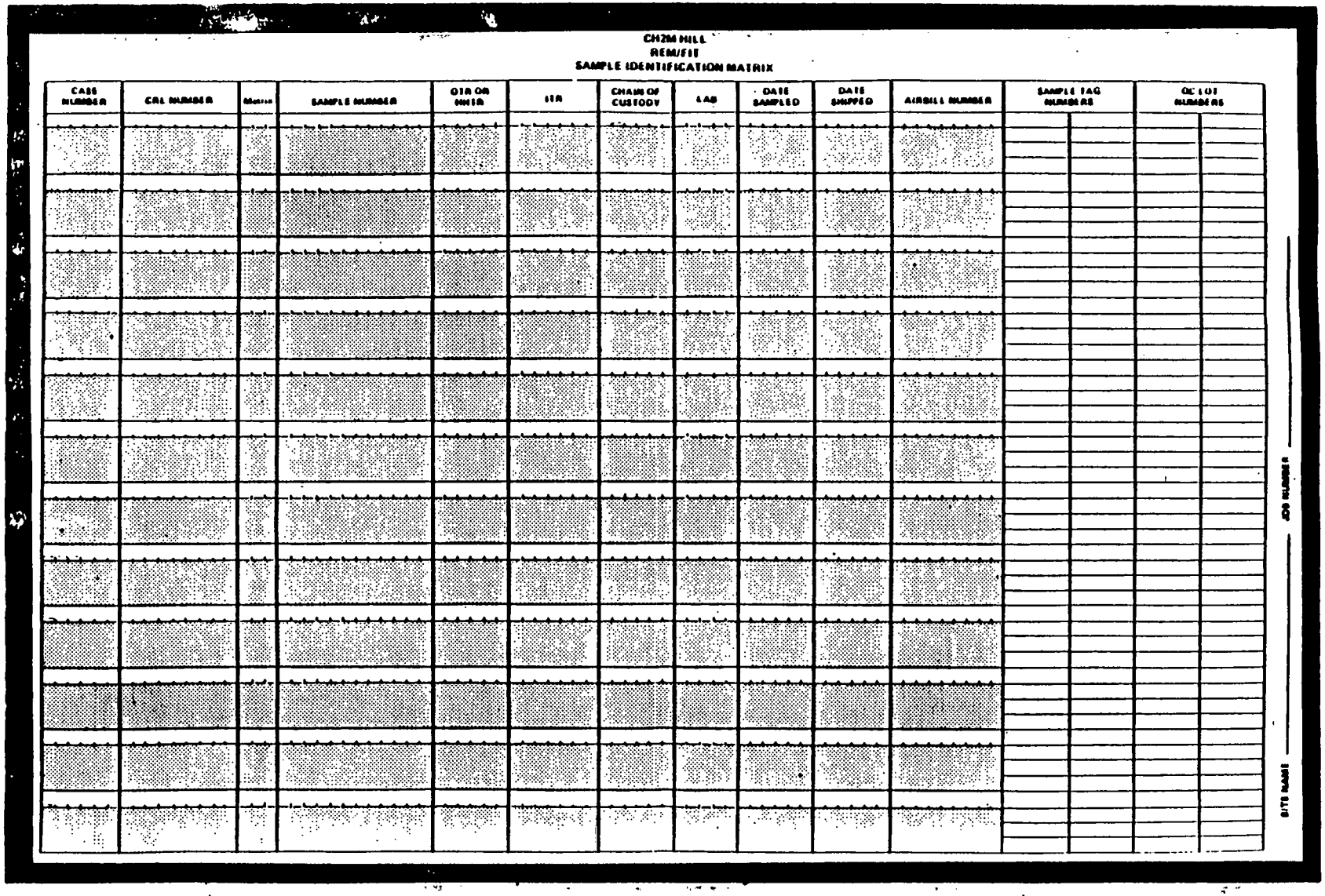
1. Make or obtain a list of the samples to be packaged and shipped on the same day and the laboratories to be used.

2. Enter the case number, CRL number, matrix, sample numbers, laboratory, date sampled, and date shipped for each sample on the matrix.

   NOTE: If portions of a given sample are to be shipped to different laboratories (for organic and inorganic analysis for instance), two entry lines will be required for that sample number to accommodate the chain-of-custody record, airbill, and traffic report numbers corresponding to each portion of the sample.

3. Obtain the QC lost numbers of the prelabled containers for each sample and enter these on the matrix.

4. Determine the number of shipping containers (coolers) required to accommodate the day's shipment. This is based on the number of samples to be shipped, the number of containers per sample, the number of sample containers



NOTE: For purposes of illustration this form has been reproduced at 50% of original size.

FIGURE 1

that will fit in each cooler, and the number of laboratories to be used.

NOTE: A group of containers for a single sample should not be split between coolers except when one portion of the sample is to be sent to one laboratory for one type of analysis and the other portion is to be sent to another laboratory for another type of analysis.

5. Complete an airbill for each laboratory address. (Note: Several coolers may be shipped to the same address under one airbill.) Shipment of medium and high concentration samples requires the use of a special airbill, including a shipper's certification for restricted articles (see Figure 12 for example).

6. Enter the airbill numbers on the matrix.

7. Assign a chain-of-custody record to each cooler and determine which sample containers will be shipped in each.

   NOTE: More than one chain-of-custody record may be needed to accommodate the number of samples to be shipped in one cooler.

8. Assign chain-of-custody numbers to each sample by entering these numbers on the matrix. (Reminder: Portions of samples for organic and inorganic analysis will usually be sent to separate laboratories. Use one line on the matrix for the organics portion information and another line for the inorganics portion information.)

9. If the samples are being shipped under a routine analytical service (RAS), determine the number of organics and/or inorganics traffic reports that will be needed. If the samples are high concentration, determine the number of high hazard traffic reports that will be needed.

10. Assign traffic report numbers to each sample and enter these numbers on the matrix.

11. Assign tag numbers to each sample container for each sample and enter these numbers on the matrix.

12. Complete traffic reports (of SAS packing lists or CRL basic data sheets) based on the information provided on the matrix.

13. Complete sample tags based on the information provided on the matrix and the parameters of analysis. Place tags in groups by sample number.

14. Complete chain-of-custody records based on the information provided on the matrix.

15. Assign two custody seals to each cooler. Enter the serial numbers of the seals in the "REMARKS" section of each chain-of-custody form and temporarily clip seals to the form.

16. Group all the paperwork associated with each cooler in a separate clip.

17. Obtain full signatures of the STL and initials of significant field team members (including yourself) on the sample tags and at the top of the chain-of-custody forms.

18. Prepare to package samples for shipment.

Following are step-by-step instructions for completing each form. The sample identification code to be used is the sample number as described in Appendix A. Other items should be self-evident from the instructions.

Sample Identification Matrix (Figure 1)

1. Indicate site name.

2. Indicate project number.

3. Enter the case number.

4. Enter the CRL number.

5. Specify the sample matrix using the two- or three digit codes listed below followed by the letter (L, M, or H) to indicate low, medium, or high concentrations:

   - SF--Surface Soil
   - SB--Subsurface Soil
   - SWO--Surface Water, Onsite
   - SWC--Surface Water, Creek
   - SDO--Sediment, Onsite
   - SDC--Sediment, Creek
   - GW--Groundwater

6. Indicate the sample number.

7. Enter the inorganics traffic report number.

8. Enter the organics traffic report number.

9. Indicate the chain-of-custody report number.

10. Indicate the laboratory to be doing the analysis.

11. Enter the date the sample was taken: month, day, year (no hyphen or slash, e.g, 051284).

12. Enter the shipping date.

13. Enter the airbill number of the shipment.

14. List sample tag numbers corresponding to sample containers shipped under the traffic report number listed in either box 7 or 8.

15. List the QC lot numbers of the containers matching the tag numbers listed in Item 14.

Note: Date recorded on this form must be suitable for computer entry. Each entry must be left justified and must not exceed the number of digits allowed in each section. If portions of samples are to be sent to more than one laboratory for analysis, allow an entire line for each laboratory to accommodate for the additional traffic report, chain-of-custody, and airbill numbers.

Sample Tag (Figure 2)

1. Enter the first six digits of the CRL sample identification.

2. Enter the last three digits of the CRL identification code.

3. Enter date of sampling.

4. Enter time of sampling (military time only).

5. Specify "grab" or "composite" sample with an "X."

6. Insert sample identification code.

7. Obtain signature of sample team leader.

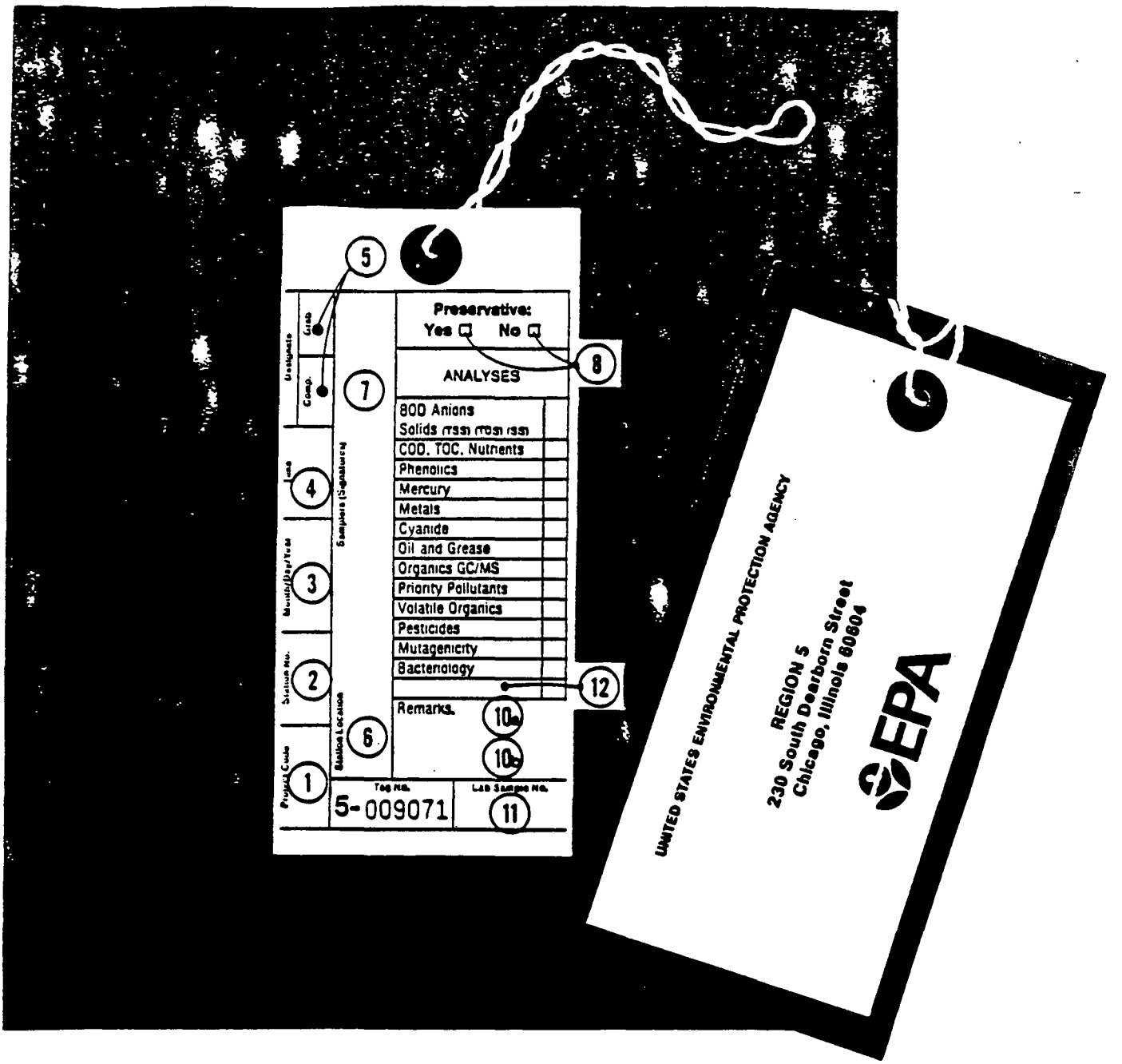
8. Indicate presence of preservative with an "X."

9. Specify all parameters for analysis with an "X" for each one.

10a. Indicate traffic report type and serial number (e.g., ITR number: MS 1534).

10b. Indicate case number (e.g., CASE #: 1234).

11. Leave BLANK (for laboratory use only).



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 2

12. Enter any desired analyses not listed on menu provided (e.g., PCB's, ammonia, sulfide, etc.) and mark box with an "X."

Inorganic Traffic Report (Figure 3)

1. Insert assigned laboratory case number.

2a. Insert CRL sample identification number.

2b. Insert sample number.

3. Insert EPA region number (e.g., V).

4. Insert sample team leader's name.

5. Insert sample team leader's office telephone number (do not use field office telephone number).

6. Insert date sample was taken.

7. Indicate sample description with an "X."

8. Insert corresponding organic traffic report number for the sample (if any).

9. Specify sample concentration with an "X."

10. Indicate sample matrix with an "X."

11. Insert "Federal Express" (or other approved carrier).

12. Indicate date of shipment.

13. Indicate airbill number corresponding with the sample shipment.

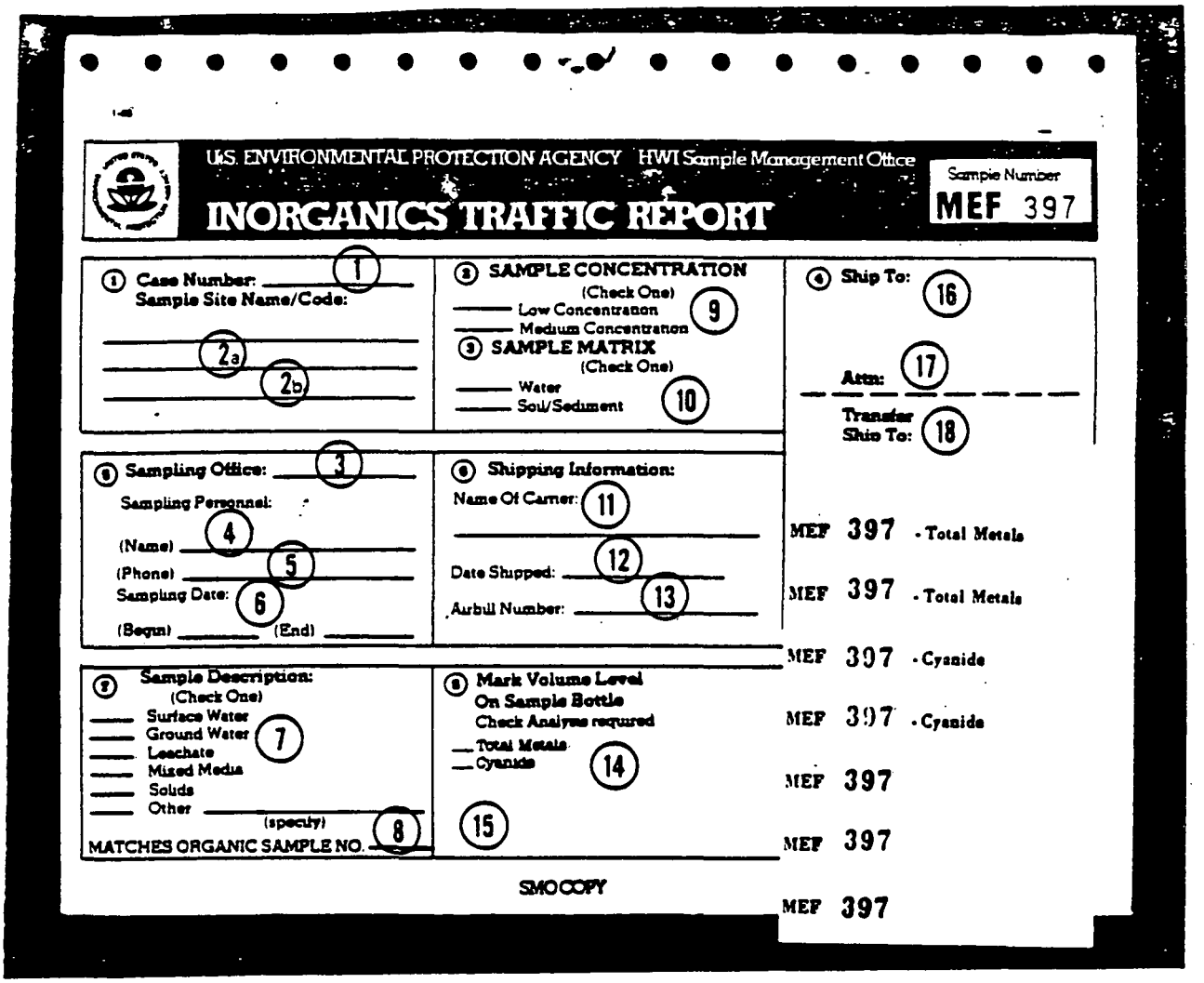
14. Check required analyses: Tasks 1 and 2 (metals) and/or Task 3 (cyanide only, ammonia and sulfide are no longer RAS, although some older traffic reports may still list them).

15. Insert the phrase "QC lot number:" and indicate the quality control lot number(s) of the container(s).

16. Insert laboratory name and address.

17. Indicate name of laboratory contact.

18. Leave BLANK (for laboratory use only).



U.S. ENVIRONMENTAL PROTECTION AGENCY HWI Sample Management Office

INORGANICS TRAFFIC REPORT

Sample Number: MEF 397

(1) Case Number: (1)
Sample Site Name/Code:
(2a)
(2b)

(2) SAMPLE CONCENTRATION (Check One) (9)
___ Low Concentration
___ Medium Concentration

(3) SAMPLE MATRIX (Check One) (10)
___ Water
___ Soil/Sediment

(4) Ship To: (16)
Attn: (17)
Transfer Ship To: (18)

(5) Sampling Office: (3)
Sampling Personnel:
(Name) (4)
(Phone) (5)
Sampling Date: (6)
(Begin) (End)

(6) Shipping Information:
Name Of Carrier: (11)
Date Shipped: (12)
Airbill Number: (13)

(7) Sample Description: (Check One)
___ Surface Water
___ Ground Water (7)
___ Leachate
___ Mixed Media
___ Solids
___ Other (specify)
MATCHES ORGANIC SAMPLE NO. (8)

(8) Mark Volume Level On Sample Bottle
Check Analyses required
___ Total Metals
___ Cyanide (14)
(15)

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MEF 397 - Total Metals
MEF 397 - Total Metals
MEF 397 - Cyanide
MEF 397 - Cyanide
MEF 397
MEF 397
MEF 397

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FIGURE 3

Organic Traffic Report (Figure 4)

1. Insert assigned laboratory case number.

2a. Insert CRL sample identification number.

2b. Insert sample number.

3. Insert EPA region number (e.g., V).

4. Insert sample team leader's name.

5. Insert sample team leader's office telephone number (do not use field office telephone number).

6. Insert date sample was taken.

7. Indicate "Federal Express" (or other approved carrier).

8. Indicate date of shipment.

9. Indicate airbill number corresponding to sample shipment.

10. Specify sample description with an "X."

11. Insert the phrase "QC lot number:" and indicate the quality control lot number(s) of the container(s).

12. Insert the phrase "matches IRT number:" and indicate the corresponding inorganics traffic report for the sample (if any).

13. Specify the sample concentration with an "X."

14. Indicate the sample matrix with an "X."

15. Indicate the number of sample containers shipped.

16. Insert an estimated sample volume in appropriate box.

17. Insert laboratory name and address.

18. Indicate name of laboratory contact.

19. Leave BLANK.

High Hazard Traffic Report (Figure 5)

1. Insert assigned laboratory case number.

2a. Insert CRL sample identification number.

U.S. ENVIRONMENTAL PROTECTION AGENCY HWI Sample Management Office

ORGANICS TRAFFIC REPORT

Sample Number: EE 242

① Case Number: (1)

Sample Site Name/Code: (2a) (2b)

② SAMPLE CONCENTRATION (Check One) (13)
___ Low Concentration
___ Medium Concentration

③ SAMPLE MATRIX (Check One) (14)
___ Water
___ Soil/Sediment

④ Ship To: (17)

Attn: (18)

Transfer Ship To: (19)

⑤ Regional Office: (3)

Sampling Personnel: (4)

(Name) (5)

(Phone)

Sampling Date: (6)

(Begin) (End)

⑥ For each sample collected specify num of containers used and mark volume le on each bottle.

	Number of Containers	Approx. Total Vo
Water (Extractable)		
Water (VOA)		
Soil/Sediment (Extractable)		
Soil/Sediment (VOA)	(15)	(16)
Other		

EE 242	· Water (Extractable)
EE 242	· Water (Extractable)
EE 242	· Water (Extractable)
EE 242	· Water (Extractable)
EE 242	· Water (VOA)
EE 242	· Water (VOA)
EE 242	· Soil/Sediment (Extractable)
EE 242	· Soil/Sediment (Extractable)
EE 242	· Soil/Sediment (VOA)
EE 242	· Soil/Sediment (VOA)

⑦ Shipping Information (7)

Name of Carrier (8)

Date Shipped: (9)

Airbill Number:

⑧ Sample Description (10)

___ Surface Water ___ Mixed Media
___ Ground Water ___ Solids
___ Leachate ___ Other (specify) _____

⑨ Sampl (20)

⑩ Special Handling Instructions:
(e.g., safety precautions, hazardous nature)
(11)
(12)

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FIGURE 4

U.S. ENVIRONMENTAL PROTECTION AGENCY CLP Sample Management Office

HIGH HAZARD TRAFFIC REPORT

Sample Number E 6361

FIELD SAMPLE RECORD

① Case Number: (1)

Sample Site Name/Code:
(2a)
(2b)

② Field Sample Description: (11)
- __ Drum
- __ Aqueous Liquid
- __ Sludge
- __ Solid
- __ Oil
- __ Other

③ Ship To: (16)

Attn: (17)

④ Sampling Office: (3)

Sampling Personnel: (4)

(name) (5)

(phone)

Sampling Date: (6)

(begin) (end)

⑤ Known or Suspected Hazards: (12)

⑥ Sample Location: (18)

⑦ Preparations Requested: (13)
(check below)

Sample Volume:
- __ Organics
- __ Volatile Organics
- __ Base Neutral Acid, TCDD (14)
- __ Pesticides PCB

- __ Inorganics
- __ Total Metals (15)
- __ Total Mercury
- __ Strong Acid Anions

⑧ Shipping Information:

(7) (name of carrier)

(8) (date shipped)

(9) (airbill number)

⑨ Special Handling Instructions:

(10)

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E 6361

E 6361

E 6361

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E 6361

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FIGURE 5

2b. Insert sample number.

3. Insert EPA region number (e.g., V).

4. Insert sample team leader's name.

5. Insert sample team leader's office telephone number (do not use field office telephone number).

6. Insert date sample was taken.

7. Indicate "Federal Express" (or other approved carrier).

8. Indicate date of shipment.

9. Indicate airbill number corresponding to sample shipment.

10. Insert the phrase "QC lot number:" and indicate the quality control lot number(s) of the container(s).

11. Indicate sample descriptions with an "X."

12. List known or suspected hazards.

13. Indicate approximate volume of sample.

14. Specify desired organic parameters to be analyzed for.

15. Specify desired inorganic parameters to be analyzed for (strong acid anions include Cl, SO_4, NO_3, F).

16. Insert laboratory name and address.

17. Indicate name of laboratory contact.

18. Leave BLANK (or make reference notes for future use).

SAS Packing List (Figure 6)

1. Insert assigned SAS case number.

2. Insert EPA region number (e.g., V).

3. Insert sample team leader's name.

4. Insert sample team leader's office telephone number (do not use field office telephone number).

5. Insert date sample was taken.

6. Indicate date of shipment.

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number (1)

SPECIAL ANALYTICAL SERVICE
PACKING LIST

Sampling Office: (2)	Sampling Date(s): (5)	Ship To: (8)	For Lab Use Only
Sampling Contact: (3) (name) (4) (phone)	Date Shipped: (6)		Date Samples Rec'd:
	Site Name/Code: (7)	Attn: (9)	Received By:

	Sample Numbers (10)	Sample Description i.e., Analysis, Matrix, Concentration (11)	Sample Condition on Receipt at Lab (12)
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			
19.			
20.			
			For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 6

7. Insert site name.

8. Insert laboratory name and address.

9. Indicate name of laboratory contact.

10. List SAS sample numbers, which should include the SAS number.

11. Specify sample matrix, concentration, tag number, and analysis to be performed (e.g., low concentration soil sample for PCB analysis, tag number 5-48246).

12. Leave BLANK (for laboratory use only).

Chain-of-Custody Form (Figure 7)

1. Enter first six digits of the CRL sample identification code.

2. Enter site name and CH2M HILL project number.

3. Obtain fill signature of sample team leader and signed initials of active team members (including paperwork person).

4. Enter last three digits of the CRL sample identification code.

5. List sampling dates for all samples.

6. List sampling times for all samples.

7. Indicate "grab" or "composite" sample with an "X."

8. List sample numbers.

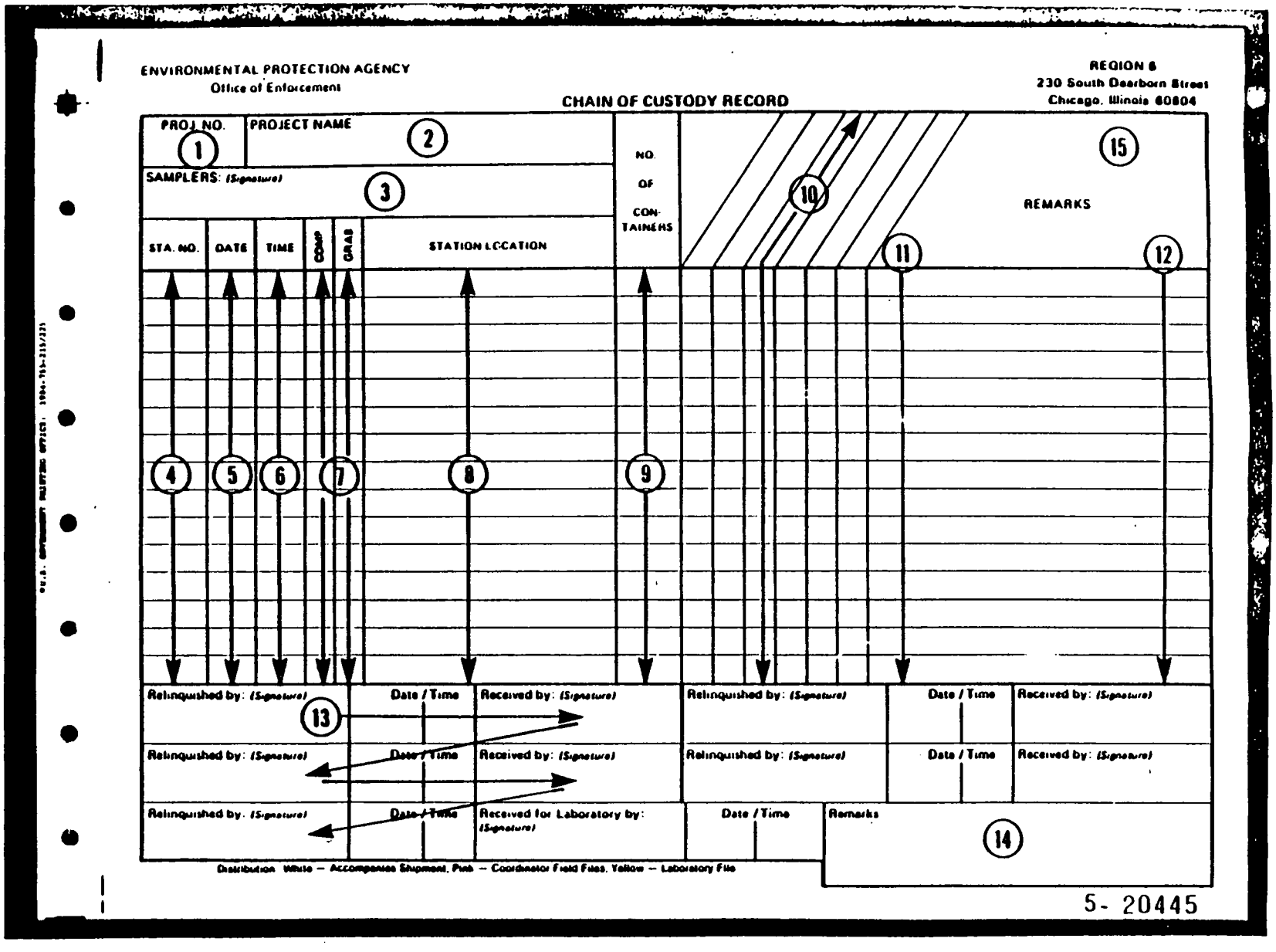
9. Enter number of containers per sample and container volume (e.g., 2-40 ml).

10. List analyses individually.

11. Construct column heading for traffic report number and list serial numbers for corresponding sample identification codes.

12. Construct column heading for "tag number" and list tag numbers for each sample container.

13. Obtain signature of sample team leader and carry out chain-of-custody procedures.



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 7

14. State carrier service and airbill number, lab service, and custody seal numbers:

15. Write in the words "CASE #:" and enter the case number.

Notice of Transmittal (Figure 8)

1. Enter name of team leader.

2. Enter team leader's firm name.

3. Enter case number.

4. Complete date.

5. Enter number of samples shipped.

6. Enter matrix of samples.

7. Enter the site name in words.

8. Enter the site location of the site (city, state).

Receipt for Samples Form

A completed Receipt for Samples Form will be used whenever splits are provided to other parties. This form must be completed and a copy given to the other party. The original will be retained in the project files. At potential source sites, splits of all samples collected must be offered to an official at the site. If the splits are declined, the Receipt for Samples Form should be so marked.

Field Log Book

All information pertinent to a field survey or sampling effort will be recorded in a log book or equivalent standardized form. Each page/form will be consecutively numbered and will be at least 4-1/2 inches by 7 inches in size. All entries will be made in indelible ink or hard lead pencil and all corrections will consist of line-out deletions that are initialed and dated. As a minimum, entries in a log book will include the following:

- Purpose of sampling.
- Location, description, and log photographs of the sampling point.
- Details of the sampling site (for example, the elevation of the casing, casing diameter and depth, integrity of the casing, etc.).

Figure 8

NOTICE OF TRANSMITTAL

DATE:

TO: CH2M HILL, REM/FIT OFFICE, Region V-X (WI)
310 West Wisconsin Avenue, Suite 700
P.O. Box 2090
Milwaukee, Wisconsin 53201

Attention: Shirley Stringer

FROM: (1) Name / (2) Firm

CH2M HILL PROJECT NO.:

Enclosed are appropriate copies of the sample documentation forms completed under Case #(3), for the (4), 19(4), shipment of (5) Quantity (6) Matrix samples from the (7) site located in (8), (8).

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- Name and address of field contact.
- Documentation of procedures for preparation of reagents or supplied which become an integral part of the sample (e.g., filters and absorbing reagents).
- Identification of sampling crew members.
- Type of sample (for example, groundwater, soil, sludge, or wastewater).
- Suspected waste composition.
- Number and volume of sample taken.
- Sampling methodology, including distinction between grab and composite samples.
- Sample preservation.
- Date and time of collection.
- Collector's sample identification number(s).
- Sample distribution and how transported (for example, name of the laboratory and cartage agent--Federal Express, United Parcel Service).
- References such as maps of the sample site.
- Any field measurements made (for example, pH, specific conductance, temperature, and water depth).
- Signature and date by the personnel responsible for observations.
- Decontamination procedures.

Sampling situations vary widely. No general rules can specify the extent of information that must be entered in a log book or standardized form. However, records will contain sufficient information so that someone can reconstruct the sampling activity without relying on the sample collector's memory. The log book and standardized forms will be kept under strict chain-of-custody.

Corrections to Documentation

Unless prohibited by weather conditions, all original data recorded on Traffic Report Forms, Sample Identification Tags, Chain-of-Custody Records, and Receipt for Sample Forms will be written with waterproof ink. No accountable serialized

documents are to be destroyed or thrown away, even if they are illegible or contain inaccuracies that require a replacement document.

If an error is made on an accountable document assigned to one individual, that individual shall make corrections by making a line through the error and entering the correct information. The erroneous information should not be obliterated. Any subsequent error discovered on an accountable document should be corrected by the person who made the entry. All subsequent corrections must be initialed and dated.

Laboratory Custody

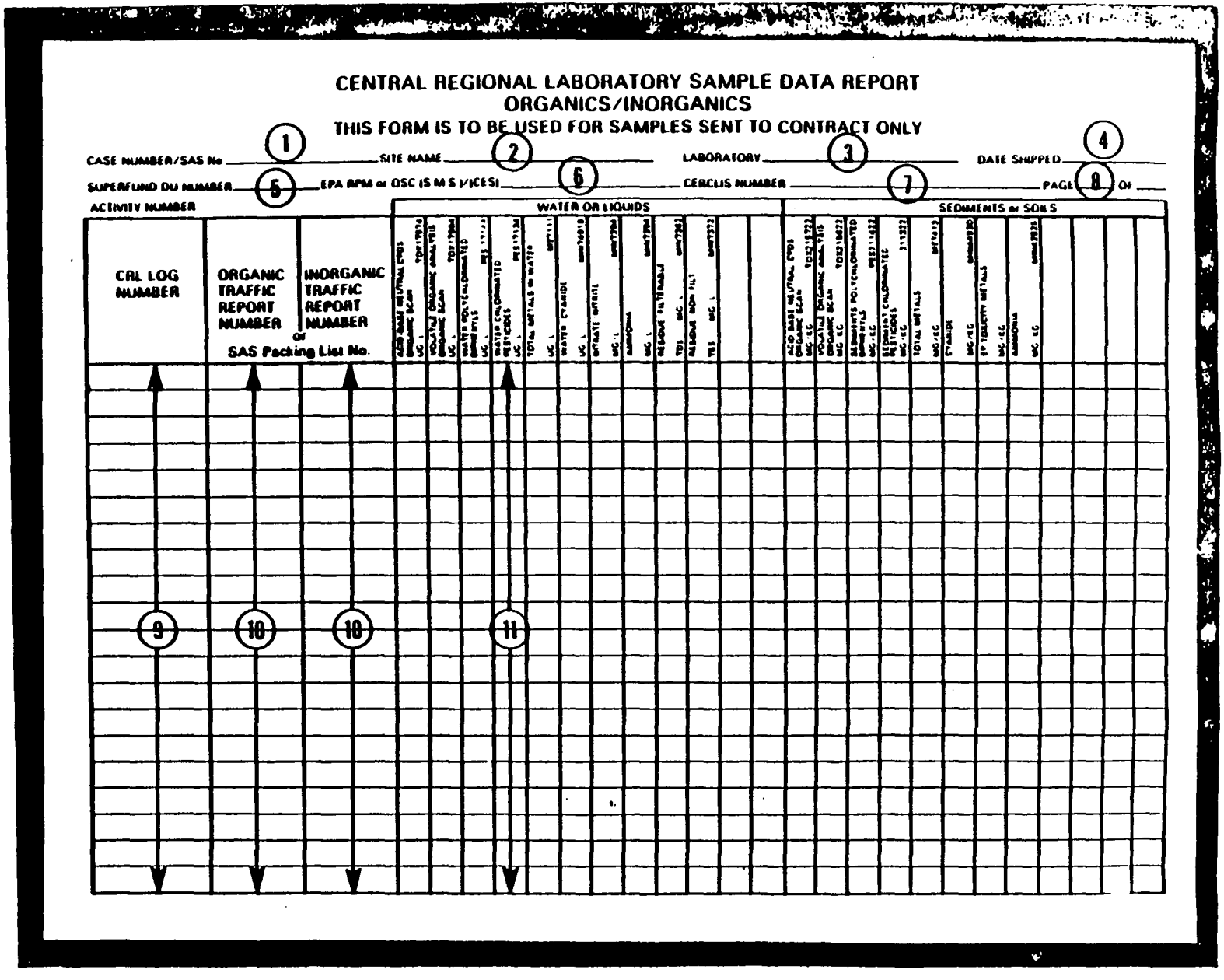
Laboratory custody will conform to procedures established for the CLP. These procedures include:

- Designation of a sample custodian.
- Correct completion by the custodian of the chain-of-custody record, sample tag, and laboratory request sheet (including documentation of sample condition upon receipt).
- Laboratory sample tracking and documentation procedures.
- Secure sample storage (of the appropriate environment--refrigerated, dry, etc.).
- Proper data logging and documentation procedures including custody of all original laboratory records.

Central Regional Laboratory Sample Data Report (Figure 9)

The Central Regional Laboratory Sample Data Report form is filled out by the CH2M HILL Sample Documentation Coordinator. A separate report is filled out for each laboratory that receives samples.

1. Enter the case number and/or SAS number.
2. Enter site name.
3. Enter the laboratory name.
4. Enter the date shipped.
5. Enter the Superfund D.U. number.
6. Enter the EPA RPM.



CENTRAL REGIONAL LABORATORY SAMPLE DATA REPORT
ORGANICS/INORGANICS
THIS FORM IS TO BE USED FOR SAMPLES SENT TO CONTRACT ONLY

CASE NUMBER/SAS No. (1) SITE NAME (2) LABORATORY (3) DATE SHIPPED (4)
SUPERFUND DU NUMBER (5) EPA RPM or OSC (S M S J/ICES) (6) CERCLIS NUMBER (7) PAGE (8) OF
ACTIVITY NUMBER

WATER OR LIQUIDS
SEDIMENTS or SOILS

CRL LOG NUMBER
ORGANIC TRAFFIC REPORT NUMBER or SAS Packing List No.
INORGANIC TRAFFIC REPORT NUMBER

ACID BASE NEUTRAL CPDS
ORGANIC SCAN
VOC 1
VOLATILE ORGANIC ANALYSIS
ORGANIC SCAN
VOC 1
WATER POLYCHLORINATED BIPHENYLS
VOC 1
WATER CHLORINATED PESTICIDES
VOC 1
TOTAL METALS in WATER
VOC 1
WATER CYANIDE
VOC 1
NITRATE NITRITE
VOC 1
AMMONIA
VOC 1
RESIDUE FILTERABLE
TDS
VOC 1
RESIDUE NON FILT
TDS
VOC 1
ACID BASE NEUTRAL CPDS
ORGANIC SCAN
VOC/LC
VOLATILE ORGANIC ANALYSIS
ORGANIC SCAN
VOC LC
SEDIMENTS POLYCHLORINATED BIPHENYLS
VOC/LC
SEDIMENT CHLORINATED PESTICIDES
VOC/LC
TOTAL METALS
VOC/LC
CYANIDE
VOC/LC
EP TOXICITY METALS
VOC/LC
AMMONIA
VOC LC

(9) (10) (10) (11)

NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 9

7. Enter the CERCLIS number.

8. Enter the page numbers.

9. Enter the CRL numbers.

10. Enter the organic or inorganic traffic report number or the SAS packing list number.

11. Check the appropriate box for the analyses to be performed.

PACKING AND SHIPPING PROCEDURES

Sample packaging and shipping procedures are based on U.S. EPA Specifications, as well as Department of Transportation (DOT) regulations (40 CFR). The procedures vary according to sample concentration and matrix and are designed to provide optimum protection of samples and the public.

All samples will be shipped within 48 hours of collection or before 50 percent of the holding time has elapsed. Shipping containers must be insulated, durable, and watertight. Bagged samples (to prevent vermiculite contamination of samples, all containers regardless of size/type must be placed inside sealed plastic bag before packing in vermiculite/zonolite) are to be cushioned within the shipping container with vermiculite packing material (Zonolite). Preformed poly-foam cooler liners are available for shipment of low-concentration samples only.

Following shipment, airbill numbers must be called in to the SMO and to the sample documentation coordinator.

Step-by-step packing instructions are provided below.

Low-Concentration Samples

1. Prepare cooler(s)d for shipment.

   - o Tape drain(s) shut.
   - o Affix "This Side Up" labels on all four sides and "Fragile" labels on at least two sides of each cooler.
   - o Place mailing label with laboratory address on top of cooler(s).
   - o Fill bottom of cooler(s) with about 3 inches of vermiculite or use preformed poly-foam liner (low concentration only).

- o Place appropriate traffic reports, SAS packing lists, or Regional field sheets and chain-of-custody records with corresponding custody seals on top of each cooler.

2. Arrange decontaminated sample containers in groups by sample number.

3. Mark volume levels on bottles with a grease pencil.

4. Secure appropriate sample tags around caps/lids of containers with string or wire.

5. Secure container caps/lids with strapping tape.

6. Arrange containers in front of assigned coolers.

7. Affix appropriate adhesive labels from assigned traffic report to each container. Protect with clear label protection tape.

8. Seal each container within a separate plastic bag.

9. Arrange containers in coolers so that they do not touch.

10. If ice is required to preserve the samples, cubes should be repackaged in double zip-loc bags, and placed on and around the containers (especially on VOA vials).

11. Fill remaining spaces with vermiculite (or place poly-foam liner cover on top of samples).

12. Sign chain-of-custody form (or obtain signature) and indicate the time and date it was relinquished to Federal Express, Purolator, Emery, or other carrier as appropriate.

13. Separate copies of forms. Seal proper copies within a large zip-loc bag and tape to inside lid of cooler. Distribute remaining copies as indicated in the following sections.

14. Close lid and latch.

15. Carefully peel custody seals from backings and place intact over lid openings (right front and left back). Cover seals with clear protection tape.

16. Tape cooler shut on both ends, making several complete revolutions with strapping tape (do not cover custody seals). See Figure 10 for an illustration of a cooler ready for shipment.

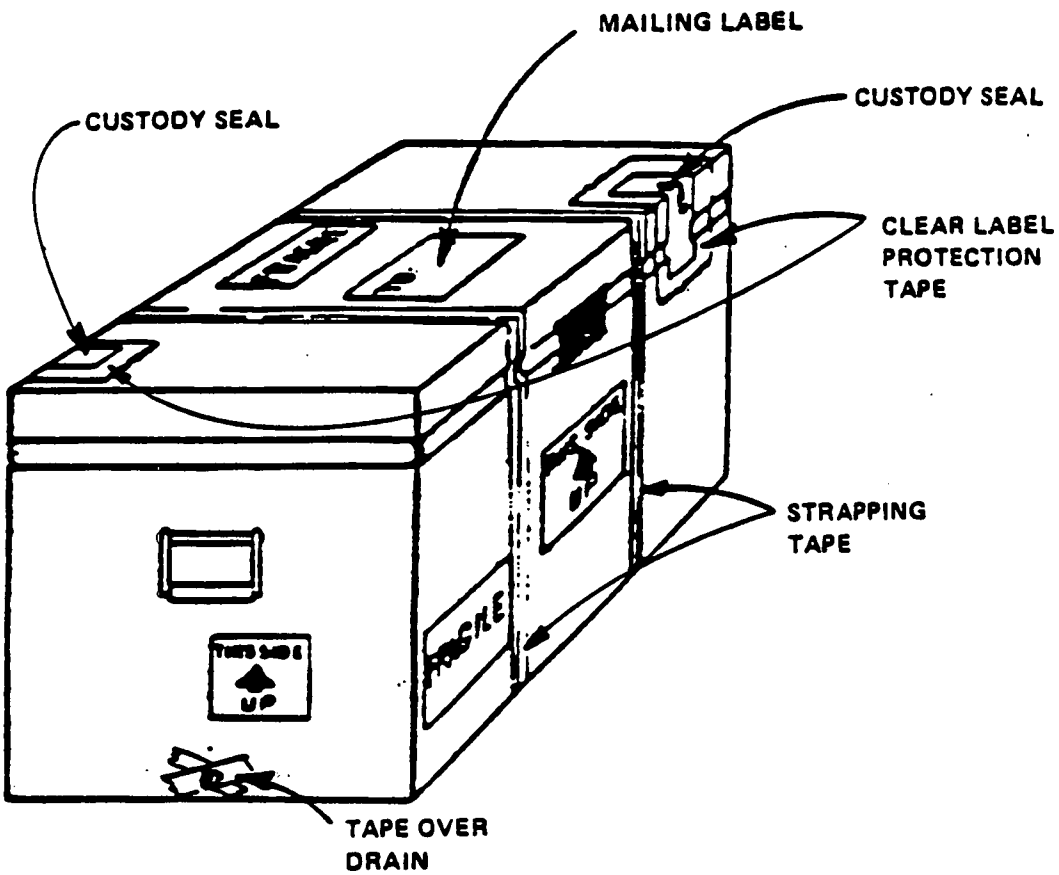

MAILING LABEL
CUSTODY SEAL
CUSTODY SEAL
CLEAR LABEL PROTECTION TAPE
STRAPPING TAPE
TAPE OVER DRAIN
UP
FRAGILE

FIGURE 10

17. Relinquish to Federal Express. Place airbill receipt inside the mailing envelope and send to the sample documentation coordinator, along with the other documentation.

18. Telephone the Sample Management Office in Alexandria, Virginia. (NOTE: This step should be omitted for samples sent to the CRL).

    Ms. Leslie Braun (subject to change)
    (703) 557-2490

    Provide the following information:

    - Your name
    - Project name
    - Case number
    - Number of samples sent to each laboratory for analysis
    - Airbill numbers

    This must be done immediately following sample shipment. (If the SMO is closed at that time, call in the information first thing the next day.)

Medium- and High-Concentration Samples:

Medium- and high-concentration samples are packaged using the same techniques used to package low-concentration samples, with several additional restrictions. First, special airbill including a Shipper's Certification for Restricted Articles is required (see Figure 11 and 12). Second, "Flammable Liquid N.O.S." or "Flammable Solid N.O.S." labels must be placed on at least two sides of the cooler. Third, sample containers are packaged in metal cans with lids before being placed into the cooler, as indicated below.

- Place approximately one-half inch of vermiculite in the bottom of the can.
- Position the sample jar in the zip-loc bag so that the sample tags can be read through the plastic bag.
- Place the jar in the can and fill the remaining volume with vermiculite.
- Close the can and secure the lid with metal clips.

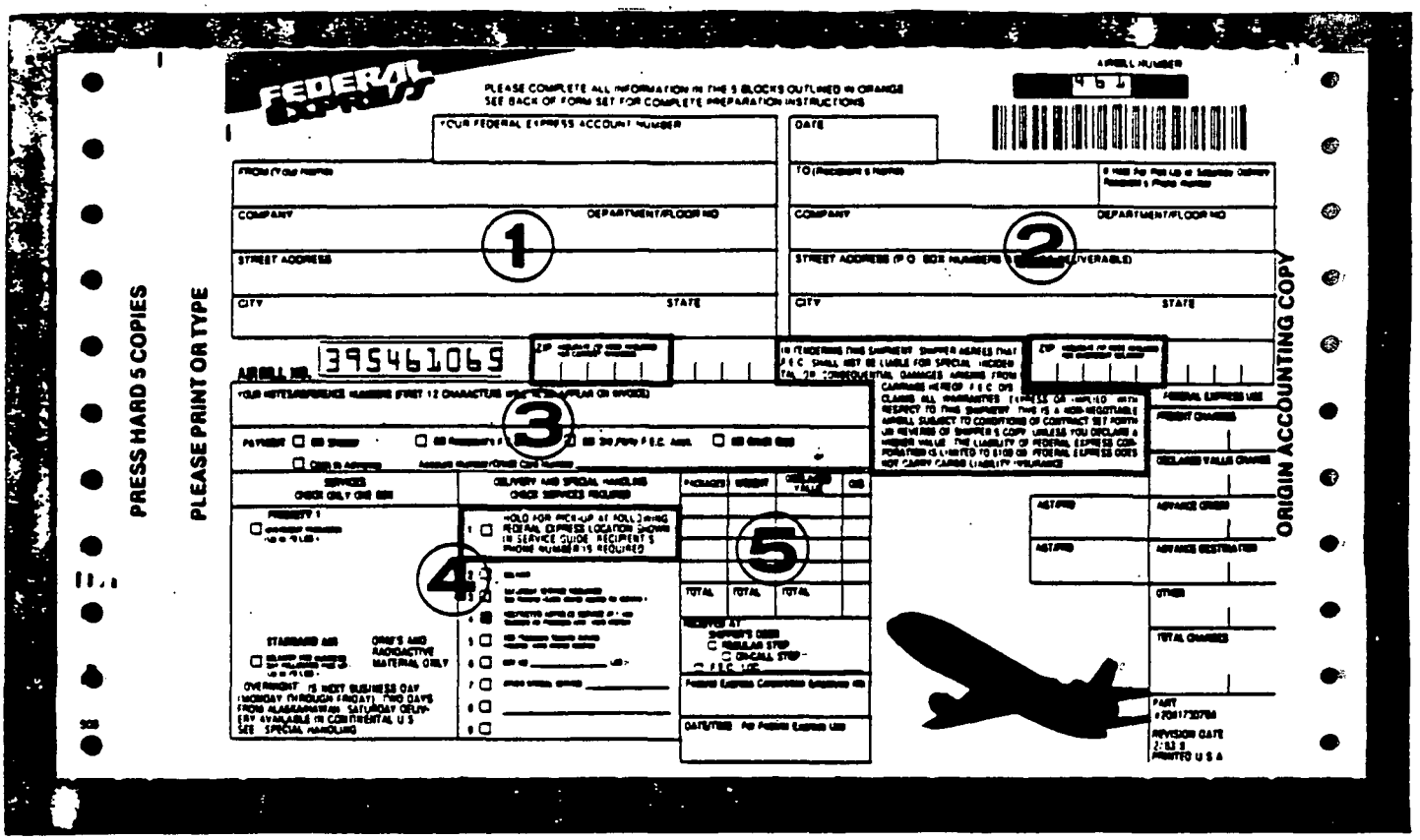


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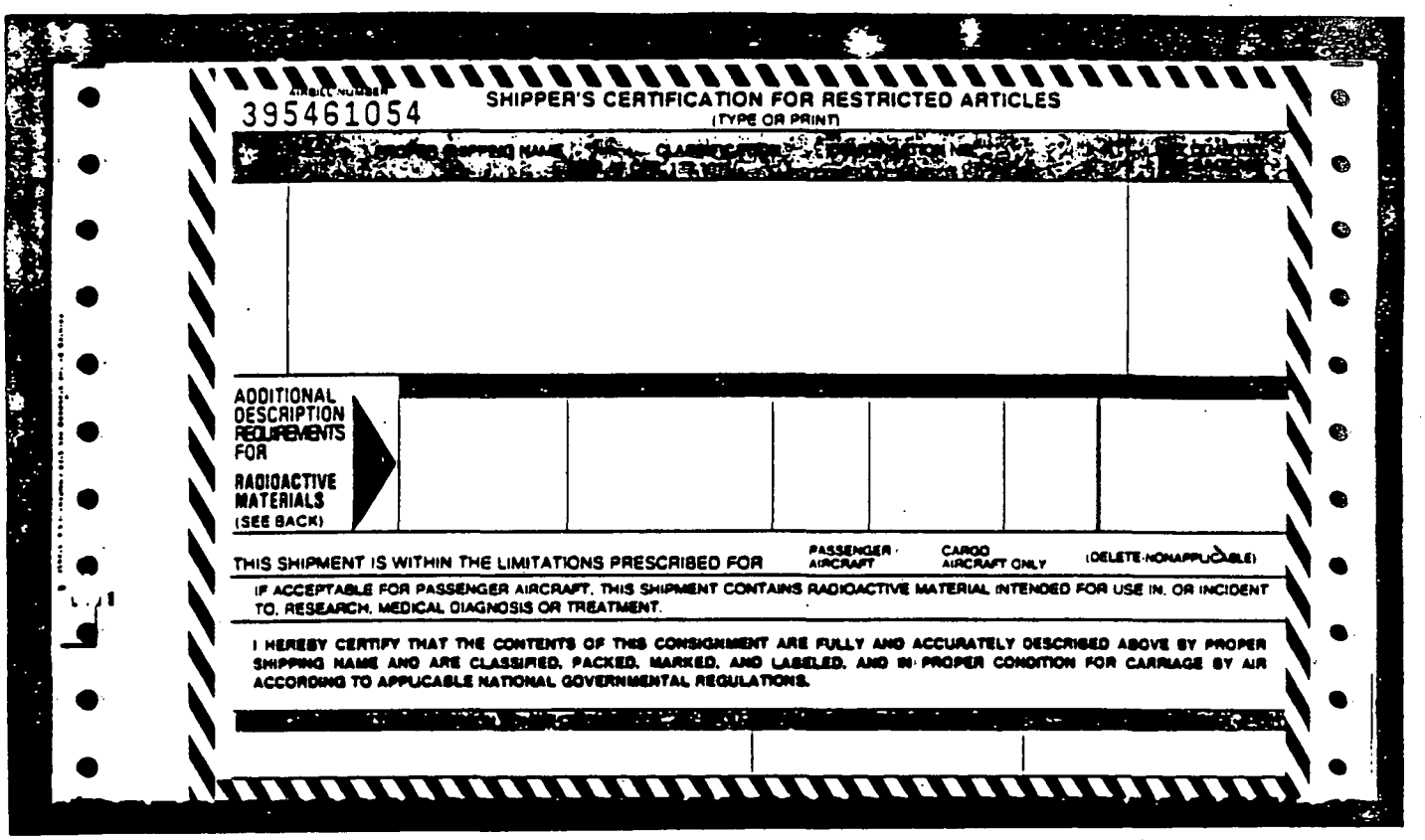
FIGURE 11



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 12

- Write the traffic report number on the lid.
- Place "This Side Up" and "Flammable Liquid N.O.S." (or "Flammable Solid N.O.S.") labels on the can.
- Place the cans in the cooler.

Distribution of Completed Documents

Final disposition of the completed documents is as follows:

- Shipped with Samples:
  - Chain-of-custody form, white original
  - Traffic report forms, white and yellow copies
  - SAS packing list, pink and gold copies
  - Sample tags
- Retained by RI Project Manager:
  - Sample identification matrix
  - Field log books (at completion of project)
- Sent to CH2M HILL Documentation Coordinator:
  - Chain-of-custody form, pink and yellow copies
  - Traffic report forms, white original and pink copy
  - SAS packing list, white original and yellow copy
  - Notice of transmittal

Special Instructions for Shipping Samples via Federal Express

1. Label cooler as hazardous shipment.
   - Write shipper's address on outside of cooler. If address is stenciled on, just write "shipper" above it.
   - Write or affix sticker saying "This Side Up" on two adjacent sides.
   - Write or affix sticker saying "ORM-E" with box around it on two adjacent sides. Below ORM-E, write NA#9188.
   - Label cooler with "Hazardous Substance, NOS.", and "liquid" or "solid", as applicable.

2. Complete the special shipping bill for restricted articles (Figures 11 and 12).

   - Under Proper Shipping Name, write "Hazardous Substance, NOS." and "liquid" or "solid", as applicable.
   - Under Class, write "ORM-E."
   - Under Identification No., write NA#9188.

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Appendix E-2
SAMPLE CUSTODY PROCEDURES

INTRODUCTION

It is U.S. EPA and Region V policy to follow the U.S. EPA Region V sample custody or chain-of-custody protocols as described in "NEIC Policies and Procedures." EPA-330/9-78-001-R, revised June 1985. This custody is in three parts: sample collection, laboratory, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if the documents:

- o Are in your possession;
- o Are in your view, after being in your possession;
- o Were in your possession and you placed them in a secured location; or
- o Are in a designated secure area.

FIELD SPECIFIC CUSTODY PROCEDURES

The sample packaging and shipment procedures summarized below will insure that the samples will arrive at the laboratory with the chain-of-custody intact.

Field procedures are as follows:

A. The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples.

B. All bottles will be tagged with sample numbers and locations. The Sample Management Office (SMO) number and stickers will be affixed.

C. Sample tags are to be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because the ballpoint would not function in freezing weather.

D. The contractor's site manager must review all field activities to determine whether proper custody

procedures were followed during the field work and decide if additional samples are required. He or she should notify the U.S. EPA Remedial Project Manager of a breach or irregularity in chain-of-custody procedures.

Transfer of custody and shipment procedures are as follows:

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

B. Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be locked and secured with strapping tape and EPA custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.

C. Whenever samples are split with a source or government agency, a separate Sample Receipt is 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 should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "received by" space.

D. All shipments will be accompanied by the Chain of Custody Record identifying the contents. The original record will accompany the shipment, and the pink and yellow copies will be retained by the sampler for return to the sampling office.

E. If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on

the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

LABORATORY CUSTODY PROCEDURES

A. CONTRACT LABORATORY

The chain-of-custody procedures for Contract Laboratory Program (CLP) are described in the SOW's for RAS's. This same custody procedure applies to SAS's. These custody procedures along with the holding time requirements for CLP samples are described in the appropriate SOW documents.

B. CENTRAL REGIONAL LABORATORY

The Central Regional Laboratory has its own regional custody scheme for Drinking Water Specific Samples. There are four possible ways in which the CRL may be involved in chain-of-custody sample tracking:

1. Samples are delivered to the CRL for in-house analysis.

2. Samples are delivered to the CRL. Some are sent out to a contractor and some remain at the CRL, or the samples are sent to several contract laboratories.

3. Samples are delivered to the CRL and the entire shipment is sent to one contract laboratory.

4. Samples are shipped directly from the field to a contract laboratory without ever being delivered to the CRL.

The internal CRL Custody Protocol has been revised so that it addresses all four of these situations and also meets all National EPA custody requirements. Moreover, the revised protocol requires only one new internal document--the Custody Logbook. This logbook replaces the existing Shipping and Receiving Log. The new procedures are applied to the four custody situations as follows in Appendix A.

FINAL EVIDENCE FILES CUSTODY PROCEDURES

The purge files from the CRL and Contract Laboratory Program (CLP) are maintained by Region V CRL Laboratory Support Team, Data Coordinator. The purge files include the chain-of-custody sheets, sample tags and raw data records.

The contractor maintains the RI files along with all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and CPMS data reviews in a secured, limited access area and under custody of the contractor's site manager.

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APPENDIX A

A. In-House Analysis

Samples are shipped or delivered to the CRL under chain of custody. The CRL Sample Custodian signs the Chain of Custody Record (see Figure A-1) in the "Received by" space. The Sample Custodian also signs in the "Received for Laboratory by" space. This document is then complete. It is filed in the folder for the given data set.

The Sample Custodian then enters the following sample information into the Custody Logbook (see Figure A-2).

1, 2, 3 - Self-explanatory.

4 - "Matrix" refers to a brief sample description, such as "water," "oil," "mud," etc. Parameter is self-explanatory.

5 - Self-explanatory.

6,7,8 - The Sample Custodian initials the date (month/day/year) and the time when samples were received. Time is expressed using a 24-hour clock, so that 1:30 P.M. is recorded as 13:30.

9 - Each shelf in all custody areas should be numbered, so that the storage location can be identified by the shelf number. This number is entered in column 9.

When an analyst checks out a sample, columns 10 thru 13 are completed.

10 - The Sample Custodian initials the correct column.

11 - The analyst initials the correct column.

12, 13 - The Sample Custodian enters the date and time.

When a sample is transferred from one analyst to another within the CRL, both analysts initial the back of the custody tag. They also enter the date and time.

e.g.,: AJ to DM, 9/12/82, 15:30

(It is not necessary to return the sample to the person who originally checked it out of custody.)

When the analyses are completed, the analyst returns the sample to the Sample Custodian. They both fill in columns 14 thru 18.

14, 15, 16, 17 - Self-explanatory.

18. - The Sample Custodian stores the sample in a custody area. The location is entered in column 18. This is probably not the same as the original location listed in column 9.

When samples are discarded, columns 19 and 20 are filled in. The tags are removed from the sample bottles and are filed in the folder for that data set. TAGS ARE NEVER DISCARDED.

Any appropriate information, including initials, is entered in column 21.

e.g., : "Sample was broken. 10/1/82, AJ"
"Sample was used up. 10/1/82, AJ"
"Insufficient sample. 10/1/82, AJ"

Even if a sample is destroyed, the tag must be returned to the custody folder. An explanation should be written on the tag and in the Custody Logbook.

B. Sample Shipments With Several Destinations

Samples are shipped to the CRL under chain of custody. The CRL Sample Custodian receives the shipment as described in Section A. The Custodian opens the sealed container, logs in all of the samples, and then repacks those samples which will be sent to a contract laboratory. The Custodian fills out new custody forms and includes them with the shipment as described in the Environmental Services Division (ESD) or National Contract Laboratory Protocols.

The Sample Custodian logs all of the samples into the Custody Logbook. The procedure is the same as described in Section A, with the following exceptions.

9 - No entry here.

10 - The Sample Custodian initials here.

11 - The Sample Custodian enters the name of the laboratory to which the samples were shipped.

14 thru 20 - These columns are used only if the contract laboratory returns the samples.

21 - The Sample Custodian enters the shipper and the airbill number.

e.g.,: "Emery, #9011625"

C. Entire Sample Shipment Sent to One Destination

Samples are shipped to the CRL under chain of custody. The CRL Sample Custodian receives the samples. The Custodian signs the chain of custody record in the "Received by" space. The Custodian logs in the samples and then packs them for shipment and includes original the chain of custody record with the samples. The custodian does not sign the "Received for Laboratory by" space (see Figure A-3).

The Sample Custodian logs all of the samples into the Custody Logbook as described in Section B.

D. Samples Shipped From the Field to Contract Laboratories

When samples are shipped directly from the field to a contract laboratory, no one at the CRL signs the chain of custody Record.

The sampling team submits a report to the CRL describing their sampling activities. The Sample Custodian enters that information into the Custody Logbook as follows:

1 thru 4 - Self-explanatory.

5 - Tag numbers may not be available in the field report.

6 thru 9 - Not applicable.

10 - The Sample Custodian enters "field."

11 - The Sample Custodian enters the name of the laboratory to which the samples were sent.

12, 13 - Shipping date and time are entered, if available.

14 thr 20 - These columns are used only if the samples are sent to the CRL by the contract laboratory.

21 - Shipper, airbill number, and any other comments are entered here.

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

FIGURE #1

REGION 5
230 South Dearborn Street
Chicago, Illinois 60604

CHAIN OF CUST RECORD

PROJ. NO.	PROJECT NAME					NO OF CONTAINERS	REMARKS
SAMPLERS: (Signature)							
STA. NO.	DATE	TIME	COMP	GRAB	STATION LOCATION		

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	

Distribution: White — Accompanies Shipment; Pink — Coordinator Field Files; Yellow — Laboratory File

Figure A-1 CHAIN OF CUSTODY FORM (CRL #1)

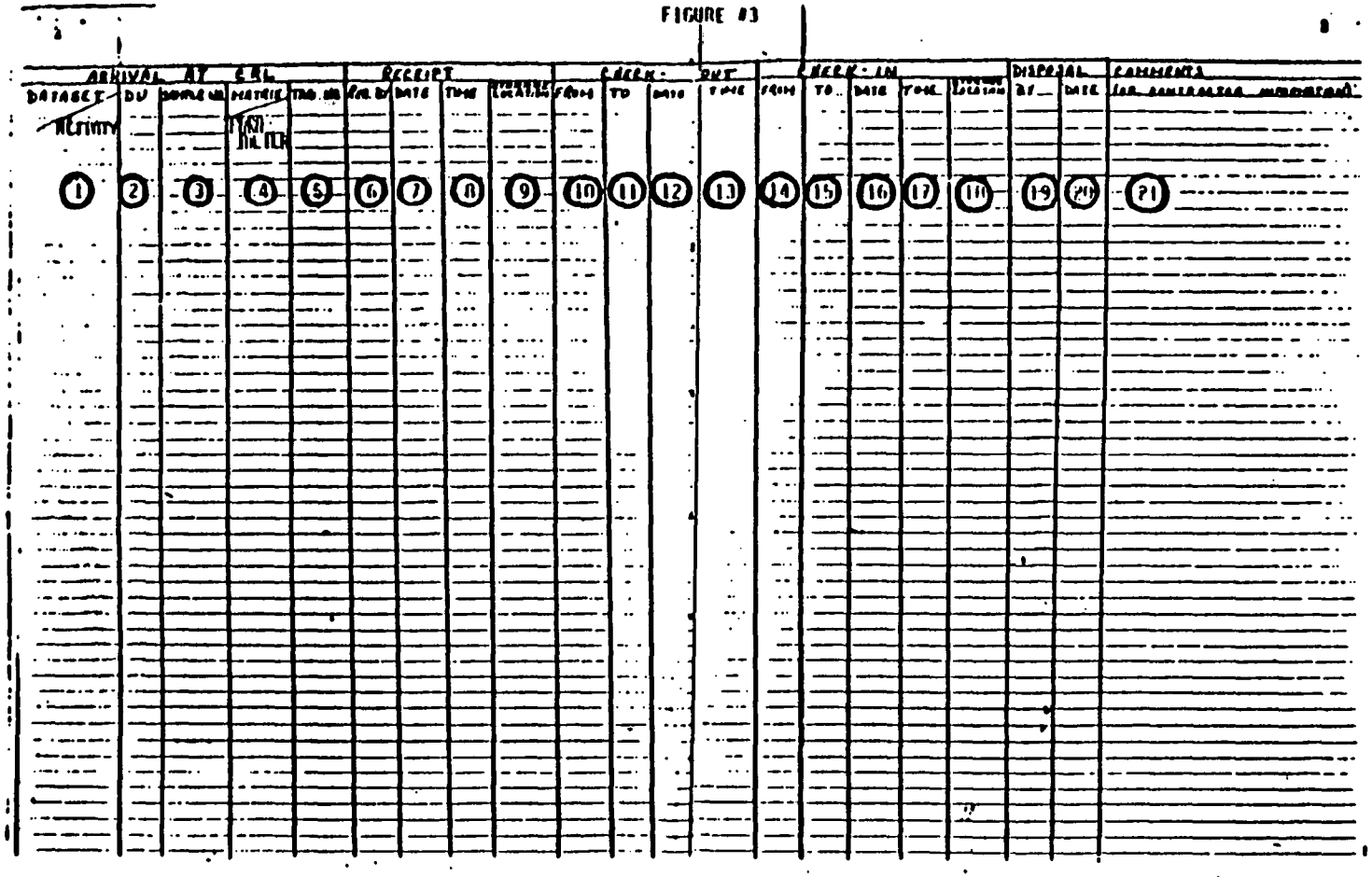
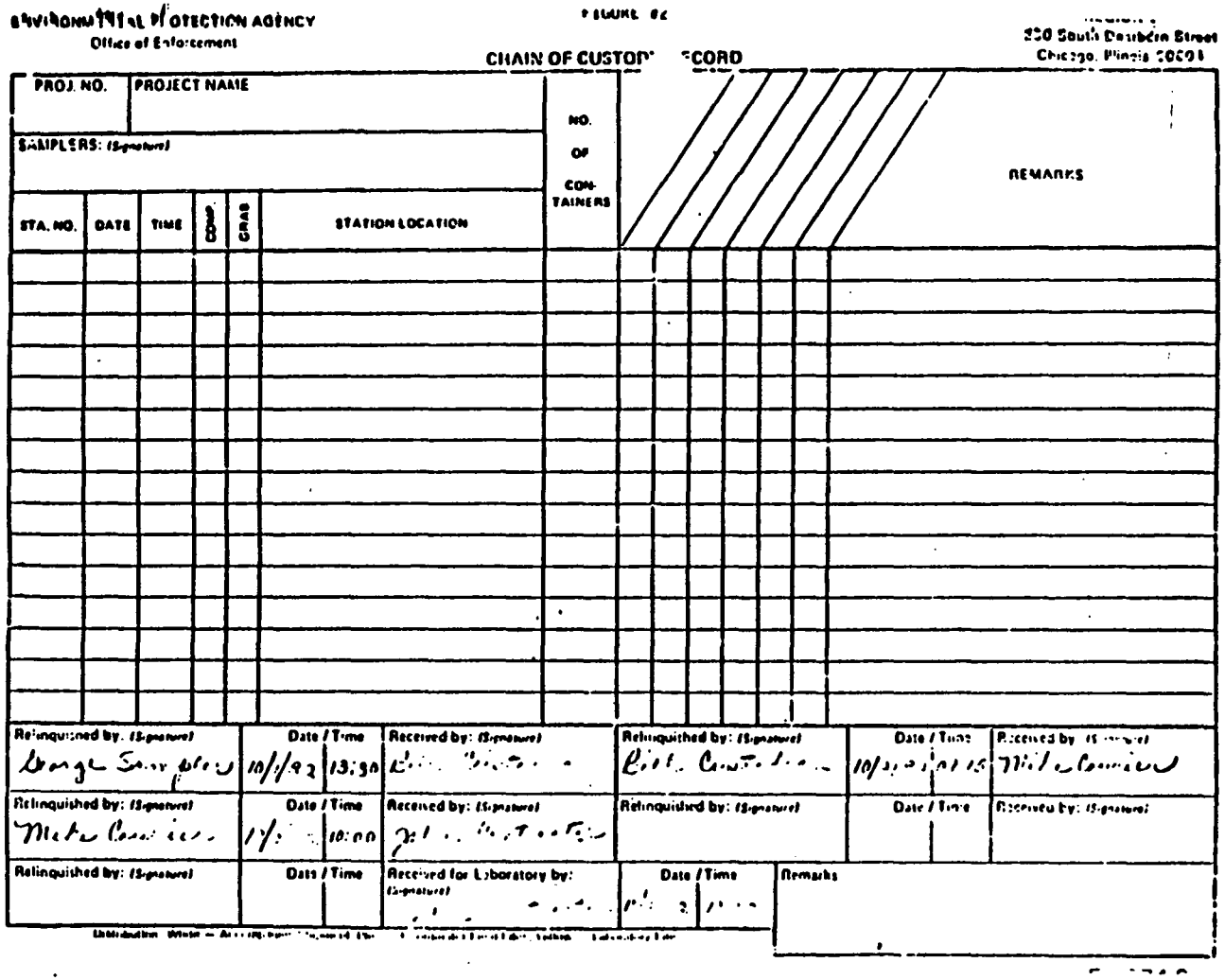


Figure A-2 CENTRAL REGIONAL LABORATORY LOG SHEET (CRL #3)



ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

FIGURE #2

CHAIN OF CUSTODY RECORD

220 South Dearborn Street
Chicago, Illinois 60604

PROJ. NO.	PROJECT NAME					NO. OF CONTAINERS	REMARKS
SAMPLERS: (Signature)							
STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION		

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Bruce Sampson	10/1/92 13:30				
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
	10:00				
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	

Distribution: White — Accompanies Shipment; Yellow — Coordinator Retains; Pink — Laboratory Copy

Figure A-3 CHAIN OF CUSTODY FORM (CRL #2)

Appendix F
ROUTINE ANALYTICAL SERVICES (RAS) PARAMETERS

Hazardous Substance List (HSL) and
Contract Required Detection Limits (CRDL)**

Volatiles	CAS Number	Detection Limits* Low Water[a] ug/L	Low Soil/Sediment[b] ug/Kg
1. Chloromethane	74-87-3	10	10
2. Bromomethane	74-83-9	10	10
3. Vinyl Chloride	75-01-4	10	10
4. Chloroethane	75-00-3	10	10
5. Methylene Chloride	75-09-2	5	5
6. Acetone	67-64-1	10	10
7. Carbon Disulfide	75-15-0	5	5
8. 1,1-Dichloroethene	75-35-4	5	5
9. 1,1-Dichloroethane	75-35-3	5	5
10. trans-1,2-Dichloroethene	156-60-5	5	5
11. Chloroform	67-66-3	5	5
12. 1,2-Dichloroethane	107-06-2	5	5
13. 2-Butanone	78-93-3	10	10
14. 1,1,1-Trichloroethane	71-55-6	5	5
15. Carbon Tetrachloride	56-23-5	5	5
16. Vinyl Acetate	108-05-4	10	10
17. Bromodichloromethane	75-27-4	5	5
18. 1,1,2,2-Tetrachloroethane	79-34-5	5	5
19. 1,2-Dichloropropane	78-87-5	5	5
20. trans-1,3-Dichloropropene	10061-02-6	5	5
21. Trichloroethene	79-01-6	5	5
22. Dibromochloromethane	124-48-1	5	5
23. 1,1,2-Trichloroethane	79-00-5	5	5
24. Benzene	71-43-2	5	5
25. cis-1,3-Dichloropropene	10061-01-5	5	5

(continued)

Volatiles	CAS Number	Detection Limits* Low Water[a] ug/L	Low Soil/Sediment[b] ug/Kg
26. 2-Chloroethyl Vinyl Ether	110-75-8	10	10
27. Bromoform	75-25-2	5	5
28. 2-Hexanone	591-78-6	10	10
29. 4-Methyl-2-pentanone	108-10-1	10	10
30. Tetrachloroethene	127-18-4	5	5
31. Toluene	108-88-3	5	5
32. Chlorobenzene	108-90-7	5	5
33. Ethyl Benzene	100-41-4	5	5
34. Styrene	100-42-5	5	5
35. Total Xylenes		5	5

[a]Medium Water Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Water CRDL.

[b]Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Soil/Sediment CRDL.

Semi-Volatiles	CAS Number	Detection Limits* Low Water[c] ug/L	Low Soil/Sediment[c] ug/Kg
36. Phenol	108-95-2	10	330
37. bis(2-Chloroethyl) ether	111-44-4	10	330
38. 2-Chlorophenol	95-57-8	10	330
39. 1,3-Dichlorobenzene	541-73-1	10	330
40. 1,4-Dichlorobenzene	106-46-7	10	330
41. Benzyl Alcohol	100-51-6	10	330
42. 1,2-Dichlorobenzene	95-50-1	10	330
43. 2-Methylphenol	95-48-7	10	330
44. bis(2-Chloroisopropyl) ether	39638-32-9	10	330
45. 4-Methylphenol	106-44-5	10	330
46. N-Nitroso-Dipropylamine	621-64-7	10	330
47. Hexachloroethane	67-72-1	10	330
48. Nitrobenzene	98-95-3	10	330
49. Isophorone	78-59-1	10	330
50. 2-Nitrophenol	88-75-5	10	330
51. 2,4-Dimethylphenol	105-67-9	10	330
52. Benzoic Acid	65-85-0	50	1600
53. bis(2-Chloroethoxy) methane	111-91-1	10	330
54. 2,4-Dichlorophenol	120-83-2	10	330
55. 1,2,4-Trichlorobenzene	120-82-1	10	330
56. Naphthalene	91-20-3	10	330
57. 4-Chloroaniline	106-47-8	10	330
58. Hexachlorobutadiene	87-68-3	10	330
59. 4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	10	330
60. 2-Methylnaphthalene	91-57-6	10	330
61. Hexachlorocyclopentadiene	77-47-4	10	330
62. 2,4,6-Trichlorophenol	88-06-2	10	330
63. 2,4,5-Trichlorophenol	95-95-4	50	1600

(continued

Semi-Volatiles	CAS Number	Detection Limits* Low Water[c] ug/L	Low Soil/Sediment[c] ug/Kg
64. 2-Chloronaphthalene	91-58-7	10	330
65. 2-Nitroaniline	88-74-4	50	1600
66. Dimethyl Phthalate	131-11-3	10	330
67. Acenaphthylene	208-96-8	10	330
68. 3-Nitroaniline	99-09-2	50	1600
69. Acenaphthene	83-32-9	10	330
70. 2,4-Dinitrophenol	51-28-5	50	1600
71. 4-Nitrophenol	100-02-7	50	1600
72. Dibenzofuran	132-64-9	10	330
73. 2,4-Dinitrotoluene	121-14-2	10	330
74. 2,6-Dinitrotoluene	606-20-2	10	330
75. Diethylphthalate	84-66-2	10	330
76. 4-Chlorophenyl Phenyl ether	7005-72-3	10	330
77. Fluorene	86-73-7	10	330
78. 4-Nitroaniline	100-01-6	50	1600
79. 4,6-Dinitro-2-methylphenol	534-52-1	50	1600
80. N-nitrosodiphenylamine	86-30-6	10	330
81. 4-Bromophenyl Phenyl ether	101-55-3	10	330
82. Hexachlorobenzene	118-74-1	10	330
83. Pentachlorophenol	87-86-5	50	1600
84. Phenanthrene	85-01-8	10	330
85. Anthracene	120-12-7	10	330
86. Di-n-butylphthalate	84-74-2	10	330
87. Fluoranthene	206-44-0	10	330
88. Pyrene	129-00-0	10	330
89. Butyl Benzyl Phthalate	85-68-7	10	330
90. 3,3'-Dichlorobenzidine	91-94-1	20	660
91. Benzo(a)anthracene	56-55-3	10	330
92. bis(2-ethylhexyl)phthalate	117-81-7	10	330
93. Chrysene	218-01-9	10	330
94. Di-n-octyl Phthalate	117-84-0	10	330
95. Benzo(b)fluoranthene	205-99-2	10	330
96. Benzo(k)fluoranthene	207-08-9	10	330
97. Benzo(a)pyrene	50-32-8	10	100

(continued)

Semi-Volatiles	CAS Number	Detection Limits* Low Water[c] ug/L	Low Soil/Sediment[d] ug/Kg
98. Indeno(1,2,3-cd)pyrene	193-39-5	10	330
99. Dibenz(a,h)anthracene	53-70-3	10	330
100. Benzo(g,h,i)perylene	191-24-2	10	330

[c]Medium Water Contract Required Detection Limits (CRDL) for Semi-Volatile HSL Compounds are 100 times the individual Low Water CRDL.

[d]Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Semi-Volatile HSL Compounds are 60 times the individual Low Soil/Sediment CRDL.

Pesticides	CAS Number	Detection Limits* Low Water[e] ug/L	Low Soil/Sediment[f] ug/Kg
101. alpha-BHC	319-84-6	0.05	8.0
102. beta-BHC	319-85-7	0.05	8.0
103. delta-BHC	319-86-8	0.05	8.0
104. gamma-BHC (Lindane)	58-89-9	0.05	8.0
105. Heptachlor	76-44-8	0.05	8.0
106. Aldrin	309-00-2	0.05	8.0
107. Heptachlor Epoxide	1024-57-3	0.05	8.0
108. Endosulfan I	959-98-8	0.05	8.0
109. Dieldrin	60-57-1	0.10	16.0
110. 4,4'-DDE	72-55-9	0.10	16.0
111. Endrin	72-20-8	0.10	16.0
112. Endosulfan II	33213-65-9	0.10	16.0
113. 4,4'-DDD	72-54-8	0.10	16.0
114. Endosulfan Sulfate	1031-07-8	0.10	16.0
115. 4,4'-DDT	50-29-3	0.10	16.0
116. Endrin Ketone	53494-70-5	0.10	16.0
117. Methoxychlor	72-43-5	0.5	80.0
118. Chlordane	57-74-9	0.5	80.0
119. Toxaphene	8001-35-2	1.0	160.0
120. AROCLOR-1016	12674-11-2	0.5	80.0
121. AROCLOR-1221	11104-28-2	0.5	80.0
122. AROCLOR-1232	11141-16-5	0.5	80.0
123. AROCLOR-1242	53469-21-9	0.5	80.0
124. AROCLOR-1248	12672-29-6	0.5	80.0
125. AROCLOR-1254	11097-69-1	1.0	160.0
126. AROCLOR-1260	11096-82-5	1.0	160.0

[e]Medium Water Contract Required Detection Limits (CRDL) for Pesticide HSL Compounds are 100 times the individual Low Water CRDL.

[f]Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Pesticide HSL compounds are 15 times the individual Low Soil/Sediment CRDL.

*Detection limits listed for soil/sediment are based on wet weight. The detection limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, as required by the contract, will be higher.

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

Table 1. Elements Determined by Inductively Coupled Plasma Emission or Atomic Absorption Spectroscopy

Element	Contract Required Detection Level[1,2] (ug/L)
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5000
Chromium total (+3, +6)	10
Cobalt	50
Copper	25
Iron	100
Lead	5
Magnesium	5000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5000
Selenium	5
Silver	10
Sodium	5000
Thallium	10
Vanadium	50
Zinc	20