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Kerr-McGee Chemical Corporation

Quality Assurance Project Plan for Predesign Task 2

Moss-American Site
Milwaukee, Wisconsin

Draft Submittal: 18 November 1991
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Vernon Hills, Illinois

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6 October 1992

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Re: Final Version - QAPP for Predesign Task 2

Dear Ms. Eleder:

Roy F. Weston, Inc. (WESTON®) on behalf of the settling defendant, Kerr-McGee Chemical Corp. is hereby transmitting the final version of the above-referenced, U.S. EPA-approved Quality Assurance Project Plan (QAPP).

Should you have any questions regarding this transmittal, please contact us.

Very truly yours,

ROY F. WESTON, INC.

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FOR PREDESIGN TASK 2
MOSS-AMERICAN SITE
MILWAUKEE, WISCONSIN

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QUALITY ASSURANCE PROJECT PLAN

FOR

PREDESIGN TASK 2

THE MOSS-AMERICAN SITE
MILWAUKEE, WISCONSIN

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Draft Submitted: 18 November 1991

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TABLE OF CONTENTS

TABLE OF CONTENTS (Continued)

TABLE OF CONTENTS (Continued)

TABLE OF CONTENTS (Continued)

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

Appendix

LIST OF ACRONYMS/ABBREVIATIONS

ARARs	Applicable or Relevant and Appropriate Requirements
ASTM	American Standards for Testing Materials
BETX	Benzene, Ethylbenzene, Toluene, Xylene
BFB	4-Bromofluorobenzene
BNA	Base-Neutral-Acid Extractables (Semivolatile Organics)
C&NW	Chicago and North Western
CDO	U.S. EPA Region V Central District Office
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act (Superfund)
CLP	Contract Laboratory Program
cm/s	Centimeters per Second
COC	Chain of Custody
CPAH	Carcinogenic Polycyclic Aromatic Hydrocarbon
CRDL	Contract Required Detection Limits
CRL	U.S. EPA Central Regional Laboratory
cu. ft.	Cubic Feet
DFTPP	Decafluorotriphenylphosphine
DL	Detection Limit
DQO	Data Quality Objective
FEMA	Federal Emergency Management Agency
FID	Flame Ionization Detector
FSP	Field Sampling Plan
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectroscopy
HASP	Health and Safety Plan
HPLC	High Performance Liquid Chromatography
HSCD	Hazardous Site Control Division
ID	Identification
KM	Kerr-McGee Corporation
KMCC	Kerr-McGee Chemical Corporation
LDRs	Land Disposal Requirements (40 CFR 268.44)
LIMS	Laboratory Information Management System
MCL	Maximum Contaminant Levels
MDL	Method Detection Limit
MPB	Maximum Probable Background
MQAB	Monitoring and Quality Assurance Branch

LIST OF ACRONYMS/ABBREVIATIONS
(Continued)

MS	Matrix Spike
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NCP	National Contingency Plan
NPDES	National Pollutant Discharge Elimination System
NPL	National Priorities List
OERR	Office of Emergency and Remedial Response
OSWER	Office of Solid Waste and Emergency Response
OVA	Organic Vapor Analyzer
PAHs	Polycyclic (Polynuclear) Aromatic Hydrocarbons
PID	Photoionization Detector
ppb	Parts per Billion
PRP	Potentially Responsible Party
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAMP	Quality Assurance Management Plan
QAPP	Quality Assurance Project Plan
QAS	Quality Assurance Section
QC	Quality Control
RA	Remedial Action
RD	Remedial Design
RD/RA	Remedial Design/Remedial Action
RF	Response Factor
RI/FS	Remedial Investigation/Feasibility Study
ROD	Record of Decision
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
SARA	Superfund Amendments and Reauthorization Act
SEWRPC	South Eastern Wisconsin Regional Planning Commission
SIM	Selected Ion Monitor
SOP	Standard Operating Procedure
SOW	Statement of Work

LIST OF ACRONYMS/ABBREVIATIONS
(Continued)

TAL	Target Analyte List (for inorganics)
TCL	Target Compound List (for organics)
U.S. EPA	United States Environmental Protection Agency
uv	Ultraviolet
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
WDNR	Wisconsin Department of Natural Resources
WESTON	Roy F. Weston, Inc.

SECTION 1

INTRODUCTION

The United States Environmental Protection Agency (U.S. EPA) requires that all environmental monitoring and measurement efforts mandated or supported by the U.S. EPA participate in a centrally managed quality assurance (QA) program. Any party generating data under this program has the responsibility to implement minimum procedures to ensure that the precision, accuracy, completeness, and representativeness of its data are known and documented. To ensure that the responsibility is met uniformly, each party must prepare a written Quality Assurance Project Plan (QAPP) for each project that it is to perform.

This QAPP presents the organization, objectives, functional activities, and specific Quality Assurance and Quality Control (QA/QC) activities associated with the Interim Predesign Activities, and specifically predesign Task 2 related to developing a low detection method for carcinogenic polycyclic aromatic hydrocarbon (CPAH) laboratory analysis and determining background CPAH concentrations in soils and sediments for the Moss-American Superfund site (hereinafter also referred to as the facility) in Milwaukee, Wisconsin. This QAPP also describes the specific protocols that will be followed for sampling, sample handling and storage, chain of custody, laboratory analyses, and field activities. The determination of background concentrations of CPAHs in soils and sediments is important to the RD/RA for the Moss-American Site, in that cleanup standards are established in the statement of work (SOW) at either risk-based levels or area background concentrations, whichever is greater. On this basis, the background determinations, if greater than risk-based cleanup standards, will define the quantity of soil and sediment requiring remediation at the Moss-American Site. This predesign determination will be essential to designing the site remedial systems, and most importantly, may also define the extent of remediation to be conducted at the facility. These data uses establish the need for implementing a system of procedures to ensure a uniform and approved program of quality assurance.

All QA/QC procedures will be in accordance with applicable professional technical standards, U.S. EPA requirements, government regulations and guidelines, and specific project goals and requirements.

This QAPP has been prepared by Roy F. Weston, Inc. (WESTON) on behalf of Kerr-McGee Chemical Corporation (KMCC) in accordance with all U.S. EPA QAPP guidance established in the following documents:

- U.S. EPA Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80.
- U.S. EPA Region V. Content Requirements for Quality Assurance Project Plan, prepared by Cheng-Wen Tsai, February 1987, revised January 1989.
- U.S. EPA Region V Model Quality Assurance Project Plan, 1991.

SECTION 2

PROJECT DESCRIPTION

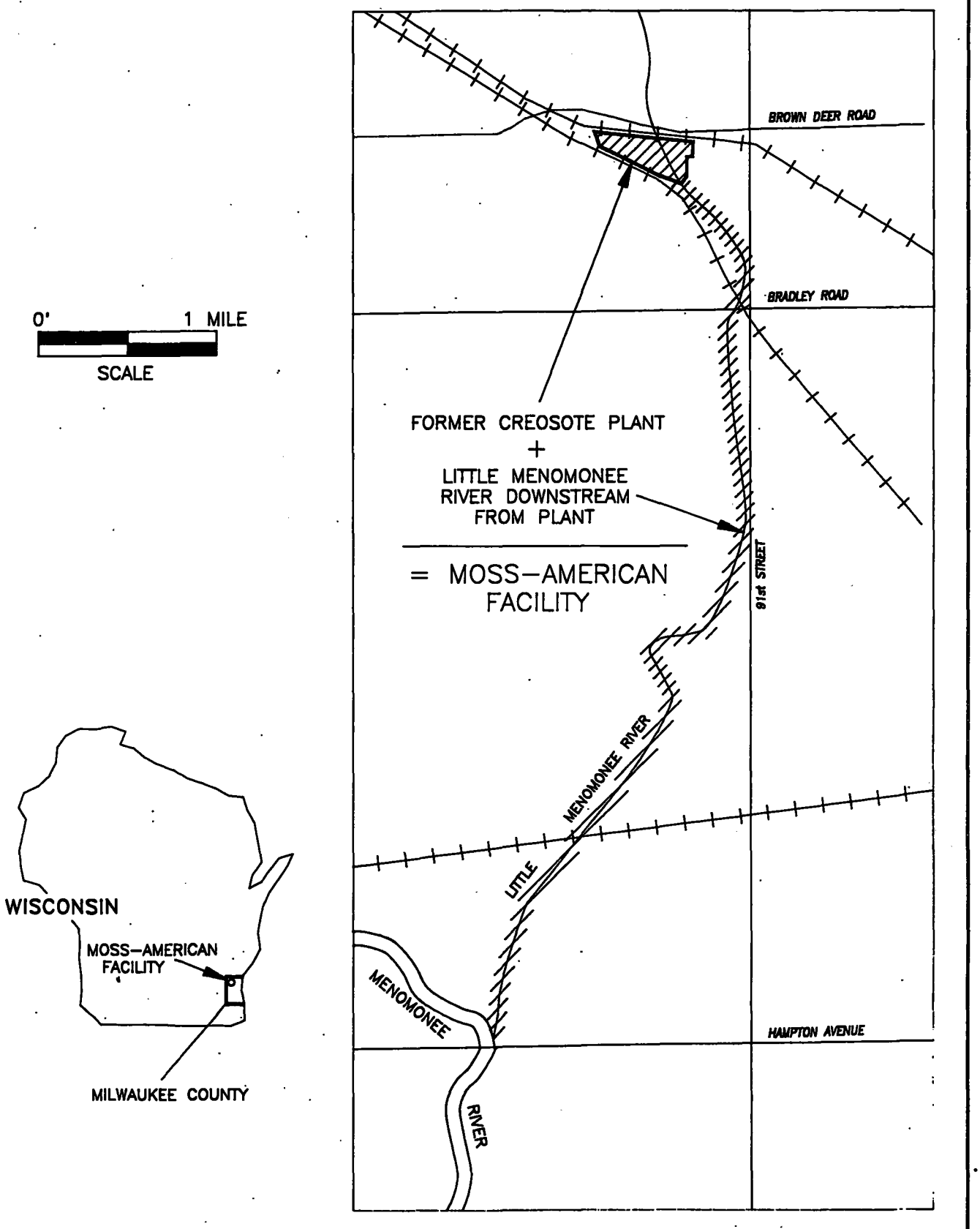
2.1 SITE LOCATION

The facility, as defined by the Consent Decree, includes the former Moss-American wood preserving plant property and approximately 5 miles of the Little Menomonee River. The Little Menomonee River, portions of which are defined as part of the facility, flows through the eastern portion of the former wood preserving plant, continuing on through the Milwaukee County Parkway, to its confluence with the Menomonee River about 5 miles south. Portions of the Little Menomonee River's floodplain are included in the Facility boundary. Fifty-one acres of the former wood preserving plant are undeveloped Milwaukee County park land. Twenty-three acres are owned by the Chicago and North Western Transportation Company and used as a loading and storage area for automobile transport. Figure 2-1 presents a general location map of the Facility.

2.2 SITE SETTING AND TOPOGRAPHY

According to the Statement of Work (SOW) for the Remedial Design/Remedial Action (RD/RA) at the Moss-American Site (U.S. EPA, 1991):

> "The Site is located in a moderately populated suburban area of mixed industrial, commercial, residential, and recreational use. Population in the nearby area is estimated at 2,036 persons per square mile."
>
> "Land use within the Menomonee River watershed is approximately 54 percent rural and 46 percent urban. Most of the urban land is in the central and southeastern portion of the watershed. The upstream watershed is predominantly rural with some new low to medium density residential uses. The Little Menomonee River is located in the upstream Menomonee River Watershed . . ."
>
> "Current land use on the site consists of an automobile transfer and storage lot on the western 23.3 acres and undeveloped county park property over the rest of the site. Site surface features are shown in [RI report] Figure 2-2. Historic land use during site operations is described in [RI report] Chapter 1 and is shown in [RI report] Figure 1-3.



WESTON MANAGERS DESIGNERS/CONSULTANTS	Three Hawthorn Parkway Vernon Hills, Illinois 60061	FIGURE 2–1	FACILITY LOCATION MAP MOSS–AMERICAN SITE Milwaukee, Wisconsin

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"The automobile storage lot is leased from the C&NW Railroad by the E&L Transport Company. New cars and trucks shipped by rail are unloaded at the lot, stored temporarily, and then shipped out by truck. The southwestern portion of the property is a paved parking and truck loading area. East of the paved area is a gravel parking area and grassy area used for overflow parking. The rail spurs on the northern part of the property are used for parking and unloading train cars. Several feet of gravel fill was added to this area to construct the spurs. Access to the automobile storage lot is limited to employees of the E&L Transport Company, C&NW Railroad, and official visitors. The property is fenced and access is controlled by security police.

"The parking areas and rail spur areas have been cut and filled to make them level. Gravel fill has also been added to much of the low-lying swampy areas. The former settling pond area is usually flooded during the wet season. The wooded areas along the river are also wet, often with ponded water. Mounds and levees (1 to 2 feet high) lie immediately adjacent to the river indicating areas where river dredgings have been dumped. The wooded areas west of the river, especially the southeastern part of the site, contain small mounds of trash.

"The Milwaukee County Soil Survey (1971) classified the developed areas on the site west of the river as loamy land, which is a miscellaneous land type consisting of fill or cut and borrow areas. The wooded areas on both sides of the river consist of Colwood silt loam, which is a poorly drained silty soil underlain by stratified lacustrine silt and very fine sand. According to the survey, the soils are moderately permeable with high available water capacity. The fields east of the river consist of Mequon silt loam and Ozaukee silt loam. The Mequon series is on the lower concave sideslope of the hillside east of the river. Slopes range from 1 to 3 percent, and the soil is somewhat poorly drained and generally not eroded. The Ozaukee series occupies convex sideslopes of glacial moraines. Slopes from 2 to 12 percent have caused moderate erosion problems. Drainage is good. The entire solum and part of the glacial moraine have been removed from the cut and borrow area in the field in the northeast corner of the property.

"The wooded areas along the river are classified as woodland group 7. The principal native trees listed by the soil survey are mixed northern hardwoods and stands of oak and aspen. Common species are soft maple, ash, and elm. Although a survey of vegetation was not conducted as part of this investigation, the general description given for the wooded area agrees with informal observations made during the field work. The swampy area west of the river contains grasses, cattails, and horsetails.

"The Moss-American site [former creosoting plant] is approximately 5.6 river miles upstream of the confluence of the Little Menomonee River with the Menomonee River. The channel runs through or adjacent to the site for approximately 2,100 feet. The average slope of the river in the vicinity of the site is 2.5 feet per mile, which is slightly less than the average subwatershed slope. Channel characteristics along the site are relatively constant with the following dimensions:

Top Width	25 to 35 feet
Bottom Width	5 to 10 feet
Channel Depth	5 to 10 feet
Base Flow Water Depth	1 to 2 feet

"Extremely dry conditions have resulted in short-term flows near zero at gauging stations upstream of the site.

"Continuous flow records near the site are not available. Peak flow rates were estimated in the Federal Emergency Management Agency (FEMA) study conducted in 1987. The following peak flow rates are identified for the Little Menomonee River at the Brown Deer Road bridge:

10-year	330 cfs
50-year	500 cfs
100-year	580 cfs
500-year	770 cfs

"Velocities for the 100-year storm vary from 0.6 to 0.2 foot per second on the site.

"The Federal Emergency Management Agency has established the 100-year flood plain for the stream reach through the Moss-American site. Approximately 25 percent (visual estimate) of the site is contained within the 100-year flood plain ([RI report] Figure 2-3). The flood plain elevation is established as 719.2 feet at the upstream site limits and 718.7 feet at the downstream limits."

2.3 SITE HISTORY

A summary of the Moss-American Site history as interpreted by the U.S. EPA in the RI report for the Moss-American Site (U.S. EPA, 9 January 1990) is presented below:

"A wood preserving plant was established on the site by the T.J. Moss Tie Company in 1921. The plant preserved railroad ties, poles, and fence posts with creosote. Kerr-McGee purchased the T.J. Moss facility in 1963. In 1965, after purchasing the American Creosote Company, Kerr-McGee changed the facility's name to Moss-American. The name was changed again in 1974 to Kerr-McGee Chemical Corporation--Forest Products Division. The plant closed in 1976. The eastern part of the property was acquired by Milwaukee County in 1978, and Chicago and North Western Railroad bought the western parcel in 1980.

"The creosoting process used at the plant consisted of impregnating the wood products with a mixture of 50 percent No. 6 fuel oil and 50 percent coal-based creosote. Impregnation was done at 180 psi and 200°F. Wood products were loaded into retorts in the processing area for treatment. Freshly treated wood was stacked on railcars parked on drip tracks and later transferred to the treated wood storage areas. Processing and storage areas at the site as they appeared in 1962 are shown in [RI report] Figure 1-3. The processing area consisted of the retort building, vertical tanks for creosote and fuel oil storage, and several smaller support buildings."

"Between 1921 and 1941, liquid wastes from the site were discharged directly to the Little Menomonee River. In 1941 a series of settling basins and a coke filter were installed for waste treatment; however, in 1954 a Public Health Engineer noted that the coke filter was not in place. At that time, the wastewater passed through an oil-water-sludge separator and was discharged to a 700-foot ditch (the settling pond area shown in [RI report] Figure 1-3) that ultimately discharged to the river. The ditch included one settling pond and hay filters installed at the head of culverts that passed under the tracks at 70- to 150-foot intervals. Subsurface drains added in 1952 drained to an open ditch along the northern property boundary and then to the river. The extent and configuration of the drain system is not documented.

"In 1966, the Milwaukee Metropolitan Sewerage Commission advised Moss-American that oil leaking from the drainage ditch and settling ponds was not permitted and they should be dredged and the pond walls rebuilt with uncontaminated clay. Moss-American complied with that request.

"The Wisconsin DNR issued an Administrative Order in 1970 requiring that Moss-American divert its process water discharge to the Milwaukee sanitary sewerage system. In 1971, the company completed the diversion project, and discharges to the river were limited to water softener wastes and stormwater runoff.

"In 1971, the settling ponds and 1,700 feet of river adjacent to the site were dredged to remove creosote and creosote-contaminated soils, and an underground clay wall was placed between the settling ponds and the river. Dredgings from the settling ponds were landfilled in a field east of the river and the ponds were backfilled with clean soil. River dredgings were spread and buried along the west bank of the river.

"The plant facilities were demolished in 1978. Some oil saturated soils (450 cubic yards) were excavated and shipped to the Nuclear Engineering Landfill in Sheffield, Illinois. Excavated areas were backfilled with clean fill material."

2.4 SITE CHARACTERISTICS

The creosote used at the Moss-American site was apparently a mixture of 50 percent coal tar creosote and 50 percent fuel oil. Chemical analyses of the specific creosote used at the site do not exist, but an interpretation of general constituents of creosote was presented in the U.S. EPA RI report.

The facility's characteristics of contamination, as interpreted by the U.S. EPA in the RI report, are described as follows:

"Coal tar creosote is a byproduct of the production of coke from coal. The 200 to 400°C fractions are distilled coal tar or creosote. Creosote is a mixture of single to multiple ring aromatic compounds.... The composition of creosote consists of neutral organic fractions such as polycyclic aromatic hydrocarbons (PAHs) and dibenzofuran. Tar acids, such as phenol and the cresols, as well as such tar bases as pyridenes, quinolines, and acridines, constitute a rather small percentage of the total weight of creosote.

"The primary potential organic contaminants of concern at the Moss-American sites are summarized in this [reference] in three groups: carcinogenic PAHs; noncarcinogenic PAHs; and benzene, ethylbenzene, toluene, and xylenes (BTXs). The carcinogenic PAH group contains the eight PAHs that have been ranked by the U.S. EPA Carcinogenic Assessment Group as class B or C carcinogens (see [RI report] Appendix K). The noncarcinogenic PAH group contains the nine other target PAH compounds. Table 3-2 [of the RI report] lists the organic compounds within each group. The BTX group represents the most common volatile organic compounds that are found as compounds of petroleum based fuels."

Industry literature, as compiled by the American Wood Preservers Association, present the following information pertaining to the general chemical composition of creosote:

> Most of the 200 or more compounds in creosote are polycyclic aromatic hydrocarbons. Only a limited number of them -- less than 20 -- are present in amounts greater than one percent. The major polycyclic aromatic hydrocarbons listed [on the next page] generally comprise at least 75 percent of the creosote.

Major Components in Creosote	Approximate Percent ±0.7%
Naphthalene	3.0
2-Methylnaphthalene	1.2
1-Methylnaphthalene	.9
Biphenyl	.8
Dimethylnaphthalenes	2.0
Acenaphthene	9.0
Dibenzofuran	5.0
Fluorene	10.0
Methylfluorenes	3.0
Phenanthrene	21.0
Anthracene	2.0
Carbazole	2.0
Methylphenanthrenes	3.0
Methylanthracenes	3.0
Fluoranthene	10.0
Pyrene	8.5
Benzofluorenes	2.0
Chrysene	3.0

The following description of Site contaminant characteristics is also according to the Moss-American RI Report (U.S. EPA, 9 January 1990) and is subject change based on the forthcoming scope of predesign phase extent of contamination tasks to be implemented at the Site:

Soils

"The extent of soil contamination within the former site boundary is shown on [RI report] Figure 3. The basis for the boundaries shown in Figure 3 is the concentration

of carcinogenic PAHs. Field observations and screening results were also used to determine the shape of the contours. Carcinogenic PAHs are shown because they are responsible for the risks associated with the site.

"The processing area and vicinity, the settling ponds, the treated storage areas (particularly the eastern edge), the northeast landfill, and the southeast landfill were identified as contaminated on the basis of the field screening results and analytical data. The most contaminated areas are the processing area (in the immediate vicinity of the old retorts), the eastern edge of the treated storage area, the northeast landfill, and the southeast landfill.

Groundwater

"The estimated lateral extent of groundwater contamination is shown in [RI report] Figure 4 along with a summary of the hydraulic characteristics of the aquifer. The shaded areas represent organic compounds detected in the groundwater samples. No inorganic contamination extends from the processing area to the river in a band that could be up to 400 feet wide. The shaded area on the map shows the maximum expected width of the band. The contaminated plume generally follows the groundwater gradient at the site, which is northeasterly [sic] toward the river.

"Groundwater contamination extends to a maximum depth of 20 feet below ground. No contaminants were detected in intermediate and deep wells at the facility. The lower extent of groundwater contamination is limited by the dense silty-clay till, which acts as a confining layer.

River Water

"Eight surface water samples were taken from the Little Menomonee River and from ditches on the site. No PAHs or other contaminants were detected in the river samples. PAHs in surface water were detected in the ditch that drains water from the site to the river. Oil from the former settling pond outfall appears to discharge to the river, producing an oily sheen on the river adjacent to the outfall during low flow conditions. During normal flow conditions, the discharge is either not noticeable or does not occur.

Sediment

"The compounds detected in the river sediment are consistent with those found onsite. The primary contaminants are PAHs. BTX compounds were not commonly found in the sediment samples. Other detected compounds were not widespread and were at low concentrations.

"The concentration of carcinogenic PAHs in sediment from the Little Menomonee River is shown in [RI report] Figure 5. The vertical axis in [RI report] Figure 5 represents the Little Menomonee River. Sample locations are shown relative to the major road crossings on the river. PAHs were detected along the entire reach from Brown Deer Road to the Menomonee River. In general, contaminant concentrations appear to decrease with distance from the site. In addition, contaminants were not detected in some samples, indicating an uneven contaminant distribution."

2.5 PROJECT OBJECTIVES

According to the Statement of Work for the Moss-American RD/RA (U.S. EPA, 1991):

> "The purpose of background sampling is to distinguish site-related contamination from naturally occurring levels (ambient), or other non-site-related levels of chemicals present in the environment due to human-made, non-site sources (anthropogenic)."

2.5.1 Specific Objectives

The specific objective of the study is to determine background concentrations with statistical rigor so that non-random and random factors can be considered at any location within the facility where an estimate of background concentration is required. The SOW specifies the objective of identifying "representative background sampling points for the sediments and soil." That is, non-random factors should be considered. The SOW also specifies the objective of calculating "maximum probable background concentration, which shall be calculated by the method identified in Appendix J of the FS or other current guidance in effect at the time the work is performed." That is, random factors should be considered.

2.5.2 Intended Data Usages

Background concentrations of CPAHs in soil and sediment will be used to assist in further determining cleanup standards. The SOW for RD/RA for the Moss-American Site (U.S.

EPA, 1991) identifies background CPAHs as a potential cleanup standard at the following locations:

- Northeast Landfill.
- Plant areas outside the floodplain.
- Plant areas inside the floodplain.
- Hotspots in the downstream floodplain.
- Soil disturbed during excavation of the new river.
- Portions of the riverbed that will not be relocated.
- The new river channel.

In each location, the cleanup standard is defined as a given numerical standard or background, whichever is greater.

The use of background concentrations for the cleanup standard will influence the subsequent phases of the project. Figure 2-2 illustrates the series of impacts arising from the use of background measurements.

2.5.3 Data Quality Objectives

Data quality objectives (DQOs) define and specify the quality of the data required to support the decisions of the remedial response activities. DQOs are determined based on the end use of the data to be collected. The data necessary to meet the required predesign Task 2 project objectives fall into a single category: defining background concentration of CPAHs in soil and sediments (background characterization). The target compounds which must be measured in determining background concentrations in sediments and soils are limited to eight CPAH compounds listed as follows:

- Benzo[a]anthracene.
- Chrysene.
- Benzo[b]fluoranthene.
- Benzo[k]fluoranthene.
- Benzo[a]pyrene.
- Indeno[1,2,3-cd]pyrene.
- Dibenz[a,h]anthracene.
- Benzo[g,h,i]perylene.

The rationale for limiting the background determination to only these eight compounds is provided by the Consent Decree requirement that specifies all cleanup objectives (for soil

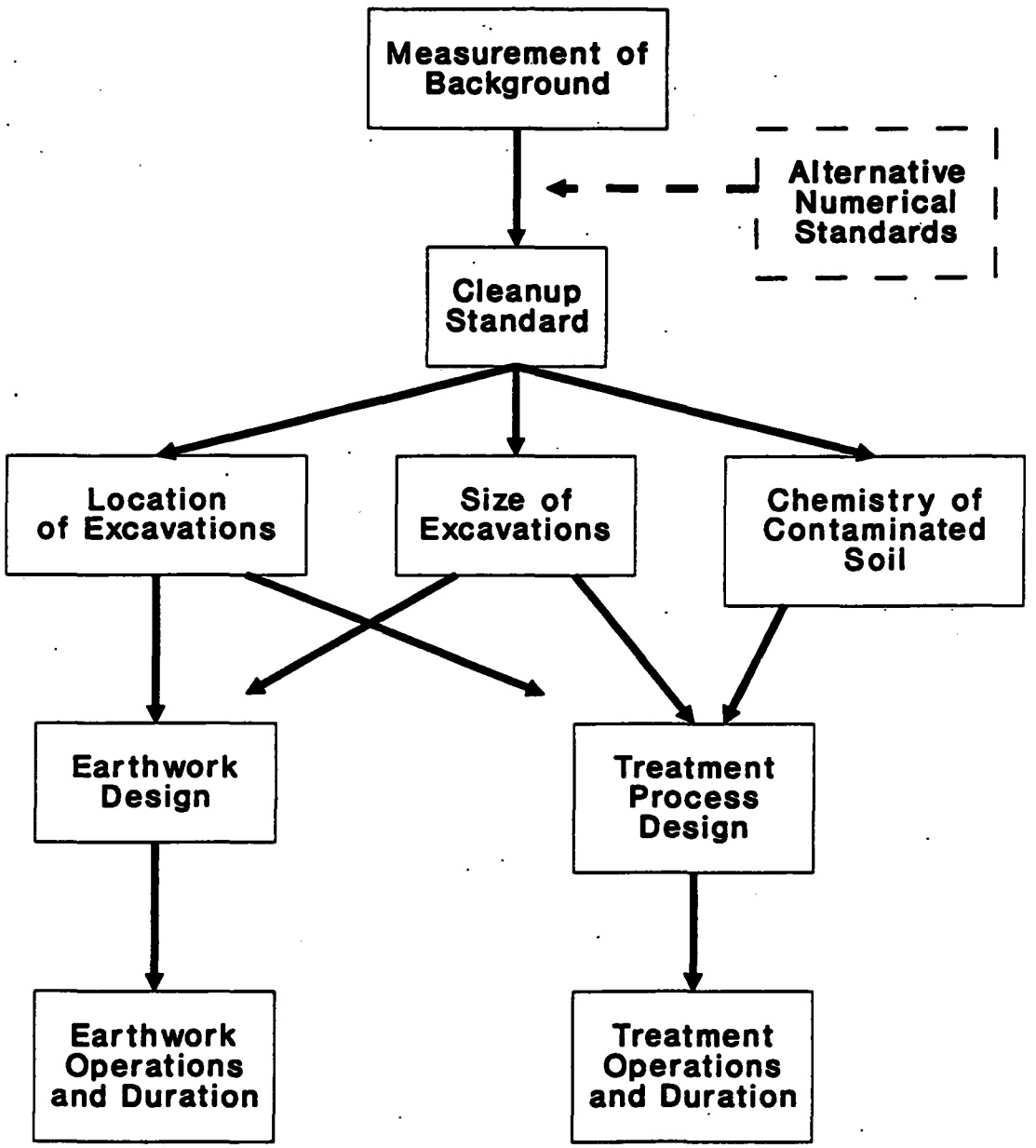

Figure 2-2

Consequences of Determination of Background

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and sediment) with respect to the sum of the eight CPAH compounds outlined above (i.e., total CPAHs).

Determining the appropriate analytical levels for data is an integral part of defining DQOs. There are five defined analytical levels:

- LEVEL I - Field screening. This level is characterized by the use of portable instruments which can provide real-time data to assist in the optimization of sampling point locations and for health and safety support. This level provides the lowest data quality but the most rapid results.

- LEVEL II - Field analysis. This level is characterized by the use of portable analytical instruments which can be used on site, or in mobile laboratories stationed near a site (close-support labs). Depending upon the types of contaminants, sample matrix, and personnel skills, qualitative and quantitative data can be obtained. This level provides rapid results and a better equality of data than in Level 1.

- LEVEL III - This level provides an intermediate level of data quality and is used for site characterization and in support of engineering studies using standard U.S. EPA-approved procedures. Engineering analyses may include mobile laboratory generated data and some analytical laboratory methods (e.g., laboratory data with quick turnaround used for screening purposes but without full quality control documentation).

- LEVEL IV - CLP RAS. This level provides the highest level of data quality and is characterized by rigorous QA/QC protocols and documentation and provides qualitative and quantitative analytical data. Some regions have obtained similar support via their own regional laboratories, university laboratories, or other commercial laboratories.

- LEVEL V - Non-standard methods. Analyses which may require method modification and/or development.

Analytical Level I will apply to readings generated during health and safety monitoring. Analytical Level V will apply to all analytical data generated from sample analyses. The data quality objectives for all associated data collection activities, data types, data uses, and other data quality control factors are summarized in Table 2-1. Table 8-1 presents contaminants of concern and associated method detection limits for the Moss-American Site

Predesign Task 2 activities. All health and safety issues associated with the field program for the Site will be addressed in the Site Health and Safety Plan.

2.6 SAMPLE NETWORK AND RATIONALE

The sampling network and rationale is addressed in Section 2 of the Field Sampling Plan (FSP) (Appendix A).

2.7 PROJECT SCHEDULE

The anticipated schedule for the Moss-American Site Predesign Task 2 activities associated with determining background concentrations of CPAHs in soils and sediments is presented in Figure 2-3.

SECTION 3

PROJECT ORGANIZATION AND RESPONSIBILITY

As outlined in the Consent Decree, KMCC will lead in developing and implementing the (RD/RA) work plan for the Moss-American Site. KMCC has contracted WESTON for the development of the predesign and remedial design technical documents and for the implementation of the interim and overall pre-design work plans. All activities will be performed in close coordination with U.S. EPA Region V and the Wisconsin Department of Natural Resources (WDNR).

All tasks that include monitoring and measurement activities and that generate or process analytical data related to environmental remedial cleanup objectives must have a QAPP. The QAPP will be prepared by WESTON and must be approved by the U.S. EPA Region V Remedial Project Manager (RPM) and the U.S. EPA Quality Assurance Officer (QAO). Environmental measurements will not be initiated until the QAPP has received the necessary approvals. The Moss-American site QAPP will be submitted to all persons concerned with obtaining and/or using the analytical data, the U.S. EPA Region V RPM, and WDNR. Key personnel responsibilities in four specific areas (project management, quality assurance, field operations, and laboratory operations) are discussed below. The organization chart is included as Figure 3-1.

3.1 PROJECT MANAGEMENT

The key operational responsibilities involving the execution and direct management of technical and administrative aspects of this project have been assigned as noted in the following subsections.

3.1.1 U.S. EPA Region V Remedial Project Manager

The U.S. EPA RPM for the Moss-American Site is Ms. Betty Lavis. The RPM has the overall responsibilities for all phases of the predesign and RD/RA activities. During Ms. Lavis's absence, Mr. Doug Ballotti, Unit Manager, will act on her behalf.

3.1.2 WDNR State Representative

The WDNR state representative is Mr. Gary Edelstein. His overall responsibility is to review project documents, monitor the progress of the Moss-American RD/RA activities,

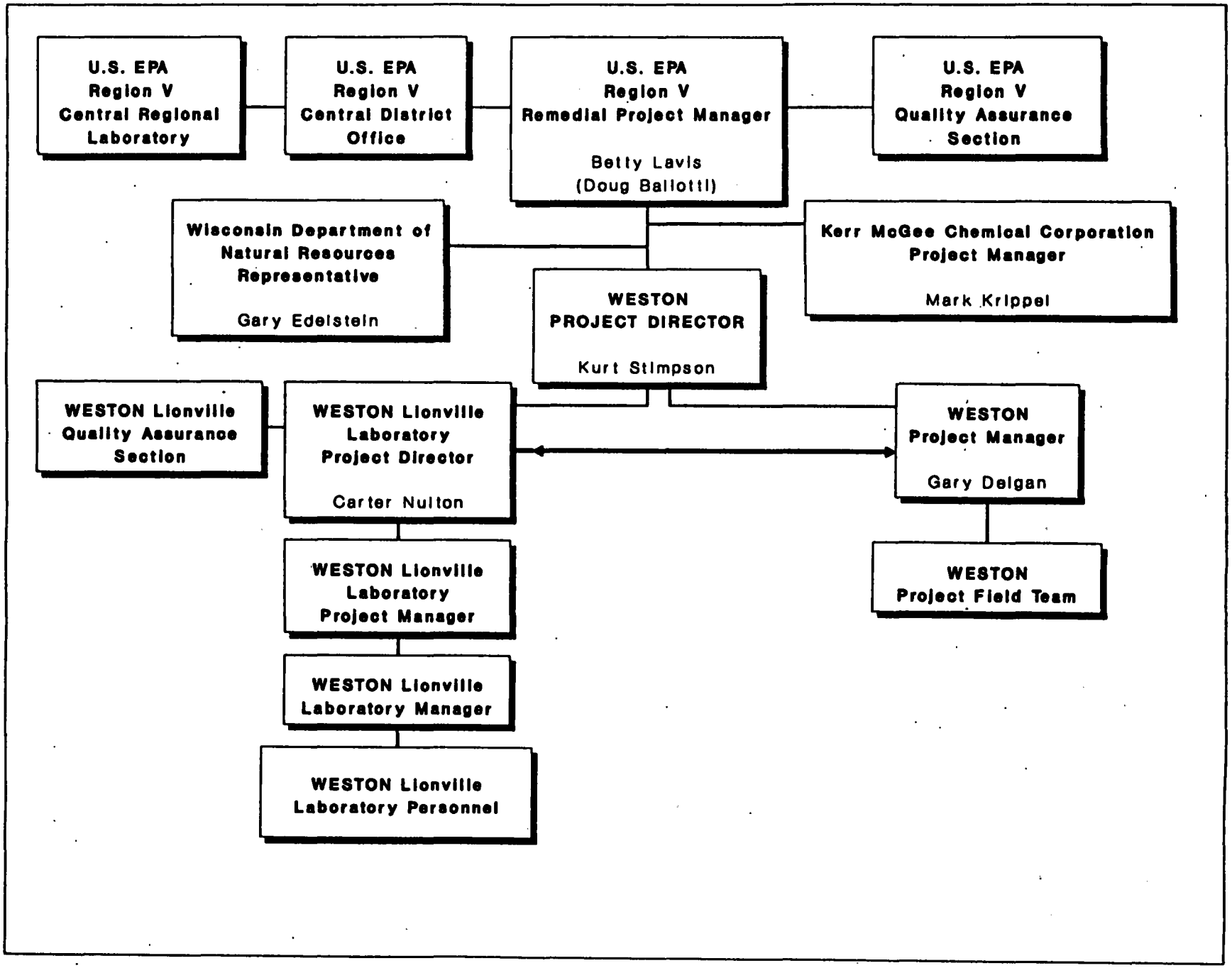


FIGURE 3-1

Project Organization Chart
Predesign Task 2 Activities Moss-American Site

and serve as a liaison between the state and U.S. EPA in order to ensure that all activities address state requirements and are executed in accordance with state regulations and/or project-specific agreements.

3.1.3 WESTON Project Director

The WESTON Project Director is Mr. Kurt Stimpson. The Project Director has overall responsibility for all site-related tasks performed under this QAPP. The Project Director is responsible for ensuring that the project meets U.S. EPA and KMCC objectives and quality standards. He is also responsible for ensuring that all work is executed in accordance with the U.S. EPA technical directives. The WESTON Project Director is responsible for assigning and monitoring the functions and responsibilities of the WESTON Project Manager.

3.1.4 Project Managers (WESTON and KMCC)

The KMCC Project Manager is Mr. Mark Krippel. The WESTON Project Manager for the Moss-American Site is Mr. Gary Deigan. The Project Managers are responsible for implementing the project, and have the authority to commit the resources necessary to meet the project objectives and requirements. A Project Manager's primary function is to ensure that the technical, financial, and scheduling objectives are achieved successfully. The WESTON Project Manager will coordinate with the WESTON Project Director, the U.S. EPA RPM, and WDNR state representative. His other responsibilities include:

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

3.2 QUALITY ASSURANCE

All aspects of the Moss-American Site investigation are subject to review and approval by U.S. EPA Region V and WESTON. The specific quality assurance tasks and responsibilities are summarized below:

3.2.1 Review/Approval of the QAPP

WESTON

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

U.S. EPA Region V

The U.S. EPA Region V Environmental Sciences Division (specifically, the Quality Assurance Section [QAS] and Central Regional Laboratory [CRL]) shall review the draft and revised QAPPs. They shall provide recommendations for approval to the U.S. EPA Region V RPM. In addition, the U.S. EPA Region V RPM shall review and approve the QAPP. The WDNR state representative will also be provided the opportunity to review and comment on the QAPP.

3.2.2 Validation of Analytical Data

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

3.2.3 Performance and Systems Audits

- External field audits of Moss-American Site activities are the responsibility of the U.S. EPA Region V CRL and/or Central District Office (CDO).
- Internal field audits are the primary responsibility of the WESTON Project Director and/or Project Manager.
- External laboratory audits will be performed by the U.S. EPA Region V CRL.
- Internal laboratory audits will be performed by the WESTON Project Manager or his designee.

3.2.4 Final Assessment of Quality Assurance Objectives

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

3.2.5 Evidence Audits of Field Records

- External evidence audits of field records are the responsibility of the U.S. EPA Region V CRL.

- Internal evidence audits of field records will be performed by the WESTON Project Manager or his designee.

3.2.6 Internal Quality Assurance Review and Approval of Reports, Standard Operating Procedures, and Field Activities

- The WESTON Project Director/Project Manager shall review all necessary reports and procedures which can impact the data quality for planned facility activities.

- The WESTON Project Director/Project Manager shall audit the implementation of the QA program (as outlined in the QAPP) to ensure conformance with WESTON, KMCC, U.S. EPA, and WDNR project requirements.

- The WESTON Field Team Leader shall report the status of the QA program to the WESTON Project Director/Project Manager on a regular basis.

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

3.2.7 Approval of Laboratory Analytical Procedures

- Externally, the U.S. EPA Region V QAS must review and approve analytical procedures.

- Internally, the KMCC Project Manager will review and approve analytical procedures.

3.3 FIELD OPERATIONS

The WESTON field team shall operate under the direction of the WESTON Project Manager when conducting field activities identified in this QAPP unless otherwise noted

herein. These activities include sample collection, field measurements, sample packaging, sample shipment, and sample/document chain-of-custody procedures. The field team shall be drawn from WESTON's pool of corporate resources. Field personnel assignments will be made prior to the commencement of sampling activities. Within the field team, there will be a minimum of three specific roles:

- Field Team Leader - responsible for the management of the field team and the supervision of all field activities in the absence of the WESTON Project Manager.

- Site Health and Safety Coordinator - responsible for the implementation of the Health and Safety Plan. Will perform Health and Safety monitoring and ensure compliance with all Health and Safety requirements for the Moss-American Site.

- Field Sample Manager/Custodian - has total custody of all samples from the time they are collected to when they are shipped. Is responsible for ensuring that all sample management handling and documentation procedures are implemented correctly.

To ensure the implementation of the "buddy system," there will be a minimum of two field personnel present at all times during sampling activities. Depending on the schedule for the field sampling activity, the WESTON Project Manager will evaluate the need for additional personnel. When necessary, the Field Team Leader may also perform in the capacity of the Site Health and Safety Coordinator. To the extent practicable, the Field Sample Manager will not be given any additional responsibilities other than field samples. All personnel will be deemed field samplers in order to ensure the full utilization of all personnel at all times. The field sampler(s) will execute collection of the samples and perform equipment decontamination. In the absence of the WESTON Project Manager, the Field Team Leader will be responsible for providing QA of field activities.

3.4 LABORATORY OPERATIONS

All laboratory analytical procedures for this subject predesign task shall be conducted by the WESTON Analytics Division Lionville Laboratory. The WESTON Project Manager shall initiate the scheduling of all analyses. He shall coordinate with the Field Team Leader in executing all follow-up laboratory arrangements. The organization and key responsibilities within the WESTON Lionville Laboratory are discussed in the following subsections.

3.4.1 Laboratory Project Director and Project Manager

WESTON recognizes the importance of efficient project management and quality control/quality assurance. In achieving this, the Analytics Division has established a Project Director/Project Manager group. This group is responsible for management of all analytical projects.

The laboratory Project Director is responsible for the overall direction of the project, and is the chief Quality Assurance Officer for the project. The Project Director is accountable for:

- Ensuring all resources of the laboratory are available for specific projects.
- Defining the level of excellence for the project performance and/or results.
- Assuring the preparation of a tailored, Project Technical Profile and/or QAPP, as necessary.
- Ensuring peer review of the adequacy of QAPPs.
- Ensuring allocation of proper quality control budgets.
- Attaining concurrence with department (e.g., laboratory) managers on performance and/or results objectives.
- Achieving acceptable project implementation performance.
- Approving the quality of the project results (e.g., data, reports).

The laboratory Project Managers are responsible for preparing the Project Technical Profile summarizing QA/QC requirements for the project, maintaining the laboratory schedule, ensuring that technical requirements are understood by the laboratory, and advising the Project Director and Laboratory Manager of all variances.

In general, project-specific QAPPs are not prepared by the laboratory. The laboratory Project Manager will provide technical guidance and the necessary laboratory-related information to the preparer, and provide peer review of the final document to ensure accuracy of the laboratory information.

3.4.2 Laboratory Manager

The ultimate responsibility for the generation of reliable laboratory data rests with the Laboratory Manager. The Laboratory Manger has the authority to effect those policies and procedures to ensure that only data of the highest attainable quality is produced. It is the Laboratory Manager's responsibility to see that all tasks performed in the laboratory are conducted according to the minimum requirements of this QAPP to ensure that the quality of service provided complies with the project's requirements.

The Laboratory Manager supports the QA Section which is not subordinate to or in charge of any person having direct responsibility for sampling and analysis, and that has additional reporting responsibilities to corporate QA.

The Laboratory Manager coordinates laboratory analyses, supervises in-house chain-of-custody procedures, schedules sample analyses, oversees preparation of analytical reports, and data review functions.

3.4.3 Laboratory Quality Assurance Personnel

The Laboratory Quality Assurance Personnel have responsibility for conducting and evaluating results from system audits. In addition, the preparation of standard operating procedures and quality assurance documentation for the laboratory shall be controlled by the QA Section. The QA Section will review program plans, as requested, for consistency with organizational and contractual requirements and will advise appropriate personnel. The QA personnel are responsible for establishing and implementing the laboratory QA plan. The QA Section will review 10 percent of the data packages.

3.4.4 Section Managers/Supervisors

To assist the Laboratory Manager in achieving his/her goals, the Laboratory Organic Section Manager and Unit Leaders are responsible for the implementation of established policies and procedures. They possess the authorities commensurate with their responsibilities for the day-to-day enforcement and monitoring of laboratory activities.

Section Managers have the responsibility for ensuring that their personnel are adequately trained to perform analyses; that equipment and instrumentation under their control is calibrated and functioning properly; and that system audits are performed regularly.

3.4.5 Report Section Manager

The Laboratory Report Section Manager is responsible for coordinating receipt of all data from the various service groups within the laboratory, reviewing data for compliance to laboratory QC criteria and/or criteria in the Project Technical Profile, and ensuring that data are reported in a timely manner and in the proper format.

3.4.6 Chemists/Technicians

Any effective laboratory quality assurance/quality control program depends on the entire organization, including management and every individual on the laboratory staff. The initial review for acceptability of analytical results rests with the analysts conducting the various tests. Observations made during the performance of an analytical method may indicate that the analytical system is not in control. Analysts must use quality control indicators to assure that the method is in control before reporting results.

3.4.7 Sample Log-In Personnel

Sample log-in personnel have the responsibilities to:

- Receive and inspect the incoming sample containers.
- Record the condition of the incoming sample containers on the chain of custody.
- Sign appropriate shipping and receiving documents.
- Verify chain of custody versus samples received.
- Notify laboratory section managers/supervisors of sample receipt and required analyses.
- Assign a unique identification number and customer account number, and enter each into the sample management system log.
- Control and monitor access/storage of samples and extracts.

SECTION 4

QUALITY ASSURANCE OBJECTIVE FOR MEASUREMENT DATA

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

4.1 LEVEL OF QUALITY CONTROL EFFORT

Field duplicate and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling program. Field duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples. One MS/MSD will be collected for every 20 or fewer investigative samples for each matrix (i.e. soil and sediment). MS/MSD samples are designated/collected for organic analyses only. The U.S. EPA Region V CRL discourages the use of aqueous field blanks for soil and/or sediment samples. Therefore, no field blanks will be collected during Moss-American Site predesign background sampling activities. One field duplicate will be collected for every 10 or fewer investigative samples for each matrix.

MS/MSD samples are investigative samples. Soil and sediment MS/MSD samples require no extra volume for extractable organics. Table 4-1 contains a summary of the overall level of QC effort for the Moss-American Site sampling activities. Sampling procedures are specified in the Field Sampling Plan (FSP) (Appendix A).

The level of QC effort provided by the WESTON Lionville Laboratory during the testing of Moss-American Site soils and sediments for CPAHs by capillary column Gas Chromatography/Mass Spectroscopy (Selected Ion Monitor) [GC/MS (SIMS)] techniques, will conform to the protocols in U.S. EPA SW846, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," 3rd Edition, Method 8270, modified for this project. (Appendix B).

Table 4-1

Summary of Background Sampling Effort
Moss-American Site
Milwaukee, Wisconsin

Sample Matrix	Laboratory Parameters	Investigative			Field Duplicate			MS/MSD[a]			Matrix Total[b]
		No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	
SOIL											
Phase I											
Background Soil	Low DL CPAH[c]	45	1	45	5	1	5	3	1	3	50
Phase II											
Background Soil	Low DL CPAH[c]	30	1	30	3	1	3	2	1	2	33
SEDIMENT											
Phase I											
Background Sediment	Low DL CPAH[c]	15	1	15	2	1	2	1	1	1	17
Phase II											
Background Sediment	Low DL CPAH[c]	40	1	40	4	1	4	2	1	2	44

Notes:

[a]MS/MSD samples are not additional samples, but instead investigative samples assigned for MS/MSD analysis. No extra volume will be collected for MS/MSD samples.

[b]Matrix totals do not include matrix spike/matrix spike duplicate samples.

[c]The SOP for low detection limit (DL) carcinogenic PAH analysis is presented in Appendix B.

4.2 ACCURACY, PRECISION, AND SENSITIVITY OF ANALYSIS

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

The standard operating procedure (SOP) for PAHs is provided in Appendix B. As part of the scope of work for this project, precision, accuracy, and method detection limits (MDLs) were determined via a MDL study. The laboratory followed U.S. EPA guidance for conducting MDL studies and provided a MDL study report. This report is also presented in Appendix B.

Sensitivity

The sensitivity for the CPAH analyses will be the achievable detection limits. Table 8-1 in Section 8 presents the MDLs for each contaminant of concern as determined from the MDL study.

Precision

In general, precision is the level of agreement among repeated independent measurements of the same characteristic, usually under a prescribed set of conditions (e.g., under the same analytical protocols). The most commonly used estimates of precision are the relative percent difference (RPD) for when only two measurements are available, and the percent relative standard deviation (% RSD) for when three or more measurements are available.

Precision of laboratory analysis will be assessed by comparing the analytical results between matrix spike and matrix spike duplicate samples. The relative percent difference (RPD) will be calculated for each target analyte pair. For the Moss-American site project, a goal of 50 percent will be targeted for precision criteria. Outliers for RPD will be evaluated and flagged on a case by case basis.

Accuracy

Accuracy is the degree of agreement of the analytical measurement with the true or expected concentration. When applied to a set of observed values, accuracy will be a combination of a random component and of a systematic error (or bias) component.

Analytical accuracy is expressed as the percent recovery of an analyte which has been used to fortify an investigative sample or a standard matrix (e.g., blank soil, analyte-free water, etc.) at a known concentration prior to analysis. See Section 13.2.2 for calculation of percent recovery.

The fortified concentration will be at 50 ng/g. Fortified standard matrices prepared in the laboratory are referenced as a blank spike, while fortified field (i.e., investigative) samples are referenced as matrix spikes.

For this project, all eight target analytes will be used as matrix spike compounds. QC limits for recovery will be 50 to 150 percent.

Recovery outliers will be evaluated on a case by case basis. If it is determined that the outliers are a result of lab error, the sample batch will be re-extracted and re-analyzed.

4.3 COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

Completeness

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

$$\text{Completeness (\%)} = \frac{\text{(number of valid data)}}{\text{(number of sample collected for each parameter analyzed)}} \times 100$$

If the percent completeness for the project is calculated to be below the QC acceptance criteria of 95 percent, the WESTON PM and PD, the KMCC PM, the U.S. EPA RPM, and WDNR representative will be notified. They will evaluate the overall impact on the project and the ability of the analytical data to meet project objectives, and determine what (if any) corrective action measures are required.

Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling network was designed to provide data representative of site conditions. During development of this network, consideration was given to past waste disposal practices, existing analytical data (if any), physical setting and processes, and constraints inherent to the Moss-American Site. The rationale of the sampling network is discussed in the FSP (Appendix A). Representativeness will be satisfied by ensuring that the FSP is followed, proper sampling technique are used, proper analytical procedure are followed and holding times of the samples are not exceeded in the laboratory. Representativeness will be assessed by the analysis of field duplicated samples.

Comparability

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

SECTION 5

SAMPLING PROCEDURES

Sampling procedures are described in the Field Sampling Plan (Appendix A).

SECTION 6

SAMPLE CUSTODY

It is U.S. EPA 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-DDI-R, Revised June 1985. This custody is in three parts: sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

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

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

6.1 FIELD CHAIN-OF-CUSTODY PROCEDURES

The key requirements for ensuring field chain of custody are summarized in this section. The specifics of sample handling and completion of sample documentation forms are detailed in Section 5 of the FSP (Appendix A).

6.1.1 Field Procedures

The field sampler is personally responsible for the care and custody of the samples until they are transferred to the Field Sample Manager and/or properly dispatched. As few people as possible should handle the samples.

All bottles will be labelled with a project sample number. The sample labels will 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 label because the indelible ink marker ballpoint pen would not function in freezing weather.

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

6.1.2 Field Logbooks/Documentation

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

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

The title page of each logbook will contain the following:

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

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

Measurements made and samples collected will be recorded. All entries will be made in ink (weather permitting) and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the station shall be recorded. The number of the photographs taken of the station, if any, will also be noted.

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

6.1.3 Transfer of Custody and Shipment Procedures

All samples will be recorded on a WESTON Analytics Division chain-of-custody form (Figure 6-1) under a unique project sample number. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the chain-of-custody form. This record documents transfer of custody of samples from the sampler to another person (such as the Field Sample Manager).

All sample shipment containers will be accompanied by the Chain-of-Custody Record identifying the contents. The WESTON chain-of-custody forms have six copies. The last copy (the yellow sheet) will be retained by the Field Sample Manager and the remaining five copies will accompany the shipment to the laboratory.

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. All shipment coolers will have two pre-numbered chain-of-custody seals placed on the outside of each cooler following closure of the cooler. Sample cooler packaging and shipment protocols are presented in Section 5.2 of the FSP. Figures 6-2 and 6-3 show examples of the WESTON Lionville Laboratory chain-of-custody seals and sample container labels.

6.1.4 Summary of Field Chain-of-Custody Procedures

The WESTON field team will consist mainly of the following:

- The Field Team Leader.
- The Site Health and Safety Coordinator.
- The Field Sample Manager/Custodian.

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

Custody Transfer Record/Lab Work Request

WESTON

WESTON Analytics Use Only

Client _____

Work Order _____

Date Rec'd. _____ Date Due _____

RFW Contact _____

Client Contact/Phone _____

WA Use Only Lab ID	Client ID/Description	Matrix	Date Collected	Refrigerator#									
				#/Type Container									
				Volume									
				Preservative									
				ANALYSES REQUESTED									

Matrix:
W - Water
S - Soil
SE - Sediment
SO - Solid
O - Oil
A - Air
WI - Wipe
DS - Drum Solids
DL - Drum Liquids
F - Fish
L - EP/TCLP Leachate
X - Other

Special Instructions:

Item/Reason	Relinquished by	Received by	Date	Time	Item/Reason	Relinquished by	Received by	Date	Time

WESTON Analytics Use Only

Samples Were:
1 Shipped or Hand-Delivered
NOTES:

2 Ambient or Chilled
NOTES:

3 Received Broken/Leaking (Improperly Sealed)
Y N
NOTES:

4 Properly Preserved
Y N
NOTES:

5 Received Within Holding Times
Y N
NOTES:

COC Tape Was:
1 Present on Outer Package Y N
2 Unbroken on Outer Package Y N
3 Present on Sample Y N
4 Unbroken on Sample Y N
NOTES:

COC Record Was:
1 Present Upon Receipt of Samples Y N

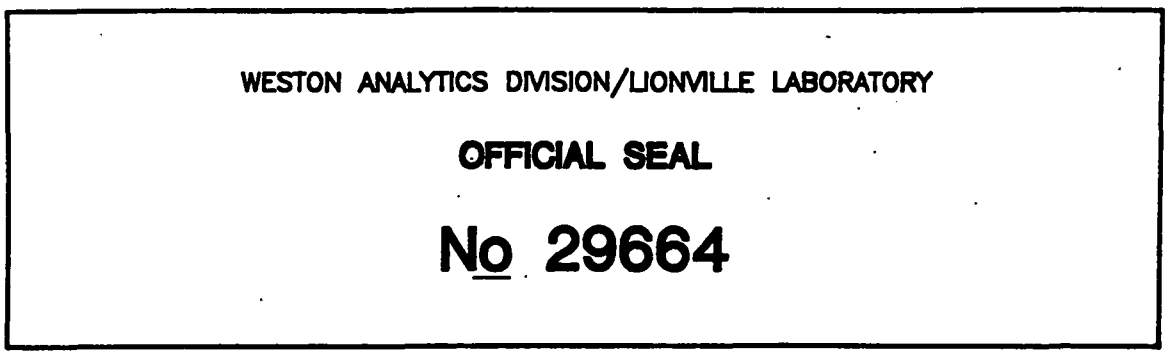
Discrepancies Between Sample Labels and COC Record? Y N
NOTES:

WESTON MANAGERS DESIGNERS/CONSULTANTS

Three Hawthorn Parkway
Vernon Hills, Illinois
60061

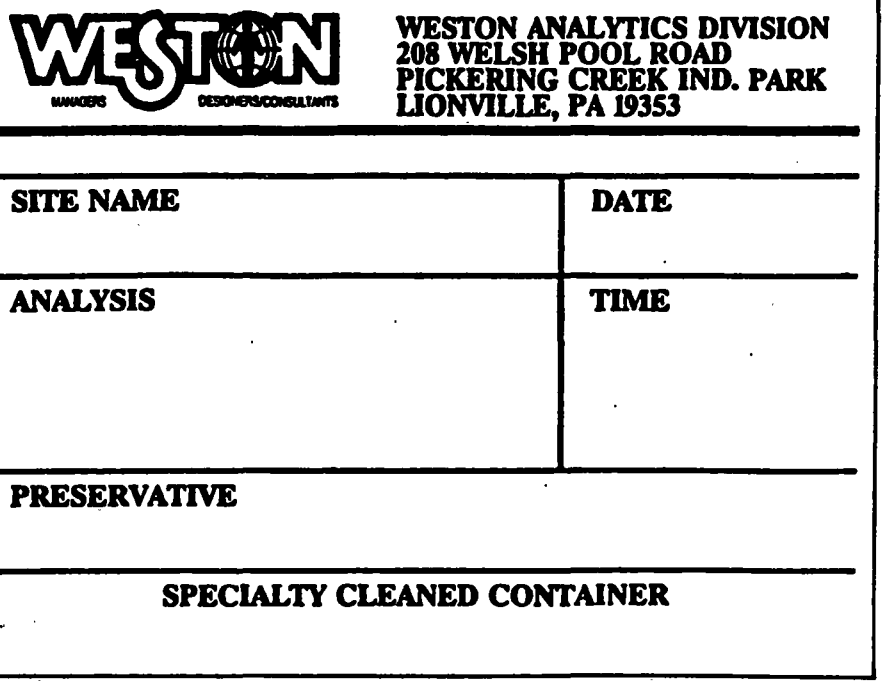
FIGURE 6-1

CHAIN-OF-CUSTODY FORM



WESTON MANAGERS DESIGNERS/CONSULTANTS	Three Hawthorn Parkway Vernon Hills, Illinois 60061	FIGURE 6-2	EXAMPLE OF A WESTON LIONVILLE LABORATORY CHAIN - OF - CUSTODY SEAL MOSS - AMERICAN SITE Milwaukee, Wisconsin

15492 A



WESTON MANAGERS DESIGNERS/CONSULTANTS	Three Hawthorn Parkway Vernon Hills, Illinois 60061	FIGURE 6-3	EXAMPLE OF A WESTON LIONVILLE LABORATORY SAMPLE CONTAINER LABEL MOSS – AMERICAN SITE Milwaukee, Wisconsin

15492 A

The Field Team Leader will have overall responsibility for ensuring the completion of all field activities in accordance with procedures described in this document. The Field Team Leader is the overall coordinator of sampling activities at the site and is the communication link between field team members and the WESTON Project Manager. The Field Team Leader will assign specific field duties to the team members based on input from the WESTON Project Manager.

The Field Sample Manager will be responsible for preparing (and reviewing for accuracy and completeness) all sample paperwork such as chain-of-custody forms, sample labels, and any other paperwork that is required for sample documentation. The Field Sample Manager will also prepare all sample shipment documentation such as airbills. If the Field Sample Manager requests assistance from other members of the field team in completing sample paperwork, the Field Sample Manager will be responsible for reviewing and ensuring the accuracy and completeness of this paperwork before he/she encloses it in the sample shipment container. All members of the field team may be involved in the actual sample packaging and shipment. The Field Sample Manager is responsible for tracking all sample paperwork from the time of receipt until the completed paperwork copies are given to the WESTON Project Manager.

The Field Team Leader is responsible for maintaining the site logbook. The site logbook will contain notes made by the Field Team Leader on site activities, which will include the tracking of the samples from the time of sample collection to the delivery of the samples to the shipping carrier. The names and function of all field team members will be listed in the logbook. During the course of sample collection activities, the Field Team Leader will document in the logbook the times and dates of all sampling activities (e.g., who collected the sample(s), when the sample(s) was collected, who delivered the samples to Field Sample Manager, when the sample coolers were delivered to the shipping carrier, etc.) If the Field Sample Manager was part of the sampling team this will be specifically noted.

The Field Team Leader will note the names of the actual samplers for each station location along with the time, date, station location identifier and sample identifiers, etc.

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

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

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

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

6.2 LABORATORY CHAIN-OF-CUSTODY PROCEDURES

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

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

A primary step in the evidentiary trail is to provide proof that the sample collected in the field is the sample that was actually analyzed. The chain-of-custody forms for field and laboratory, when properly completed, provide the necessary information.

In addition to providing accountability for the physical location of the sample, sample integrity is dependent on proper collection and storage of the sample. Description of chain-of-custody procedures associated with sample collection, receipt, storage, preparation, analysis, and general security procedures are described in subsequent sections of this chapter.

The area supervisors are responsible for the records received or generated by their respective areas at the laboratory. Laboratory documentation used to establish chain-of-custody and sample identification may include the following:

- Field chain-of-custody forms or other paperwork which arrives with the sample.

- Custody Transfer Record/Laboratory Work Request also referred to as the field/laboratory chain-of-custody form.

- Sample labels or tags attached to each sample container that may contain the following information: sample date; time (2400 clock); sample description ; sample matrix; sample temperature upon receipt; filtration, preservation, and known hazards information; sample management (disposal); project sample number; and parameter group. These labels/tags are verified for accuracy against the paperwork received with the samples. The signed chain-of-custody form will serve as documentation of this verification, rather than attempt to peel or remove tags/labels to place in the written documentation file.

- Custody seals attached to shipment containers. Custody seals will prevent the container from being opened without authorization. The intact condition of the custody seals will serve as documentation that the shipment container was not tampered with after having left the custody of the Field Sample Manager. This will be noted on the chain-of-custody form by the laboratory sample custodian upon receipt at the laboratory.

- Sample preparation logs, (i.e., extraction and digestion information recorded in hard-bound laboratory books that are filled out in legible handwriting, and signed and dated by the chemist).

- Sample analysis logs, (e.g., metals, GC/MS, etc. information recorded in hard-bound laboratory books that are filled out in legible handwriting, and signed and dated by the chemist).

- Sample storage log (same as the laboratory chain of custody).

6.2.1 Sample Receipt

A designated laboratory sample custodian is responsible for samples received at WESTON. In addition to receiving samples, the sample custodian is also responsible for documentation of sample receipt, storage before and after sample analysis, and documentation of eventual proper disposal of samples. Upon receipt, the sample custodian will:

- Inspect the sample container for integrity. The presence of leaking or broken containers will be noted on the chain-of-custody form (Figure 6-1). The sample custodian will sign (with date and time of receipt) the chain-of-custody form, thus assuming custody of the samples. If chain-of-custody forms are not included, the sample custodian will initiate these forms.The sample custodian will inform the laboratory Project Director and/or Laboratory Manager of the missing documentation. Corrective action procedures will determine future action associated with the samples.

- Coordinate sample bottle information (e.g., sample tag/label, etc.), logbook information, chain-of-custody records, and all pertinent information associated with the sample to verify sample identity and to assure that all information is correct. Any inconsistencies will be resolved with the field sampling representative and corrective action specified before sample analysis proceeds.

- Assign a unique WESTON batch number to each sample received. The WESTON batch number will be recorded on the chain of custody and on the bottle labels using a permanent marker. The WESTON batch number is a tracking number that is the primary means of tracking a sample through the laboratory. Samples are logged into a hard-bound sample logbook by documenting appropriate information.

- Move the samples to one of the locked sample storage refrigerators (maintained at 4° ±2° C) for storage prior to analysis. The storage location will be recorded on the chain-of-custody form.

- Maintain the original of the chain-of-custody form in the sample log-in area. Copies of the chain of custody are provided to the laboratory Report Manager, to each laboratory Section Manager, to the respective Unit Leaders, to the Project Manager, and to the QA Section.

- Alert appropriate production unit of any analyses requiring immediate attention due to short holding times.

- Log the sample information into the Laboratory Information Management System (LIMS). These data include laboratory number, field sample number, dates collected and received, project or client identification, and parameters to be analyzed.

6.2.2 Laboratory Sample Storage

Samples will be maintained in storage in one of the locked storage refrigerators prior to sample preparation and analysis. The SOPs for sample storage are summarized below.

Storage refrigerators are maintained at 4° ±2° C. The temperature is monitored by the laboratory security system and is additionally recorded daily in a bound logbook by the QA Section. During working hours, if equipment failure (compressor failure, door left open, etc.) results in the temperature of the storage refrigerator exceeding the upper or lower control limits, an audible alarm will sound and the samples will be moved to suitably controlled storage until the problem has been corrected. During off working hours, the alarm is automatically transferred to the security agency who alerts (via beeper call) laboratory and maintenance personnel so that prompt corrective action can be taken.

Refrigerator storage is designed to segregate samples to prevent cross-contamination and to prevent sample mix-up. This includes storage of volatiles samples separate from semivolatiles and inorganics samples. Within the refrigerators, samples are stored by WESTON batch number for easy retrieval.

Access to laboratory facilities is restricted to laboratory personnel or escorted guests. Therefore, once custody transfer to the laboratory has been completed, the sample is considered placed and stored in a designated secure area accessible only to authorized personnel (i.e., the laboratory facility). At this point, no further custody transfer documentation is required until sample disposal.

Stricter custody procedures which account for sample transfers from storage to analyst and vice versa within the laboratory are required for some projects. Generally, data for these projects will be used for litigation purposes. The samples are stored in a locked walk-in refrigerator, and the key is securely kept by the sample custodian. When the samples are relinquished to an analyst, both the analyst and the sample custodian are required to sign and date the appropriate lines on the laboratory chain-of-custody form (also described as the Custody Transfer Record/Laboratory Work Request Form). When the samples are returned to the appropriate cooler, both parties must again sign the original chain-of-custody form. All samples at the Lionville facility will be maintained at this level of custody.

6.2.3 Laboratory Sample Tracking

The SOPs for laboratory tracking are summarized in this section.

Organic Preparation/Analysis

Samples are received by the Organic Sample Preparation Section for extraction prior to analysis by gas chromatography, GC/MS, or liquid chromatography. All pertinent data are recorded in a bound laboratory notebook, and assigned a preparation batch number. This extraction information is transferred to the LIMS and a hard-copy Sample Extraction Record is generated. A copy of this form is shown in Figure 6-4. The original is placed on the facing page of the laboratory notebook where extraction data have been entered and is used for custody transfer documentation to the analyst. Copies are provided to the analyst to inform them that extracts are ready for analysis.

6.2.4 Sample Disposition

All samples will be held a minimum of 60 days after the data report is submitted to the client. Samples may be held longer due to special requests or specific contract requirements. All hazardous samples will be disposed of commercially or returned to the client.

When samples are transferred from the laboratory to any other destination, chain-of-custody protocols are followed.

6.2.5 Laboratory Recordkeeping

Data related to sample manipulation/preparation/analysis procedures and observations will be documented by the analyst/technician in the sample extraction log, sample digestion log,

SAMPLE EXTRACTION RECORD

Sheet no.: 1

Extract. Date: 03/20/91 Extraction Batch No: 91LE0560 Analyst: JS Method: CONT

Test: 0625 Cleanup Date: Analyst: Client: AAA COMPANY, INC

LIMS Report Date: 04/01/91 Solvent: DCM Adsorbent:

Sample No:	Client Name / Client ID	pH	Initial WT/VOL	Surr. Mult.	Spike Mult.	Final VOL BASE	Final VOL ACID	Split Mult.	GPC Y/N	% Solids	C/D FACTOR
9103L344-	AAA COMPANY, INC										
005 T	ES-1	7	100	1.0		1	1	1.0	N		10.0
005 TR	ES-1	7	100	1.0		1	1	1.0	N		10.0
005 TS	ES-1	7	100	1.0	1.0	1	1	1.0	N		10.0
006 T	ES-2	7	100	1.0		1	1	1.0	N		10.0
007 T	ES-3	7	100	1.0		1	1	1.0	N		10.0
008 T	ES-4	7	100	1.0		1	1	1.0	N		10.0
9103L353-	CANTWAIT ENVIRONMENTAL										
004 T	8013	7	100	1.0		1	1	1.0	N		10.0
004 TR	8013	7	100	1.0		1	1	1.0	N		10.0
004 TS	8013	7	100	1.0	1.0	1	1	1.0	N		10.0
91LE0560-MB1 T		7	1000	1.0		1	1	1.0	N		1.0
91LE0560-MB1 TS		7	1000	1.0	1.0	1	1	1.0	N		1.0
91LT0038-LB1 T		7	100	1.0		1	1	1.0	N		10.0

Comments:
Surrogate: 500 UL ESU 27K @ 100/200 UG/ML
Spike: 500 UL TCLP SPIKE @ 100/200 UG/ML

Extracts Transferred	Relinquished By	Date Time	Received By	Date Time	Reason for Transfer

WESTON MANAGERS DESIGNERS/CONSULTANTS

Three Hawthorn Parkway
Vernon Hills, Illinois
60061

FIGURE 6-4

SAMPLE EXTRACTION RECORD FORM
WESTON LIONVILLE LABORATORY

sample distillation log, analysis log, or the technician's personal logbook. These are hard-bound notebooks which are issued by the Laboratory Quality Assurance Section. laboratory notebook pages are signed an dated daily by laboratory analysts. Corrections to notebook entries are made by drawing a single line through the erroneous entry and writing the correct entry next to the one crossed out. A reason for the correction will be noted, as appropriate. All corrections are initiated and dated by the analyst.

6.2.6 Laboratory Building Security

The WESTON Lionville Laboratory maintains controlled building access at all times. All non-WESTON laboratory personnel are required to sign in at the receptionist's desk and are escorted by laboratory personnel while in the building.

The laboratory is locked at all times and monitored by an ADT Security System, unless a receptionist is present to monitor building access (e.g., between the hours of 8:00 a.m. and 5:00 p.m., Monday through Friday at designated facilities). This security system not only monitors building access, but also monitors the temperature in the sample storage refrigerators. If the control temperature range is exceeded during working hours, an audible alarm sounds. During nonworking hours, a silent alarm alerts ADT. Response by laboratory personnel is described below.

The locked building is accessed by laboratory employees by using a card key. Additionally, a passcode for the Building Security System may be required if no other employees are in the building.

Any breach of security during nonworking hours releases a silent alarm to the security agency who alert the local law enforcement agency and one of three laboratory personnel via beeper call. Police response to security alarms takes place within 5 minutes and laboratory personnel are on-site within 20 minutes.

6.3 FINAL EVIDENCE FILES CUSTODY PROCEDURES

WESTON is the custodian of the evidence file and will maintain the contents of the evidence files for all Moss-American Site activities. The content of the evidence file will include all relevant records, reports, correspondence, logs, field logbooks, laboratory sample preparation and analyses logbooks, data packages, pictures, chain-of-custody records/forms, data review reports, etc.

The WESTON office evidence files will be under the custody of the WESTON Project Manager in the WESTON Vernon Hills, Illinois office in a secured, limited access area.

The WESTON Lionville Laboratory will also maintain an evidence file for analytical and related data that are generated. The file will be managed in the following manner:

- All raw data such as hard-bound laboratory notebooks and logbooks, strip charts and instrument printouts, LOTUS spreadsheets, and magnetic tapes are to be retained for a minimum of five years. All raw data and final reports are documented and stored in a manner which is easily retrievable.

- All hard-bound laboratory notebooks and logbooks are assigned a book number by the QA Section. A new book will be assigned for each instrument or parameter as the most current book is completed.

- Instrument printouts and strip charts for the GC, HPLC, and GC/MS groups are stored in file cabinets in each specific laboratory area. Older documents are stored by date of analysis in WESTON's secure archives area.

- Final sample reports are filed alphabetically by client for future reference. After one year, these records are transferred to WESTON's secure archives area, and kept on file for a minimum period of five years, unless otherwise specified.

SECTION 7

CALIBRATION PROCEDURES AND FREQUENCY

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

7.1 FIELD INSTRUMENTS AND EQUIPMENT

Instruments and equipment used to gather, generate, or measure environmental data must be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with manufacturer's specification. During background soil and sediment sampling activities at the Moss-American Site, no field instruments or equipment will be used to generate environmental data. An organic vapor flame ionization detector (FID) and/or a photoionization detector (PID) may be used for health and safety monitoring purposes only.

7.2 CALIBRATION PROCEDURES FOR LABORATORY INSTRUMENTS

All instruments must be calibrated prior to use as a measurement device to establish the instrumental response to known reference materials. The manner in which various instruments are calibrated is dependent on the particular type of instrument and its intended use. All sample measurements are made within the calibrated range of the instrument. Preparation of all reference materials used for calibration will be documented in a standards preparation notebook.

Instrument calibration typically consists of two types, initial calibration and continuing calibration. Initial calibration procedures establish the calibration range of the instrument and determine instrument response over that range. Typically, three to five analyte concentrations are used to establish instrument response over a concentration range. The instrument response over the range is generally absorbance, peak height, etc., which can be expressed as a linear model with a correlation coefficient (e.g., for atomic absorption, inductively coupled plasma, UV-visible-infrared spectrophotometry, ion chromatography) or as a response factor or amount vs. response plot (e.g., for gas chromatography, gas chromatography/mass spectrometry, high performance liquid chromatography).

Continuing calibration usually includes measurements of the instrument response to fewer calibration standards and requires instrument response to compare with certain limits (e.g., ±10 percent) of the initial measured instrument response. Continuing calibration may be used within an analytical sequence to verify stable calibration throughout the sequence, and/or to demonstrate that instrument response did not drift during a period of nonuse of the instrument.

Specific instrument calibration procedures are summarized below.

Gas Chromatography/Mass Spectrometry

All GC/MS instrumentation is calibrated to set specifications prior to sample analysis. These specifications vary depending on the requirements of the analytical program and the designated analytical method.

Tuning and GC/MS Mass Calibration

The mass spectrometer will be calibrated with perfluorophenanthrene (FC 5311) as required to ensure correct mass assignment. Each work shift samples will be analyzed within a 12-hour period initiated by the injection of either an initial calibration or a continuing calibration solution.

GC/MS - Initial Calibration

After an instrument has been mass calibrated, initial calibration curves for analytes appropriate to the analyses to be performed are generated for five solutions containing known concentrations of authentic standards of compounds of concern. These solutions are generally cocktails of the method target analytes. The calibration curves will bracket the anticipated working range of analyses.

Linearity is verified by evaluating the response factors (RF) for the initial calibration standards. All compounds must have a % RSD of ≤25 percent.

Once an acceptable calibration is obtained, samples may be analyzed within a 12-hour period. At that time, the instrument must meet continuing calibration criteria prior to further analysis. A continuing calibration standard may be analyzed in lieu of a full five-point calibration if the specific criteria are met (see next page). Otherwise, a five-point curve must be re-established.

Calibration data, to include linearity verification, will be maintained in the laboratory's permanent records of instrument calibrations.

GC/MS - Continuing Calibration

During each operating shift, a single calibration standard may be analyzed to verify that the instrument responses are still within the initial calibration determinations. The response factor for each target compound in the daily standard is calculated and recorded, then compared to the average RF from the initial calibration. For the Moss-American Site Predesign Task 2 analyses, calibration standards will be prepared as discussed in Appendix B of the SOP. The SOP, Section 8.4, contains additional information regarding the frequency of continuing calibration.

If significant (>30 percent deviation) RF drift is observed for any analyte, appropriate corrective actions will be taken to restore confidence in the instrumental measurements. If criteria cannot be met, an acceptable five-point initial calibration must be re-established.

SECTION 8

ANALYTICAL PROCEDURES

8.1 Laboratory Analytical Procedures

All soil and sediment samples collected from the Moss-American Site during interim predesign field activities will be analyzed by the WESTON Analytics Division, Lionville Laboratory. All samples will be analyzed for the eight CPAH compounds (contaminants of concern) presented in Table 8-1. The analytical procedure is:

> U.S. EPA Method 8270 - GC/MS Technique, Modified for Low Detection Limits using SIMS (Appendix B).

This method is referenced from the U.S. EPA SW 846, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," 3rd Edition.

The method has been selected in order to achieve low detection limits for the eight CPAH compounds of interest, as necessary for the determination of background CPAH concentrations in soil and sediment. As part of the evaluation of this analytical method for the project, a method detection limit study was conducted by the WESTON Lionville Laboratory in October 1991. The method detection limit study confirmed that the modified Method 8270 can be utilized to analyze for low level CPAHs. The results of the method detection limit study and the modified Method 8270 standard operating procedure (SOP) are herein presented as Appendix B. The method detection limit for each contaminant of concern (as determined from the method detection limit study) is also presented in Table 8-1.

The modified Method 8270 is designed for samples with total CPAH levels below 100 parts per billion (ppb). If during the analysis of Phase I area background soil and/or sediment samples exhibit CPAH concentrations above 100 ppb, the modified Method 8270 will continue to be used following the dilution of the sample extract into the calibration range. This approach will enable the original sample size of 10 grams to be used thereby maintaining the representativeness of the sample. The U.S. EPA RI report data suggests that background concentrations of CPAHs may exhibit both ppb and parts per million (ppm) ranges. If the analytical results from the Phase I program indicate that the area background soils and/or sediments exhibit total CPAH levels above 100 ppb, the analytical methods shown below may be utilized during Phase II, if the second phase is implemented. The

Table 8-1

Carcinogenic Polycyclic Aromatic Hydrocarbons
Contaminants of Concern and Method Detection Limits
Moss-American Site

Compound	Method Detection Limit (ng/g)*
Benzo (a) Anthracene	0.26
Benzo (b) Fluoranthene	0.40
Benzo (k) Fluoranthene	0.83
Benzo (g,h,i) Perylene	0.16
Benzo (a) Pyrene	0.19
Chrysene	0.28
Dibenz (a,h) Anthracene	0.44
Indeno (1,2,3-cd) Pyrene	0.40

* MDLS (ng/g) are based on the extraction of a 10 g sample. See Appendix B for the MDL study report and the modified Method 8270 SOP.

implementation of these methods will occur following the development and approval of a QAPP addendum.

The two candidate methods to be evaluated are:

- U.S. EPA Method 8310 - HPLC with UV-fluorescence detection.
- U.S. EPA Method 8100 - GC/FID after soxhlet extraction by U.S. EPA Method 3550.

An addendum to this QAPP will be prepared and submitted to the U.S. EPA prior to the implementation of the above method(s).

The SOP in Appendix B presents protocols for GC/MS tuning and calibration.

8.2 FIELD SCREENING ANALYTICAL PROTOCOLS

No field screening or field measurements will be performed during background soil and sediment sampling activities at the Moss-American Site.

SECTION 9

INTERNAL QUALITY CONTROL CHECKS

9.1 FIELD SAMPLE COLLECTION

The assessment of Quality Control (QC) for field sampling will be made through the collection of field duplicate samples in accordance with the applicable procedures and frequency described in the FSP, Appendix A of the QAPP.

9.2 FIELD MEASUREMENT

No field measurements will be performed during background soil and sediment pre-design sampling activities at the Moss-American Site.

9.3 LABORATORY INTERNAL QUALITY CONTROL CHECKS

The daily quality of analytical data generated in the WESTON laboratories is controlled by the implementation of its Analytical Laboratory Quality Assurance Program Plan.

Quality, as the term is used herein, is defined as the level of excellence needed to conform to an established standard. Generally, quality will refer to the excellence of end results and/or the excellence of performance required to attain the established standard.

QA is defined as those planned and systematic actions necessary to provide adequate confidence to WESTON and its clients that the services provided meet mutually accepted quality standards consistent with project scope and budget. Quality assurance is attained through the implementation of a quality control program.

QC is defined as the operational processes employed to ensure an objective level of excellence. Established performance criteria are defined for all areas, including:

- Administrative and technical methods and procedures.
- Position accountability, duties and authority.
- Performance monitoring.
- Peer and supervisory review, check, approval, and sign-off.

QC provides the tools to measure and evaluate the conformance of the operational procedures to criteria.

In order to assess the validity of a reported results, QC indicators are placed in the measurement system to provide a tool for evaluating how well the method worked. There are QC indicators to evaluate method performance at both the preparation and the measurement steps, and QC indicators to evaluate matrix effects.

The types of internal quality control checks used in the WESTON Lionville Analytical Chemistry Laboratory are described in this section.

9.3.1 Method Performance QC Indicators

- Preparation Batch - Samples to be analyzed in the laboratory for this project will require extraction before analysis can be done. During the extraction step, samples are arranged into discreet, manageable groups, called preparation batches, to facilitate and control uniform treatment for all samples. Each preparation batch will have a maximum of 20 investigative samples of the same matrix (e.g., soil or sediment). In addition, QC indicators such as blanks, spikes, and duplicates are added to each preparation batch to monitor the performance of the system. All QC associated with a preparation batch will be carried through the entire analytical procedures, from prep to final analysis.

- Preparation Blanks - The preparation blank, also referenced as a method blank or reagent blank, is used to monitor potential contamination from the sample preparation process. Preparation blanks will be prepared by processing sodium sulfate, through the entire analytical scheme. The reagent blank weight must be approximately equal to the sample weights being processed. Results will be calculated based on starting with a "blank" soil approximately equal to the weight of the samples.

  Preparation blanks are analyzed at a rate of one per prep batch (20 or fewer samples).

- Blank Spikes - The blank spike is sodium sulfate (approximately equal in weight to the samples being processed) fortified (spiked) with the analytes of interest at a concentration in the mid-range of the calibration curve. It is processed through the entire preparation and analysis procedures concurrently

to the investigative samples to demonstrate acceptable method performance, independent of the investigative sample matrix. To facilitate comparison to the actual field samples, final results for the blank spike will be calculated as nanogram per gram (ng/g), assuming 100 percent solids and a weight equivalent to the aliquot used for the corresponding investigative samples. Blank spikes will only be analyzed and reported if the associated matrix spikes yield poor results or if the preparation batch includes no matrix spikes.

9.3.2 Matrix QC Indicators

Matrix QC indicators include duplicates and matrix spikes (MS). Over the last several years, matrix spike duplicates (MSD) have become popular replacements for laboratory duplicates, as they provide measurement data for precision assessment when no target compounds are indigenous to the sample selected for duplicate analysis.

A matrix spike is an aliquot of an investigative sample which is fortified (spiked) with the analytes of interest and analyzed with an associated sample batch to monitor the effects of the investigative sample matrix (matrix effects) on the analytical method.

For this project, MS/MSDs analyses will be performed at a rate of 5 percent (1 per 20 samples of the same matrix). All eight analytes of interest will be spiked into the sample at a mid-range calibration level.

9.3.3 Surrogates and Internal Standards

Two surrogates will be spiked into all samples prior to sample preparation to assess extraction and analysis efficiency. The surrogate compounds to be used are: chrysene-d12 and dibenzo(a,h)anthracene-d14. Samples with surrogate recoveries of less than 50 percent or greater than 120 percent will be re-extracted and re-analyzed if it is determined that the outliers are not due to matrix effects.

Three internal standards will be added to the sample prior to analysis but after sample preparation. The internal standards to be used are: pyrene-d10, benzo(a)pyrene-d12, and benzo(g,h,i)perylene-d12.

Solvent/Reagent Water Approval

Pre-purchase approval of solvents, including bottled water purchased for field sampling projects, is performed for all solvents purchased in large quantities. This includes, but is not

limited to, acetone, acetonitrile, ethyl ether, freon, hexane, isooctane, methanol, methylene chloride,, toluene, bottled deionized water, and bottled HPLC water. Prior to purchase, a candidate lot of solvent is put in reserve at the vendor's warehouse. A sample case of the lot of solvent is provided by the vendor to the laboratory for testing. If the solvent passes acceptance criteria, the vendor is notified and holds the sample in reserve for laboratory use. The approved lot of solvent is shipped to the laboratory in increments until the entire lot has been received. Prior to exhaustion of the reserve lot, the process will be repeated with a new lot to ensure a constant supply of approved solvent.

The laboratory's on-tap deionized water supply is similarly tested on a monthly basis for selected parameters. Samples are collected and submitted for analysis by laboratory personnel.

Balances, Refrigerators

All sample/standards storage refrigerators and freezers are monitored daily. Refrigerators are monitored twice daily, and include the walk-in coolers in the sample receipt areas as well as those located within the individual laboratories. Balance calibration for all analytical balances is checked daily per WESTON OP21-06-102, "Daily Balance Check."

Instrument Time Check Verifications

An independent check of GC and GC/MS instrument time clocks is performed randomly and at a minimum prescribed frequency by the Laboratory QA Section.

SECTION 10

DATA REDUCTION, VALIDATION AND REPORTING

10.1 FIELD MEASUREMENTS

No field measurement data will be generated during background soil and sediment pre-design field sampling activities.

10.2 LABORATORY SERVICES

10.2.1 Data Reduction

Data reduction is performed by the individual analysts and consists of calculating concentrations in samples from the raw data obtained from the measuring instruments. The complexity of the data reduction will be dependent on the specific analytical method and the number of discrete operations (e.g., extractions, dilutions, and concentrations) involved in obtaining a sample that can be measured. The analyst will reduce or calculate all raw data into the final reportable values or enter all necessary raw data into LIMS in order for the database system to calculate the final reportable values. Copies of all raw data and the calculations used to generate the final results, such as hard-bound laboratory notebooks, strip-charts, chromatograms, LOTUS spreadsheets, and LIMS record files, will be retained on file to allow reconstruction of the data reduction process at a later date.

For data reporting, rounding will not be performed until after the final result is obtained to minimize rounding errors, and results will not normally be expressed in more than two (2) or three (3) significant figures. All results will be reported with the proper measurement units (e.g., mg/L, μg/kg, etc.). Appendix B presents the formulas to be used in determining the concentration of contaminants in samples.

10.2.2 Data Review/Data Reporting

Data Review

The individual analyst constantly reviews the quality of data through calibration checks, quality control sample results, and performance evaluation samples. These reviews are performed prior to submission to the Section Manager or the Laboratory Project Manager.

The Section Manager and/or the Laboratory Project Manager review data to ensure consistency with laboratory QC requirements, to verify reasonableness with other generated data, and to determine if program requirements have been satisfied. Selected hard copy output of data (chromatograms, spectra, etc.) will be reviewed to ensure that results are interpreted correctly. Unusual or unexpected results will be reviewed, and a resolution will be made as to whether the analysis should be repeated. In addition, the Laboratory Project Manager or Section Manager will recalculate selected results to verify the calculation procedure. The SOP in Appendix B contains guidance on the evaluation of surrogate and MS/MSD recovery data.

Prior to final review/sign-off by the Laboratory Project Manager, the Data Reporting Section will verify that the report deliverable is complete and in proper format, screen the report for compliance to laboratory and client QA/QC requirements, and ensure that the case narrative covers any noted deficiencies The Laboratory Project Manager will be the final laboratory review prior to reporting the results to the client's Project Manager (Project Manager).

The Laboratory Quality Assurance (QA) Section independently conducts a complete review of selected reports to determine if laboratory and client quality assurance/quality control requirements have been met. The Laboratory QA Section will also review 10 percent of teh data packages. Discrepancies will be reported to the appropriate Section Manager and/or Laboratory Project Manager for resolution.

Data Reporting

Reports will contain final results (uncorrected for blanks and recoveries), blank and recovery results, methods of analysis, levels of detection, surrogate recovery data, and method blank data. In addition, special analytical problems, and/or any modifications of referenced methods will be noted. The number of significant figures reported will be consistent with the limits of uncertainty inherent in the analytical method. Consequently, more analytical results will be reported to no more than two (2) or three (3) significant figures. Data are normally reported in units commonly used for the analyses performed. Concentrations in solid or semi-solid matrices are expressed in terms of weight per unit weight of sample (e.g., nanograms per gram [ng/g]).

Reported detection limits will be the concentration corresponding to the low level instrument calibration standard after all method concentration, dilution, and/or extraction factors are accounted for, unless otherwise specified by program requirements.

10.2.3 Data Validation

Data validation will be performed by trained WESTON personnel. Validation will be accomplished by comparing the contents of the data packages and QA/QC results to the requirements contained in the method SOP. The validation procedures will be based on the following U.S. EPA Region V validation protocol:

- Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses - U.S. EPA, February 1988.

Any deviations from the above protocol will be based on the requirements of the modified low concentration CPAH Method 8270 SOP (Appendix B).

The final data report to be provided by WESTON Lionville Laboratory is a data documentation package assembled in accordance with U.S. EPA Contract Laboratory Program requirements or as near as possible given the difference in the modified Method 8270. Briefly summarized, the report will include:

- Cover page/laboratory chronicle.
- Chain-of-Custody Sample Request Forms.
- Case narrative.
- Tabulated results (including QC results) on CLP forms when appropriate.
- All associated raw data for standards and samples.

The final data report will be given to the WESTON and KMCC Project Managers and the WESTON Project Director, and it will be available to the U.S. EPA upon request.

SECTION 11

PERFORMANCE AND SYSTEM AUDITS

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

11.1 FIELD AUDITS

Internal audits of field activities at the Moss-American Site will be the primary responsibility of the WESTON Project Director and/or Project Manager. In the absence of both persons, the QA of field activities will be conducted by the designated Field Team Leader. The audits will include examination of field sampling procedures and records; sample collection, handling and packaging protocols; chain-of-custody procedures, etc. in order to ensure compliance with established procedures. These audits will occur at the onset of the project to verify that all established procedures are followed. Follow-up audits will be conducted to correct any deficiencies that were previously identified and to verify that QA procedures are maintained throughout the project.

External field audits are the responsibility of the U.S. EPA Region V CRL and/or Central District Office (CDO).

11.2 LABORATORY AUDITS

Performance audits test the laboratory's ability to correctly assay an unknown sample. They may be single blind or double blind. In a single blind study, the analyst is not provided with the acceptable result for the unknown sample until after the experimental results are reported; however, it is known that the sample is a performance test. In a double blind performance test, the analyst not only has no knowledge of the acceptable result, but the sample is disguised in such a manner as to maintain anonymity as a performance test sample.

Systems audits and surveillances evaluate the operational details of the QA program. An audit consists of a systematic procedure to ascertain the implementation of a specific QA requirement, such as sample tracking or chain-of-custody procedures. Audits will be conducted by persons other than those who performed or directly supervised the work being

inspected. A surveillance consists of inspection or monitoring of a specific targeted area for compliance to requirements, such as an evaluation of a single analytical method to ensure conformance with the written SOP.

External Audits

The Lionville Laboratory QA Manager is responsible for scheduling and coordinating all external audits. External performance and system audits of the laboratory for the Moss-American project will be conducted by the U.S. EPA CRL.

Internal Audits

The Lionville Laboratory QA Manager has overall responsibility for monitoring the internal Quality Assurance/Quality Control program. The QA Section Manager has a staff to provide in-house audits, and to review and evaluate analytical data packages.

Internal performance audits conducted at the bench level provide the analyst with a tool to self-evaluate the acceptability of a specific data set. This is accomplished through analysis of laboratory control samples or spike blanks of known concentration to the analyst which must meet minimum performance standards. When these QC checks are performed in duplicate, method accuracy and precision information can be generated to demonstrate the proper functioning of the total measurement system.

As an additional feature of the laboratory's internal QA Program, double blind performance evaluation samples are periodically submitted to the laboratory for analysis. These samples originate both internally and externally, and are scheduled through the laboratory's project management system to ensure anonymity. Over the course of a year, samples are submitted to cover all routinely analyzed methods.

Externally originated double blinds are analyzed quarterly by the Lionville's Laboratory for full organic and inorganic target compound list parameters in both soil and water. Externally originated samples are purchased from a commercial vendor (currently Environmental Resources Associates) in a constituted form. WESTON initiates these external double-blind samples using the same procedures utilized for routine clients through a designated project manager, to include, for example, assigning of work order numbers, forward scheduling the analyses (using a "fake" client name, which changes quarterly), generation of bottle orders so that samples arrive in standard containers, etc. This system effectively gets samples into the laboratory for unbiased analysis. Results are compiled by the project manager and submitted to the QA Section for review and evaluation. Any noted

deficiencies are addressed with the appropriate laboratory service group and a corrective action plan is implemented, as needed.

Internally originated samples are handled in the same manner as the externally purchased double blinds, except that they are prepared by the laboratory, unknown to the analysts, using U.S. EPA, National Insititute of Standards and Technologies, or commercially available reference materials.

Internal laboratory systems audits and surveillances will be conducted and documented on a quarterly basis, at a minimum. Each quarter's audit will target a limited section of the laboratory, and be coordinated such that the entire laboratory is planned for QA audit at least once annually. Unique client audit procedures and data requirements will be complied with as contractually specified. The internal audit consists of a review of laboratory systems, procedures and documentation. Any deficiencies and/or deviations are documented and a summary report is prepared.

Items which may be included for focus in routine laboratory system audits and surveillances include, but are not limited to:

- life of reagents
- holding times
- interferences (if any)
- maintenance logs
- standards traceability
- preparation of glassware
- sample preservation
- equipment/instrumentation
- computer spreadsheets
- calculations
- standard deliverables
- lab book documentation
- safety
- method detection limits
- current standard operating practice

The system audit report is distributed to the responsible party, including the appropriate supervisor. A maximum of two weeks is given to address any recommended corrective actions. The original copy of the completed responses is kept on file in the QA Section.

SECTION 12

PREVENTATIVE MAINTENANCE PROCEDURES

12.1 FIELD EQUIPMENT

No field measurement equipment will be utilized during Moss-American predesign background sampling activities.

12.2 LABORATORY EQUIPMENT

The ability to generate valid analytical data requires that all analytical instrumentation be properly and regularly maintained. The responsibility of routine care lies with the analysts using the instruments. Guidance on required routine maintenance, as well as troubleshooting information, is provided in the respective instrument manuals and laboratory operating procedures. For more extensive preventative maintenance or emergency repair service, the analytical laboratory maintains full service contracts on all major instruments. The elements of the maintenance program are discussed below.

12.2.1 Instrument Maintenance Log Books

Each analytical instrument is assigned an instrument log book. All maintenance activities are recorded in the instrument log. The information entered in the instrument log includes:

- Date of service or maintenance.
- Person performing service or maintenance.
- Type of service performed and reason for service.
- Replacement parts installed (if appropriate).
- Documentation of the re-establishment of working order.
- Miscellaneous information.

If service is performed by the manufacturer, a copy of the service record (when available) is affixed to the notebook page,, or cross-referenced in the notebook to a separate maintenance file. The service record should include sufficient detail to describe the service performed (e.g., not just "service call," but "replaced pump motor gear"). If the service record does not spell out this information, it must be written separately into the maintenance log.

12.2.2 Instrument Maintenance and Repair

Preventative maintenance and repairs that cannot be performed by laboratory staff are contracted to the manufacturer's service department, or to an authorized maintenance vendor. WESTON's service agreements provide for preventative maintenance, emergency service, and emergency shipping of spare parts. Annual service of the laboratory balances is an example of contracted preventative maintenance. For emergency response, service contracts on the Gas Chromatographs, GC/MS instruments and AA-ICP require on-site response within 48-72 hours. (Typically, service representatives are at the laboratory within 24 hours of a service call.) The service contracts also provide for 24-hour delivery of critical spare parts in response to a service request.

The maintenance procedures and frequencies for major analytical instrumentation are summarized in Table 12-1.

12.2.3 Spare Parts

WESTON Laboratory maintains an inventory of routinely required spare parts (for example, spare sources, vacuum pumps and filaments for GC/MS, spare torches, burner heads for AA-ICP).

The instrument operators have the responsibility, with the appropriate Section Manager, to ensure that an acceptable inventory of spare parts is maintained.

12.2.4 Contingency Plans

Properly maintained equipment will provide dependable service; however, emergencies cannot be totally avoided. Major equipment, such as the LIMS and GC/MS instrumentation, are backed up with an uninterrupted power supply (UPS) to provide continuous operation through electrical power outages and "brown outs". If a power failure occurs during non-working hours (defined here as other than the normal 8:00 a.m. to 5:00 p.m. work week), the same security system which controls building access will activate an alarm to the security agency. Supervisory and building maintenance personnel are notified via beeper call, and can be on site within 20 minutes or remain on stand-by alert until the emergency is passed or further action is necessary. Additionally, some laboratory personnel from night shift will often already be on site. Service is generally restored within an hour, and the UPS coverage is sufficient to carry operations through until electric service is restored. For prolonged power outages, laboratory personnel on stand-by alert will prepare for an organized, systematic shut-down of major equipment. A decision on the need for auxiliary back-up generators to run storage refrigerators will be made.

Table 12-1

Equipment Maintenance Summary
WESTON Lionville Laboratory

INSTRUMENT	PROCEDURE	FREQUENCY
Finnigan GC/MS	Change column	As needed; depends on ability to meet performance criteria
	Change injector sleeve	
	Change septa	
	Clean ionizer source	Quarterly or as needed
	Change filament	Quarterly or as needed
	Change electron muliplier	As needed
	CARD GAGE MAINTENANCE:	
	Change air filter	Monthly/Quarterly
	Clean cooling fans	Monthly/Quarterly
	All PCRAs: reseat boards connectors and check all voltages on PCRAs to see if within specifications. Adjust if necessary	Monthly/Quarterly
	POWER CONTROLLER MAINTENANCE:	
	Clean cooling fans	Quarterly
	All PCRAs: reseat all connections	Quarterly
	VACUUM SYSTEM:	
	Mechanical pumps: change oil	Quarterly or as needed
	Diffusion pump: change oil	Annually or as needed
	Turbo pump: change oil, cooling fan, check water level in recirculator, change 50/50 mixture water/ethylene glycol	Quarterly or as needed
	COMPUTER SYSTEM:	
	Clean or replace cooling fans	Monthly/Quarterly
	All PCRAs: reseat boards, cables	Monthly/Quarterly
	Disk drive (CDC):	
	change filter	Quarterly
	change pre-filter	Monthly
	Disk drive (Priam/Winchester): clean cooling fans	Quarterly
	Tape streamer: clean tape head, clean capstan surface	Monthly or as needed
	Printronix printers (MVP, P300): check print quality	Quarterly

Table 12-1 (cont.)

Equipment Maintenance Summary
WESTON Lionville Laboratory

INSTRUMENT	PROCEDURE	FREQUENCY
Balances	Class "S" weight check	Daily, when used
	Clean pan and check if level	Daily
	Field service	Annually
Conductivity Meter	0.01 M KCl calibration	Daily
	Conductivity cell cleaning	As required
Deionized/Distilled	Check conductivity	Daily
	Check deionizer light	Daily
	Monitor for VOAs	Daily
	System cleaning	As required
	Replace cartridge & large mixed bed resins	As required
Drying Ovens	Temperature monitoring	Daily
	Temperature adjustments	As required
Refrigerators/ Freezers	Temperature monitoring	Daily
	Warning system checked	Monthly
	Temperature adjustment	As required
	Defrosting/cleaning	As required
Vacuum Pumps/	Drained	Weekly
Air Compressor	Belts checked	Monthly
	Lubricated	Semi-annually
pH/Specific Ion Meter	Calibration/check slope	Daily
	Clean electrode	As required
Centrifuge	Check brushes and bearings	Every 6 months or as needed
Water Baths	Temperature monitoring	Daily
	Water replaced	Monthly or as needed

With respect to instrument-related downtime, an attempt is made to maintain adequate redundancy in instrumentation to cover short-term losses due to repairs. For long-term downtime, arrangements can be made to rent appropriate equipment until necessary repairs can be completed.

SECTION 13

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

13.1 FIELD MEASUREMENTS

No field measurements will be generated during Moss-American Site predesign background sampling activities.

13.2 LABORATORY DATA

Laboratory results will be assessed for compliance with required precision, accuracy, completeness and sensitivity as follows:

13.2.1 Precision

Precision of laboratory analysis will be assessed by comparing the analytical results between MS/MSD for organic analysis. The % RPD will be calculated for each pair of duplicate analysis using the Equation 13-1.

$$\%RPD = \frac{S - D}{(S + D)/2} \times 100 \qquad \text{Equ. 13-1}$$

Where: S = First sample value (MS value)
D = Second sample value (MSD value)

13.2.2 Accuracy

Accuracy of laboratory results will be assessed for compliance with the established QC criteria that are described in Section 4 of the QAPP using the analytical results of method blanks, reagent/preparation blank, and matrix spike/matrix spike duplicate samples. The percent recovery (%R) of matrix spike samples will be calculated using Equation 13-2.

$$\%R = \frac{A - B}{C} \times 100 \qquad \text{Equ. 13-2}$$

Where:

A = The analyte concentration determined experimentally from the spike sample;

B = The background level determined by a separate analysis of the unspiked sample and;

C = The amount of the spike added.

13.2.3 Completeness

The data completeness of laboratory analyses results will be assessed for compliance with the amount of data required for decision making. Data completeness will be calculated using Equation 13-3.

$$\text{Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100 \quad \text{Equ. 13-3}$$

13.2.4 Sensitivity

The achievement of method detection limits depend on instrumental sensitivity and matrix effects. Therefore, it is important to monitor the instrumental sensitivity to ensure the data quality through constant instrument performance. The instrumental sensitivity will be monitored through the analysis of method blank and the low concentration calibration standards.

SECTION 14

CORRECTIVE ACTION

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

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

Any nonconformances with the established quality control procedures in the QAPP or FSP will be identified and corrected in accordance with the QAPP. The U.S. EPA RPM or her designee will issue a Nonconformance Report for each nonconformance condition.

14.1 FIELD CORRECTIVE ACTIONS

During all field activities, technical staff and project field personnel will be responsible for reporting all suspected technical or QA nonconformances or suspected deficiencies of any activity or issued document by reporting the situation to the Field Team Leader or his/her designee. The Field Team Leader will be responsible for assessing the suspected problem and notifying the WESTON PM of the problem and anticipated change, and implementing the change.

If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the Project Manager. The Project Manager will be responsible for informing the WESTON Project Director, the KMCC Project Manager, the U.S. EPA RPM, and WDNR of the problem. The Project Manager will be responsible for ensuring that corrective action for nonconformances are initiated by:

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

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

All changes will be evaluated based on the potential to impact the quality of data. The Project Manager has ultimate responsibility for all site activities. The Project Manager or his designee must approve all changes verbally and/or in writing prior to field implementation by the Field Team Leader. The WESTON Project Director, the KMCC Project Manager, the U.S. EPA RPM, and WDNR will be notified when field changes are implemented.

All problems and corrective actions will be documented in the field log book by the Field Team Leader. No field team member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by the Field Team Leader following instructions from the Project Manager (or his designee) and/or the U.S. EPA RPM.

14.2 LABORATORY CORRECTIVE ACTIONS

Laboratory corrective action may be immediate or long-term. Immediate corrective action to correct or repair non-conforming equipment and systems is generally initiated as the result of QC procedures. The individual has relatively quick feedback that a problem exists, e.g., calibration does not meet or QC check samples exceed allowable criteria, and can take immediate action to repair the system.

Long-term corrective action is generally initiated due to QA issues. These are most often identified during audits. This involves a deeper investigation into the root-cause of the nonconformance, and may take much longer to identify and resolve. Staff training, method

revision, replacement of equipment, LIMS reprogramming, etc., may be indicated by long-term corrective action.

All corrective actions, whether immediate or long-term, will comprise the following steps to ensure a closed-loop corrective action system.

- Define the problem.
- Assign responsibility for investigating the problem.
- Determine a corrective action to eliminate the problem.
- Assign and accept responsibility for implementing the corrective action.
- Establish effectiveness of the corrective action and implement the correction.
- Verify that the corrective action has eliminated the problem.

The initial responsibility to monitor the quality of a function or analytical system lies with the individual performing the task or procedure. Quality indicators are evaluated against laboratory established or client specified QA/QC requirements. If the assessment reveals that any of the QC acceptance criteria are not met, the analyst must immediately assess the analytical system to correct the problem. Figure 14-1 presents WESTON's laboratory Corrective Action Documentation Form. When an acceptable resolution cannot be met and/or data quality is negatively impacted, the analyst will notify the appropriate supervisor and initiate a Sample Discrepancy Report Form (Figure 14-2).

When the appropriate corrective action measures have been defined and the analytical system is determined to be "in control" or the measures required to put the system "in control" have been identified and scheduled, the problem and resolution or planned action is documented in the appropriate notebook. If a Sample Discrepancy Report was required, the report will be routed for proper authorizations and signatures.

Data generated concurrently with an out-of-control system will be evaluated for usability in light of the nature of the deficiency. If the deficiency does not impair the usability of the results, data will be reported and the deficiency noted in the case narrative. Where sample results are impaired, the Laboratory Project Manager is notified by a written Sample Discrepancy Report and appropriate corrective action (e.g., re-analysis) is taken and documented.

The Laboratory QA Section has the authority to stop the analysis and to hold all analyses of samples affected by an out-of-control situation. The method cannot be restarted without the above documentation leading to the QA Section's approval to restart the method. For cases where suspension of the method was imposed by QA, QA sign-off is required prior to reinstatement of the affected method.

WESTON CORRECTIVE ACTION DOCUMENTATION

AUDIT REPORT #

INSTRUCTIONS: 1) ORIGINATOR complete PERSON RESPONSIBLE FOR RESPONSE and DESCRIPTION OF PROBLEM blocks.
2) Originator forward form to PERSON RESPONSIBLE FOR RESPONSE.
3) Develop/plan a SEQUENCE OF CORRECTIVE ACTION and obtain INITIAL CA APPROVAL sign-off from supervisor.
4) Forward original form to QA for sign-off and FOLLOW-UP ACTION. This allows all pertinent action to be documented on the original form. On completion of the corrective action, the form is signed off by QA, distributed, and the original archived with the QA records.

DATE/ORIGINATOR

PAGE ___ OF ___

PERSON RESPONSIBLE FOR RESPONSE (corrective action plan and implementation of corrective action plan):

DISTRIBUTION:
___ LABORATORY MANAGER
___ INORGANIC MANAGER
___ GC/MS MANAGER
___ GC/EXTR MANAGER
___ QA MANAGER
___ QA REPORT FILE
___

DESCRIPTION OF PROBLEM and when identified:

CAUSE OF PROBLEM if known or suspected:

SEQUENCE OF CORRECTIVE ACTION (CA) planned (signature/date): _____

INITIAL CA APPROVAL: Supervisor signature/date:
QA signature/date:

DESCRIPTION OF QA FOLLOW-UP ACTION (include signature/date):

FINAL CA APPROVED (QA signature/date):

RFW 21-21-006/C-03/91

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

FIGURE 14-1

CORRECTIVE ACTION DOCUMENTATION FORM
WESTON LIONVILLE LABORATORY

WESTON SAMPLE DISCREPANCY REPORT (SDR)

SDR IN-PROGRESS ROUTING: (see other side)

Initiator _____
Date _____
Client _____
RFW Lot # _____
Samples _____

Parameter: _____
Matrix: _____
Prep Batch: _____
Urgency:
Immediate ___ Other

Category for Discrepancy:
___ Log-In
___ LIMS
___ Analysis/Sample
___ Project Revision
___ Other:

A. Reason for SDR:

A1a.
Requires Verification By (circle): Log-In or Prep Group

___ Missing Sample/Extract
___ Wrong Sample Pulled
___ Improper Bottle Type
___ Container Broken
___ Preservation Wrong
___ Received Past Hold
___ Insufficient Sample
___ Label ID's Illegible

A2.
Verified By (circle): Log-In or Prep Group
(signature) (date)

_____ _____
_____ _____
_____ _____
_____ _____
_____ _____
_____ _____
_____ _____
_____ _____

A1b.

___ Re-Log: Tech Profile Error..Client Changed Request..
Sampler Error on C-O-C..Transcription Error..
Wrong Test Code, Re-Log As _____
___ Re-Leach: Metals/Inorg/VOA/BNA/Pest/Herb/____
___ Re-Digest: AA/ICP/HG/_____
___ Re-Extract: BNA/PEST/_____
___ QC Out: SURR/MS...High/Low/<10%/Missing/2X
___ QC Out: B/BS/BSD/LCS/LCS-D...High/Low
___ Hold Time Exceeded: Prep/Analysis/Report
___ Not Amenable to Analysis
___ Other (describe)

B. PM Instructions For Disposition (signature/date): _____

___ Cancel ___ Add ___ Subout Analysis
___ Place On Hold ___ Take Off Hold
___ Change W.O. # to: _____
___ MS/MSD on Sample ____.if enough sample: ORG/INORG
___ MS/DUP on Sample ____.if enough sample: ORG/INORG
___ Change Client name to: _____
___ Wrong Test Code, Re-Log As _____
___ Include in Narrative

___ Other, explain:

C. FINAL ACTION: a clear description of what was done for resolution, when it was done, and by whom it was done

Action Taken:
___ Revision To Chain-of-Custody Completed
___ LIMS Corrections Completed
___ Other, explain

Action By (name/date): _____
Forward to Pat Feldman, QA for distribution ---->

D. Distribution of Completed SDR

X Initiator: _____
X Lab Manager: J.R. Tuschall
X QA (original): D.S. Therry
___ Data Reporting: _____
___ _____

Distributed By:
(signature/date)

RFW 21-21-005/E-10/90 (SDR Revision 5.0)

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

FIGURE 14-2

SAMPLE DISCREPANCY REPORT FORM
WESTON LIONVILLE LABORATORY

SDR CIRCULATION

Forward To:			Received:	
(√)	Name	Date	Initials	Date
LAST: Over for Final Copy and Distribution				

SUMMARY INSTRUCTIONS:

1. Initiator complete the top header section, and Section "A" Reason for SDR:

   If "A1a": route the YELLOW copy to "A2" Log-In/Sample Prep (circle one) for Verification.

   If "A1b": check/circle/fill in the applicable spaces,
   route the YELLOW copy to "B" PM Instructions for Disposition,
   or complete "C" Final Action.

2. After "A2" Verification: route the YELLOW copy to "B" PM Instructions for Disposition (if necessary), or complete "C" Final Action

3. After "B" PM Action: route YELLOW copy to the person responsible for taking the Final Action "C" to resolve the SDR.

4. Final Action: Describe the action taken for final resolution of the SDR in the lower left hand box "C" of the YELLOW copy.

5. Forward the completed YELLOW copy of the SDR to QA for distribution "D".

The Section Manager, with the respective Unit Leaders and Supervisors, are responsible for correcting out-of-control situations, placing highest priority on this endeavor.

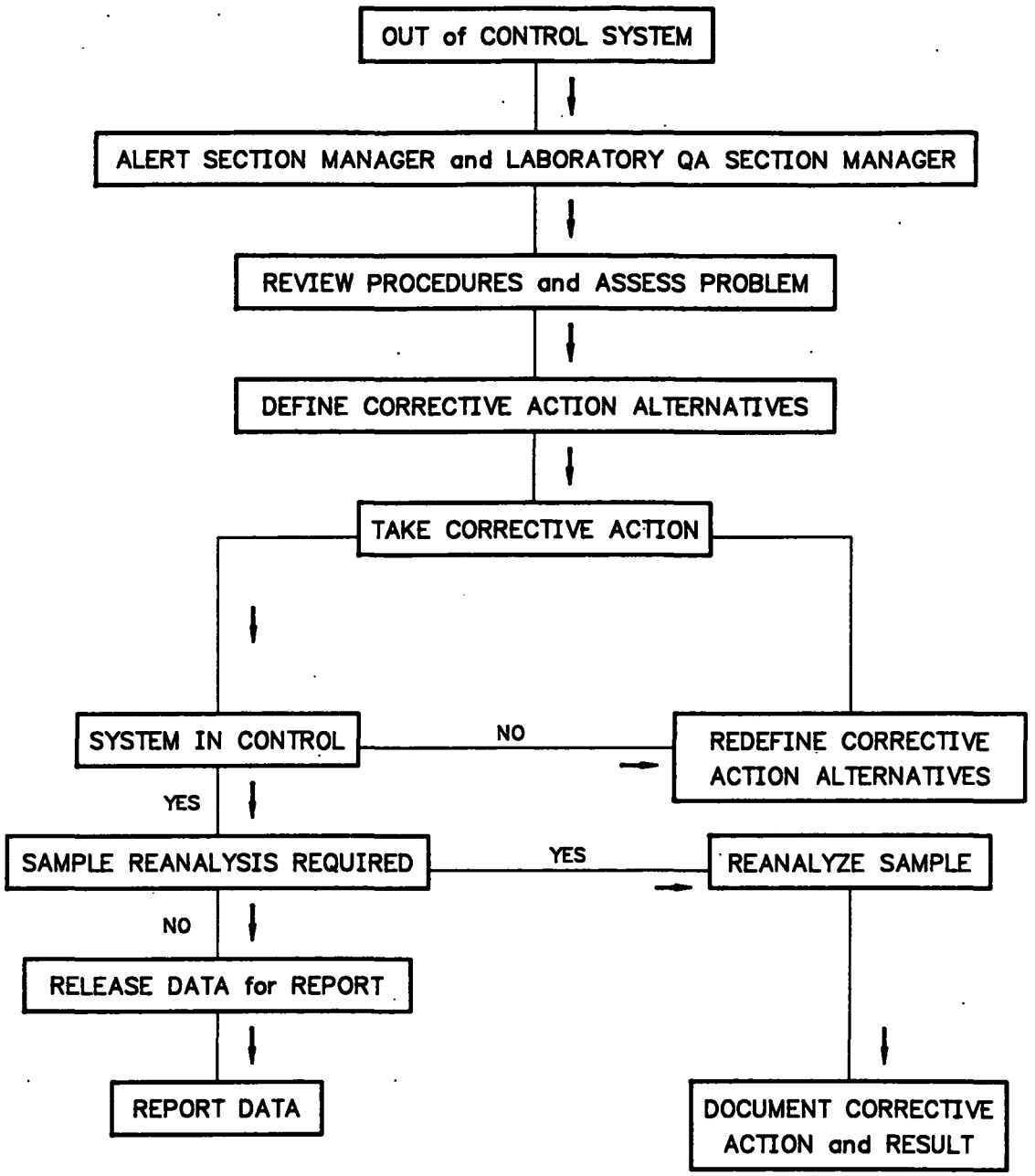
Any out-of-control situations that are not acceptably addressed at the laboratory level may be reported to Corporate Quality Assurance Management by the Laboratory Quality Assurance Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation. This provides laboratory QA personnel non-laboratory management support, if needed, to ensure QA policies and procedures are enforced.

The critical path assessing laboratory corrective action is presented in Figure 14-3.

Responses to External On-Sites/Performance Samples

When the results from an external on-site audit or performance evaluation study are received by the laboratory, a summary of the results is distributed to appropriate laboratory personnel.

If deficiencies exist, the person responsible for the response will issue a memo addressing the findings and resultant steps to correct the deficiency. Upon receipt of all corrective action responses, the Laboratory QA Section will forward the information to the WESTON Project Manager and the U.S. EPA RPM.



68992

FIGURE 14-3

WESTON MANAGERS DESIGNERS/CONSULTANTS

Three Hawthorn Parkway
Vernon Hills, Illinois
60084

CRITICAL PATH for
LABORATORY CORRECTIVE ACTION

SECTION 15

QUALITY ASSURANCE REPORTS TO MANAGEMENT

The WESTON Project Manager will audit the implementation of this QAPP. The preparation of a QA Report is not anticipated except as necessitated by problems arising during the project. Should these problems require the preparation of a QA Report, this task will be the responsibility of the WESTON Project Manager. The report may also include an assessment of field activities, data quality and the results of system and/or performance audits, as applicable. Any QA Report prepared by the WESTON Project Manager will be submitted to the WESTON Project Director, the KMCC Project Manager, the U.S. EPA RPM, and WDNR. The final project report will include QA information regardless of whether or not QA problems are observed.

APPENDIX A

FIELD SAMPLING PLAN

FIELD SAMPLING PLAN
FOR PREDESIGN TASK 2
MOSS-AMERICAN SITE
MILWAUKEE, WISCONSIN

Prepared for

Kerr-McGee Chemical Corporation
Kerr-McGee Center
Oklahoma City, Oklahoma

Prepared by

Roy F. Weston, Inc.
Three Hawthorn Parkway
Vernon Hills, Illinois 60061

18 November 1991

Final Revision: October 1992

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

SECTION 1

INTRODUCTION

This document presents the Field Sampling Plan (FSP) for determining area background concentrations of carcinogenic polycyclic aromatic hydrocarbons (CPAHs) in soils and sediments, for purposes of establishing clean-up standards at the Moss-American Site (hereinafter referred to as the facility). This work is being conducted as part of Predesign Task 2 of the Statement of Work (SOW). Specifically, the FSP addresses:

- Sampling plan rationale.
- Number and type of samples.
- Field sample collection procedures.
- Responsibilities of sampling personnel.
- Sample identification.
- Sample containers and preservation.
- Sample packaging and shipment.
- Chain of custody.
- Documentation.
- Quality assurance/quality control (QA/QC) of field sampling.

SECTION 2

SAMPLE NETWORK DESIGN AND RATIONALE

In support of the objectives outlined in the Quality Assurance Project Plan (QAPP), a stratified random sampling program will be employed to determine background concentrations of CPAHs in soil and sediment.

2.1 STATISTICAL RATIONALE

The two fundamental considerations for the statistical treatment of background data collection are the mean concentration and the variability of background CPAH concentrations. Assuming that background concentrations are normally and randomly distributed, the principles of elementary statistics can be applied to describe the true CPAH background concentrations. The arithmetic mean concentration describes what concentration of CPAH is typical. The variability, summarized by the standard deviation, describes how much variation in CPAH concentration from point to point is typical. Given these two descriptors, it is possible to conduct a variety of statistical analyses such as hypothesis testing and calculation of confidence limits.

Although a normally distributed random spread of background CPAH concentrations in all environmental media throughout northern Milwaukee would be the statistical ideal, it is almost surely not the case. Soils and sediments are not expected to exhibit comparable magnitudes or variations of background concentration CPAH throughout this entire region. For example, a wetland soil would probably have a different mean CPAH concentration relative to an upland soil. Many factors could be assumed to influence CPAH concentrations in a non-random way. For soils, influencing factors could include soil characteristics, vegetative cover, adjacent land use, and topography. For sediments, influencing factors could include current velocity, sediment particle size, organic carbon content, and adjacent land use.

To provide a practical method of addressing the non-random variation induced by the non-random influencing factors, five environmental settings are identified to serve as the basis of accounting for non-random influencing factors. Within a given environmental setting, it is expected that the non-random influencing factors would be sufficiently similar, so the assumption of normal distribution would not be violated.

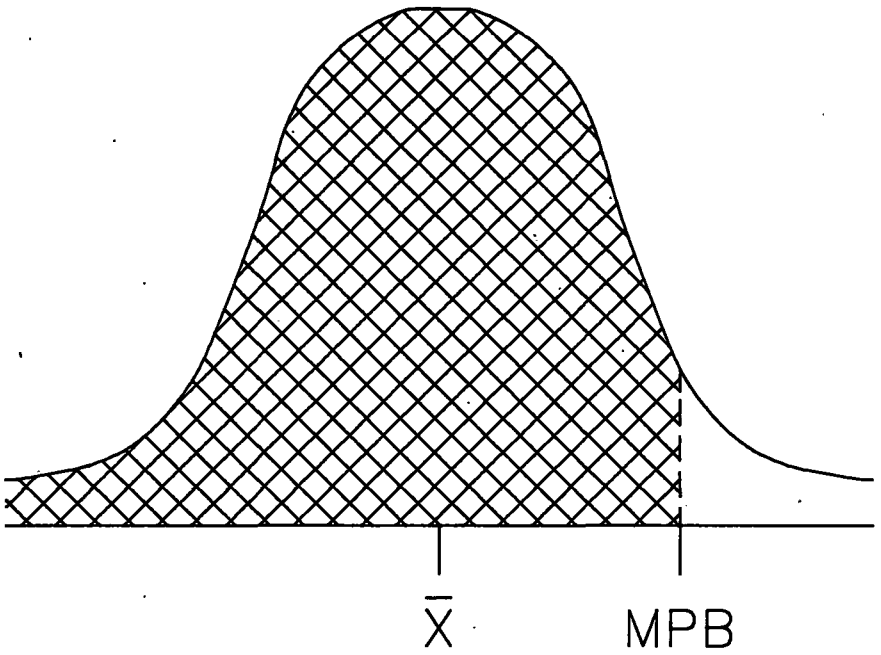
Having minimized non-random influences by isolating or "stratifying" the data from individual environmental settings, random factors remain to be addressed by statistical

analysis. The SOW for the facility (U.S. EPA, 1991) calls for use of the Maximum Probable Background (MPB) method. The MPB method described in Appendix J of the Moss-American Feasibility Study (FS) Report (U.S. EPA, 1990) accounts for the random variability of CPAH in the environmental background by equating background with the mean concentration plus the standard deviation times 1.65. Figure 2-1 illustrates the MPB method.

2.2 SOIL AND SEDIMENT SAMPLING PROGRAM

The determination of background CPAH concentrations in soil and sediment is important in determining cleanup standards for the various locations of the facility. The following summary of cleanup standards taken from the Consent Decree (U.S. EPA, 1991) will be applied during the remedial action:

Media/Location	Summary of Cleanup Standard
Soil on former wood treating plant not within 100-year floodplain.	Background or 6.1 mg/kg total CPAHs, whichever is greater, and visibly contaminated soil.
Soil on former wood treating plant within 100-year floodplain.	Background or 0.061 mg/kg total CPAHs, whichever is greater.
Soil in the 100-year floodplain downstream of the former wood treating plant.	Visibly contaminated soil and hot spots containing total CPAHs in excess of background or 6.1 mg/kg, whichever is greater.
Soil in the northeast landfill.	Background or 0.061 mg/kg total CPAHs, whichever is greater.
Sediment in reaches of Little Menomonee River that are not relocated.	Background or total CPAHs in excess of SQC (3 mg/kg), whichever is greater.
Soil in the new Little Menomonee River channel.	Total CPAH greater than SQC or background, whichever is greater.
Soil disturbed during river relocation construction.	Background or 6.1 mg/kg total CPAHs, whichever is greater.



$$P\ (X \leq MPB) = 0.95$$

$$MPB = \bar{X} + 1.65 * S$$

WHERE:

P(X < MPB) = 0.95 => THE PROBABILITY (p) THAT A SAMPLE CONCENTRATION (x) WILL BE LESS THAN OR EQUAL TO THE MPB IS 0.95.

MPB = $\bar{x}$+1.65 X s => THE MAXIMUM PROBABLE BACKGROUND (MPB) CONCENTRATION EQUALS THE MEAN SAMPLE CONCENTRATION ($\bar{x}$) PLUS 1.65 TIMES THE SAMPLE STANDARD DEVIATION (s). ASSUMES A NORMAL DISTRIBUTION TO COMPUTE A ONE-SIDE 95% UPPER CONFIDENCE LIMIT ON THE MEAN; THUS, $Z_{\alpha=0.05} = 1.65$.

WESTON MANAGERS DESIGNERS/CONSULTANTS	Three Hawthorn Parkway Vernon Hills, Illinois 60061	FIGURE 2-1	MPB METHOD MOSS-AMERICAN SITE Milwaukee, Wisconsin

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Area background concentrations of CPAHs have not been determined for the facility. This soil and sediment sampling program has been developed to provide samples for laboratory analysis and for the statistical determination of MPB, as previously described in Subsection 2.1.

The determination of background will be conducted in phases for both soil and sediment. During Phase I, sediment background concentrations upstream of the former wood preserving facility and background soil concentrations in habitats that are representative of the habitats that currently exist in the floodplain of the former facility and the former Northeast Landfill will be examined. Phase II will examine the sediment background in downstream reaches of the Little Menomonee River and soil background in habitats that are representative of habitats that occur in the floodplain downstream of the former facility.

This sampling plan has been divided into two phases to allow for examination of the Phase I data prior to implementing the Phase II work. Phase I data may determine that background concentrations are significantly lower than the corresponding risk-based cleanup criteria. If this proves to be the case, the time and expense to sample and analyze background soil and sediment downstream of the former facility would not be justified. Thus, Phase II activities would not be performed.

All background sediment and soil sampling will be conducted in demographic areas that represent Residential/Agricultural development. Figure 2-2 depicts regional land use in the vicinity of the Moss-American facility. Figures 2-3 and 2-4 illustrate habitats and floodplains in various demographic settings that may be candidates for sampling background soils for MPB.

The following subsections describe the planned Phase I and potential Phase II soil and sediment sampling designs.

2.2.1 Soil Sampling Design

Phase I of this soil sampling program is designed to determine background concentrations of CPAHs in environmental settings similar to the environmental settings on the former wood preserving facility and the Northeast Landfill. Specifically, cleanup standards for soils within the 100-year floodplain and Northeast Landfill may be tied to area background concentrations of CPAH. Soil settings within these areas are best described and defined based upon terrestrial habitat. Terrestrial habitats are established by the Corps of Engineers under the National Wetlands Inventory (NWI). Terrestrial habitats provide a convenient and scientifically-sound basis for identifying comparable environmental settings, as they are well defined and have been mapped along the Little Menomonee River. The

R	I&A	A	A&I	I,A&R	A&C	R	R&I	A&I	A	R
R	R	A&R	G	I&C	A&I	A&R	R	R	R	R
S	R	A&R	A	A&R	R&A	R&I	R	R	R	R
S	A&R	A&R	R	R	D-R	R	R	R&I	R	R
A	A&R	A&R	R	R	D-R	D-R	P&I	D-R	I&R	R
A&R	R&A	R&A	R	A	D-R	D-R	D-R	D-R	D-R	R

MENOMONEE RIVER

LITTLE MENOMONEE RIVER

MILWAUKEE RIVER

N

0' 1 Mile

SCALE

LEGEND

G= Golf
I= Industrial
R= Residential
S= Swamp
D-R= Dense Residential
P= Park
A= Agricultural
C= Commercial

NOTE: Interpreted from Aerial Photographs gathered for Wetlands Inventory, flown May 1980.

WESTON MANAGERS DESIGNERS/CONSULTANTS

Three Hawthorn Parkway
Vernon Hills, Illinois
60061

FIGURE 2-2

REGIONAL LAND USE

MOSS-AMERICAN SITE
Milwaukee, Wisconsin

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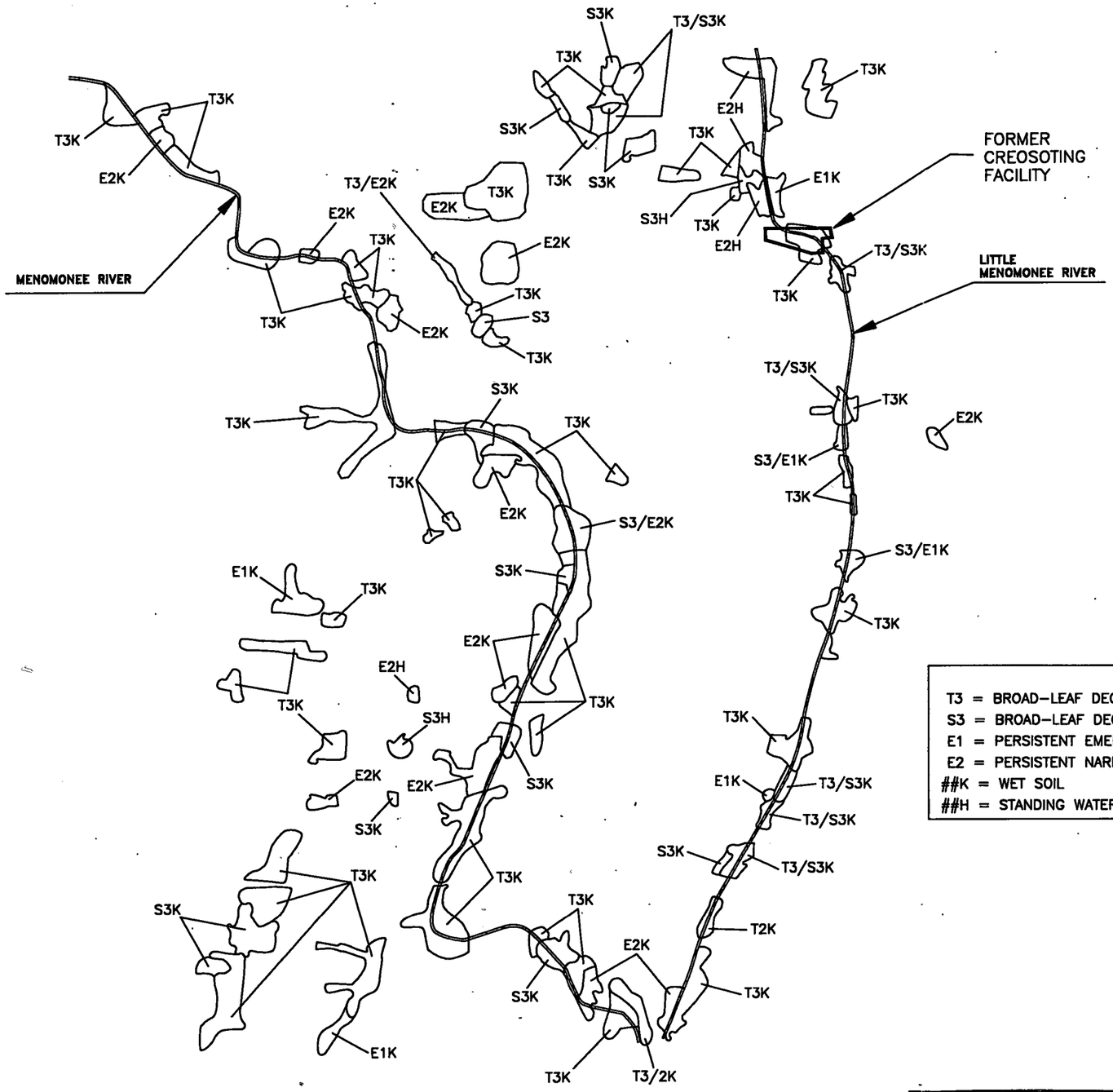

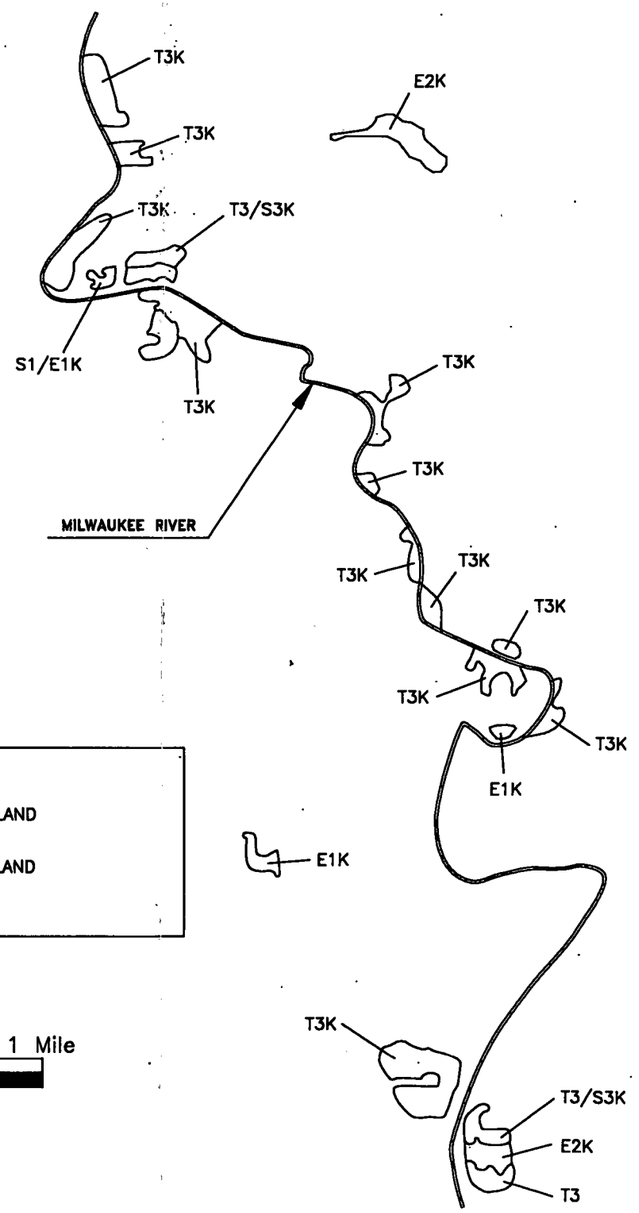

LEGEND

T3 = BROAD–LEAF DECIDUOUS FOREST WETLAND
S3 = BROAD–LEAF DECIDUOUS SCRUB/SHRUB WETLAND
E1 = PERSISTENT EMERGENT
E2 = PERSISTENT NARROW–LEAFED EMERGENT WETLAND
##K = WET SOIL
##H = STANDING WATER

0' 1 Mile
SCALE

SOURCE: WISCONSIN WETLANDS INVENTORY
REVISED 2–27–89 BY: WDNR AND SEWRPC

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

FIGURE 2–3

REGIONAL WETLANDS

MOSS–AMERICAN SITE
Milwaukee, Wisconsin

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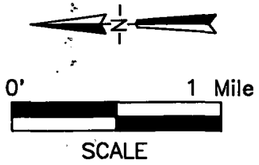

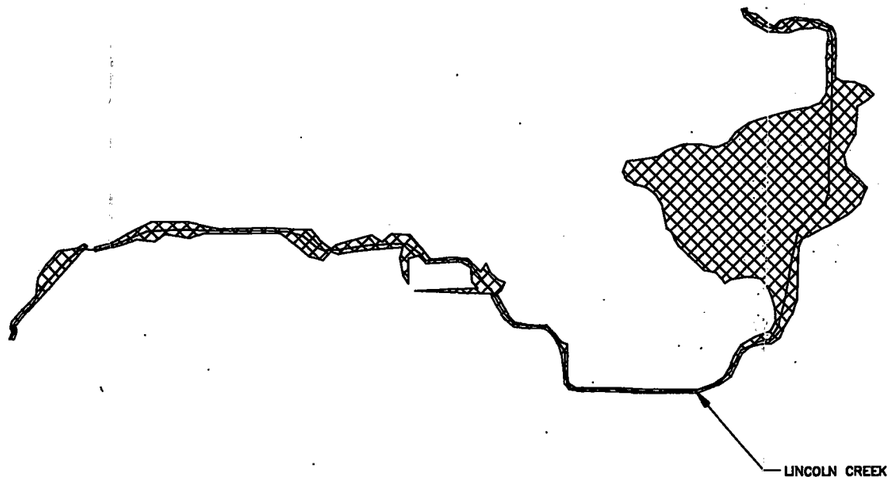

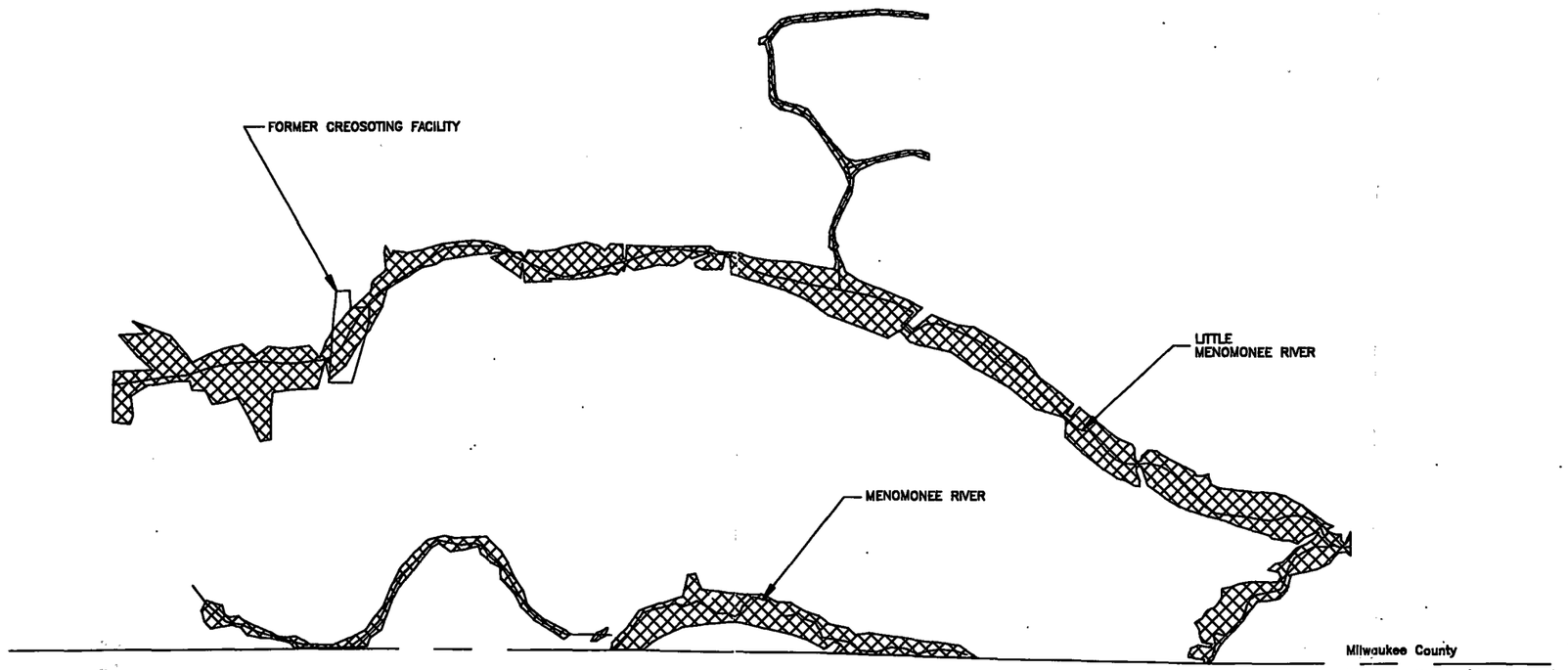

= 100 Year Flood Boundary
Source: FEMA – 1 Mar 1982

FIGURE 2–4

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

REGIONAL FLOODPLAINS
MOSS–AMERICAN SITE
Milwaukee, Wisconsin

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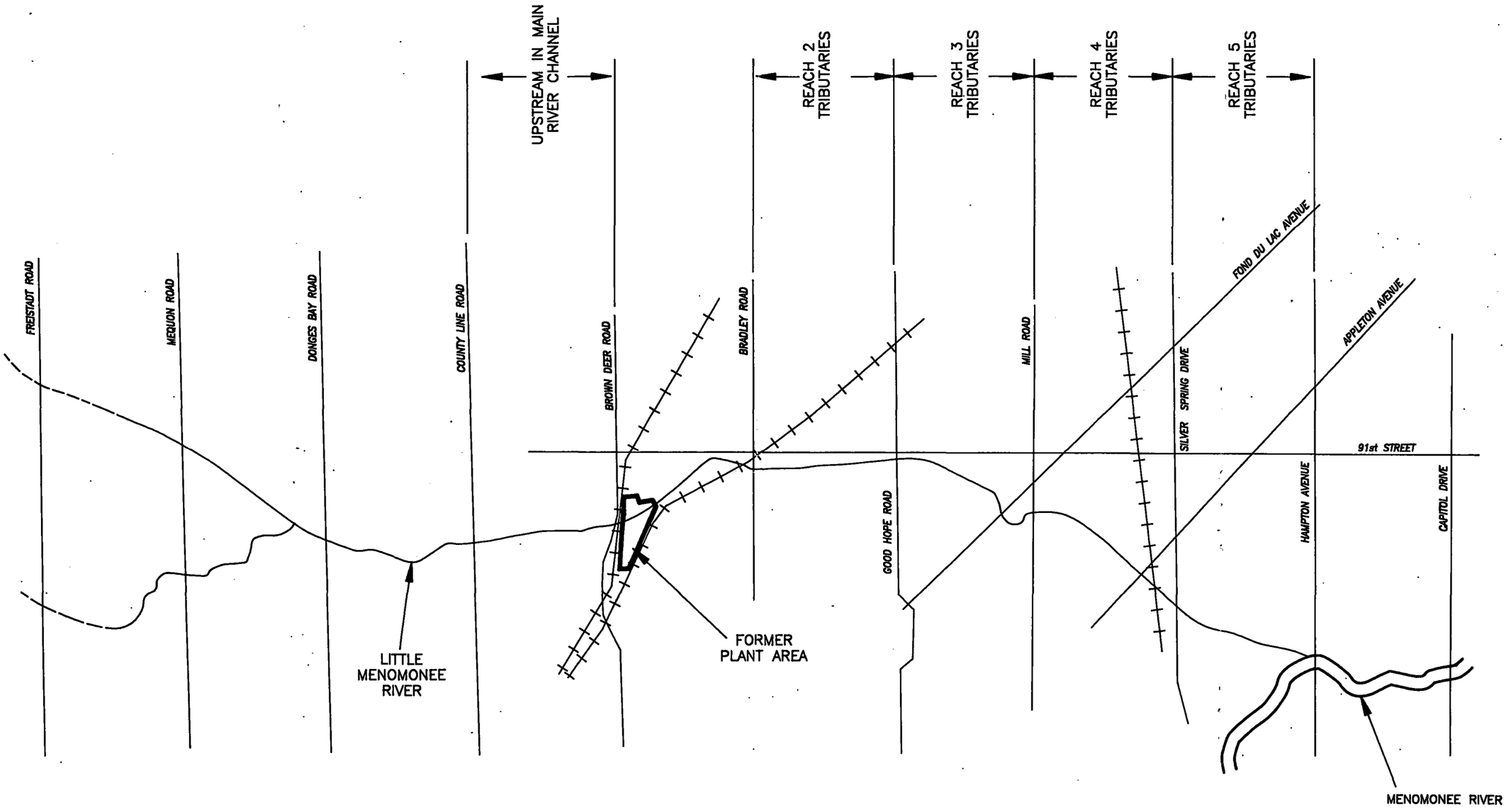

WESTON MANAGERS DESIGNERS/CONSULTANTS

Three Hawthorn Parkway
Vernon Hills, Illinois
60061

FIGURE 2-6

PROPOSED SAMPLING AREAS FOR SEDIMENT
MOSS-AMERICAN SITE
Milwaukee, Wisconsin

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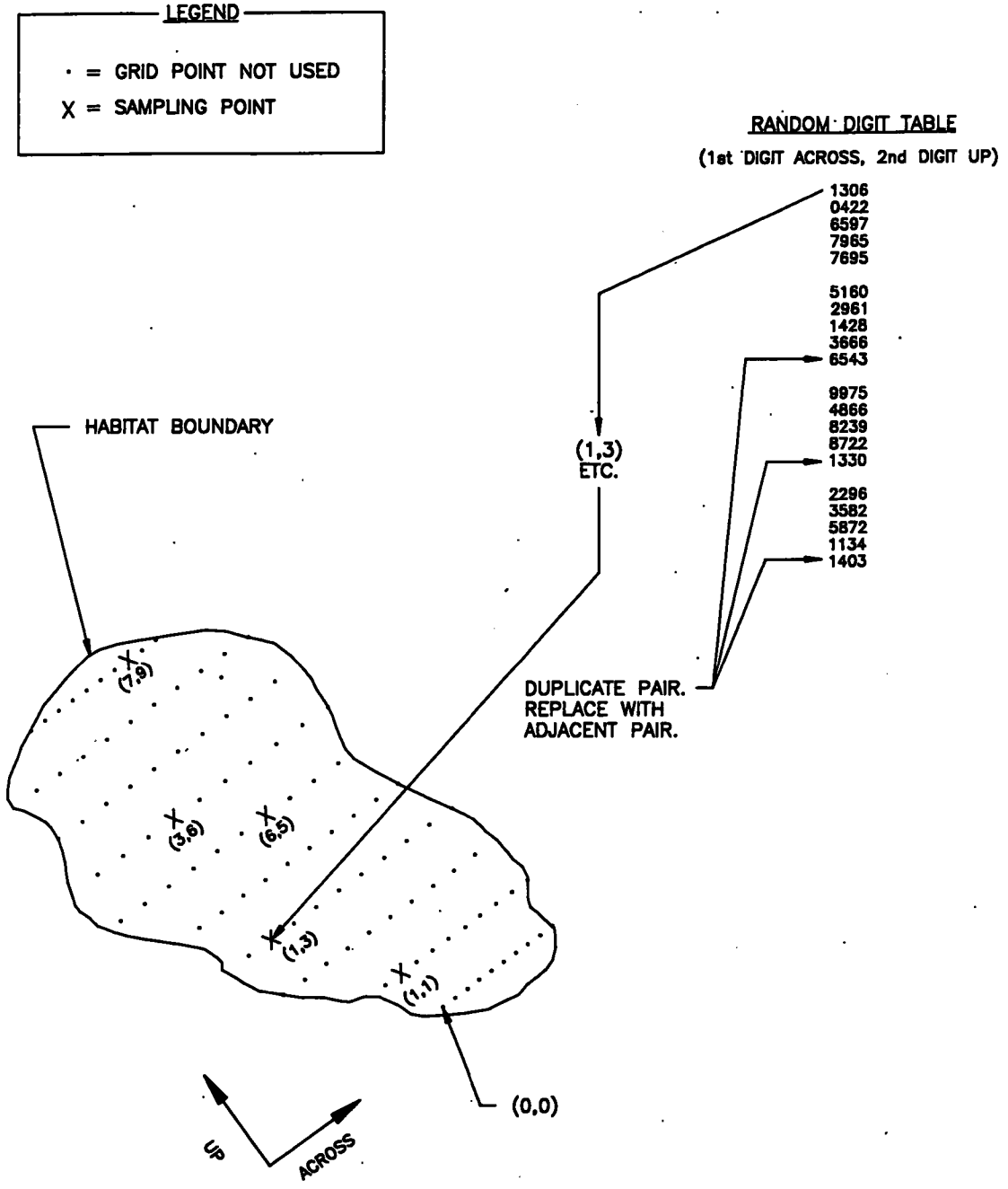
NWI has established two habitats in the floodplain on the former facility: broadleaf, deciduous forest wetlands and broadleaf, deciduous scrub-shrub wetlands. A third habitat, a non-wetland, non-forested upland area, is located at the site of the former Northeast Landfill.

CPAH background for the two floodplain habitats and the upland area habitat will be determined by identifying similar environmental settings in the vicinity and by implementing a system of stratified random collection of samples. WESTON, U.S. EPA, and WDNR will conduct a site visit to identify and mutually agree upon a total of nine locations for background sampling. WESTON and the agencies should be represented by terrestrial ecologists experienced in wetlands delineation and soil science. The nine locations will be based upon identification of three areas, representative of each of the three habitats on the facility. The locations will be selected from upstream or nearby watersheds in similar topographic and demographic settings. Each location will be identified, described and depicted on a topographic map.

Professional judgment will also be used in selecting sampling locations to avoid sampling areas that may have been impacted by airborne contamination from the site, areas affected by other past waste or product management activities that contribute PAHs to the environment, areas affected by major transportation activities (e.g., major highways and railroads), and areas of fill.

Following a mobilization period, a sampling team will return to the site to establish grids and collect soil samples from each of the three habitats. Grid size will be dependent upon the size of the location selected. It is likely that grids will measure 100 feet x 100 feet with a 10-foot interval or 200 feet x 200 feet with a 20-foot interval. A table of random numbers will be used to select five locations on each grid for sampling. Figure 2-5 illustrates the selection process that will be used to identify the five sample collection locations within each grid. Five samples will be collected from each of the three representative grids for each of three habitats. This approach will yield a total of 15 samples/habitat and a total of 45 samples for the first phase of soil background sampling.

Phase II background soil sampling may be undertaken after completion and evaluation of Phase I data. If area background (MPB) determined in the Phase I is significantly lower than the risk-based cleanup standards, then it may be unnecessary to further investigate background for the remaining habitats that are represented downstream of the facility. If area background exceeds risk-based cleanup standards, Phase I soil background data may be subjected to appropriate statistical tests (ANOVA, Newman-Keuls, Tukey's) to determine the usefulness of stratification. If area background exceeds risk-based cleanup standards, Phase II soil sampling will be undertaken. Phase II soil sampling will follow the same



WESTON MANAGERS DESIGNERS/CONSULTANTS	Three Hawthorn Parkway Vernon Hills, Illinois 60061	FIGURE 2-5	SAMPLE GRID EXAMPLE MOSS-AMERICAN SITE Milwaukee, Wisconsin

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procedure described for Phase I. The focus will be on determining MPB concentrations of CPAHs in floodplain habitats. The floodplain of the Little Menomonee River downstream of the former wood preserving facility contains a variety of habitats.

Based upon present data, the probable habitats to be sampled during Phase II will include:

- Emergent, persistent wetlands (Figure 2-3).
- Areas within 100-year floodplain but outside wetlands (Figure 2-4).
- Additional upland habitats.

In addition to the habitats listed previously, the broadleaf deciduous forest wetlands (Figure 2-3), broadleaf deciduous scrub-shrub wetlands (Figure 2-3), and nonwetland, nonforested upland occur downstream of the facility. MPB for these habitats will be based upon Phase I work.

The same procedure for identifying representative habitats, establishing grids, and collecting samples during the first phase of soil sampling will be followed in the Phase II. In consultation with the U.S. EPA and WDNR, representative habitats for each of the NWI-identified habitats that have been mapped along the river downstream of the former facility will be selected. The representative habitats will be identified in upstream floodplain areas or in nearby watersheds in similar topographic and demographic settings.

The results of the first phase of soil background analysis may indicate that alternate laboratory analytical method(s) may be utilized which yield reliable data in the second phase of soil sampling. Alternative methods would be undertaken to reduce laboratory costs and turnaround time. This is discussed in Section 8 of the accompanying QAPP.

Table 2-1 presents a summary of the anticipated Phase I and Phase II soil background sampling effort for the Moss-American Site.

2.2.2 Sediment Sampling Design

Background CPAH concentration in sediments are needed to derive cleanup standards for the Little Menomonee River.

The determination of MPB for sediments will be conducted in two phases using the rationale described in Subsection 2.2.1. In the event that Phase I sediment MPB concentrations are significantly less than the Sediment Quality Criteria (SQC), Phase II sediment sampling may not be implemented.

Table 2-1

Summary of Background Sampling Effort
Moss-American Site
Milwaukee, Wisconsin

Sample Matrix	Laboratory Parameters	Investigative			Field Duplicate			MS/MSD[a]			
		No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	Matrix Total[b]
SOIL											
Phase I											
Background Soil	Low DL CPAH[c]	45	1	45	5	1	5	3	1	3	50
Phase II											
Background Soil	Low DL CPAH[c]	30	1	30	3	1	3	2	1	2	33
SEDIMENT											
Phase I											
Background Sediment	Low DL CPAH[c]	15	1	15	2	1	2	1	1	1	17
Phase II											
Background Sediment	Low DL CPAH[c]	40	1	40	4	1	4	2	1	2	44

Notes:

[a]MS/MSD samples are not additional samples, but instead investigative samples assigned for MS/MSD analysis. No extra volume will be collected for MS/MSD samples.

[b]Matrix totals do not include matrix spike/matrix spike duplicate samples.

[c]The SOP for low detection limit (DL) carcinogenic PAH analysis is presented in Appendix B.

The following background settings will be evaluated during Phases I and II of the sediment sampling:

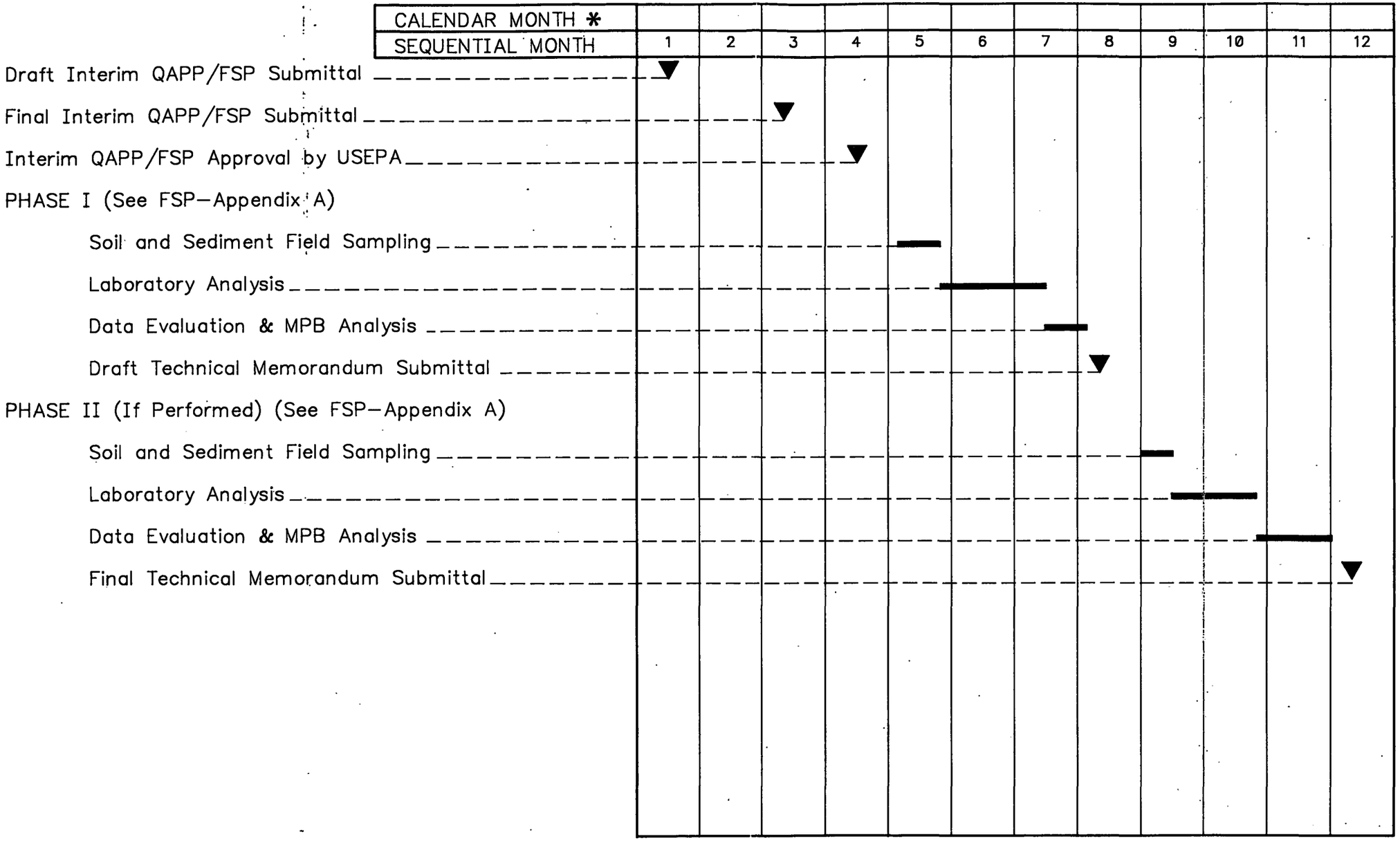
- Upstream of the former wood preserving plant in the main channel of the Little Menomonee River.
- Non-site-related tributaries (ditches, storm sewers, catch basins and manholes, and creeks) to the Little Menomonee River adjacent to
  - Reach 2
  - Reach 3
  - Reach 4
  - Reach 5.

Reach 1 of the Little Menomonee River (i.e., the reach adjacent to the former wood preserving plant property) will not be sampled because of the scarcity of non-site-related tributaries and because upstream sediment background values are appropriate estimates of background for Reach 1.

In general terms, the methodology for background sediment sampling in any of the settings will involve identifying locations prone to deposition of sediment. For Phase I, WESTON, U.S. EPA, and WDNR will meet at the site to walk the river upstream from the site. WESTON will be represented by an aquatic ecologist and/or a hydrologist with experience in the evaluation of sediment particle size based on visual and textural observation. Professionals with similar skills should be on hand for U.S. EPA and WDNR. The field team (WESTON, U.S. EPA, and WDNR) will identify and agree upon candidate sediment sample collection locations. Locations should represent depositing substrates (fine sands, silts, and clays) with adequate sediment to assure collection of an ample amount of sediment for laboratory chemical analysis. In addition, professional judgement will be used while selecting sampling locations to avoid sampling obvious upstream point and non-point source discharges such as tank farms, major highways, and landfills. Each location will be described in a field record, noted on a topographic map, marked with a flagged stake, and located with two witness points. The field team will identify at least 25 potential sampling points. Figure 2-6 depicts the areas where sediment sampling locations will be reviewed and selected.

Following a mobilization period, WESTON will use a table of random numbers to select a total of 15 locations for sampling.

The use of a trained hydrologist to locate depositional regimes is not a departure from the principle of using random sampling, but rather a reflection of the fact that sediment is not ubiquitous. The ultimate determination of prospective sediment locations to be sampled will be provided by a random number table.



NOTE:

* CALENDAR MONTHS WILL BE FILLED IN UPON APPROVAL OF THE QAPP/FSP.

FIGURE 2-3

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

ANTICIPATED PROJECT SCHEDULE
SOIL/SEDIMENT BACKGROUND
CPAH DETERMINATIONS
MOSS-AMERICAN SITE
Milwaukee, Wisconsin

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Phase II sediment sampling will include collection of sediment samples in upstream tributaries of the subject reaches of the Little Menomonee River, in a similar manner as described for Phase I. While the U.S. EPA has recognized the importance of collecting this data, any decision or agreement on the use of this data in an MPB determination or as a cleanup standard for downstream reaches of the Little Menomonee River has been deferred.

In previous work by U.S. EPA, stream segments have been delineated in approximately one mile lengths corresponding to major road bridge river crossings. The roadway crossings also coincide with the tributaries to the river, which are primarily roadside ditches and storm sewers.

The same approach to be applied upstream of the former facility will be used in investigating background CPAH sediment concentrations in the downstream segments. WESTON, U.S. EPA, and WDNR will assign specialists to examine every stream segment to identify tributaries. Based upon this survey, the three parties will settle upon candidate sample collection points. Locations selected will be outside of the influence of historic flooding events of the Little Menomonee River. As for Phase I, professional judgement will be used in selecting sample locations to avoid obvious upstream point source discharges. Ideally, each stream segment will offer at least 25 candidate sample collection locations. Locations will be identified, recorded, and marked using the same procedure described for sediment sampling upstream of the facility. If available, 10 locations will be selected for sampling in each segment using a table of random numbers.

To summarize, the sediment sampling program will be conducted in two phases:

- Phase I: 15 sediment samples will be collected from locations upstream of the facility in the main channel of the Little Menomonee River.

- Phase II: If implemented in full, 10 sediment samples will be collected for each background setting (Reach 2, Reach 3, Reach 4, and Reach 5) from the non-site-related tributaries to the Little Menomonee River.

Samples will be handled using established techniques and analyzed using a method defined in the approved QAPP. Depending on the concentrations determined in the first phase investigation, it may be possible to use alternate laboratory method(s) to analyze second phase sediment samples. This is discussed in Section 8 of the accompanying QAPP. The analytical data will be used to calculate the MPB for each stream segment. Analysis of variance (ANOVA) testing may be conducted to determine if there are statistically significant ($P \leq .05$) differences in the concentrations of CPAH between stream segments.

Table 2-1 presents a summary of the sediment background sampling effort for the facility.

SECTION 3

FIELD SAMPLE COLLECTION PROCEDURES

3.1 BACKGROUND SOIL

As many as 83 background soil samples (including field duplicates) may be collected as a part of the Predesign Task 2 work for the facility. A composite sample will be collected to a depth of 12 inches below ground surface at grid locations selected at random. If the designated sample grid location cannot be hand-excavated due to access restrictions, structures, or other obstacles, the field sampler will move to the closest place suitable for soil sample collection and dig there. Using a decontaminated shovel, the top 12 inches of soil will be spaded to loosen the soil stratum from which the sample will be withdrawn. If the soil to be sampled is particularly hardened, a freshly decontaminated pick will be used to loosen a volume sufficient for sampling. A decontaminated stainless steel scoop will be used to withdraw the soil sample from the loosened area. The sample will be homogenized in accordance with procedures in Subsection 3.4 and then placed in the required sample container(s).

3.2 BACKGROUND SEDIMENT

As many as 61 background sediment samples (including field duplicates) may be collected as part of the Predesign Task 2 study. The samples will be collected from a variety of types of locations, ranging from submerged river bottoms to dry catch basins. A brief description of methods to collect sediment samples in likely locations follows; however, the field sampler would be expected to exercise judgement and display ingenuity in obtaining sediment samples.

Submerged River, Tributary, or Ditch Sediment

Using a decontaminated core sampler the field technician will remove sediment samples from the designated bottom location and place them in a decontaminated stainless steel bowl. This process will be repeated until an adequate volume of sample material is obtained.

Sampling of sediments within the river shall proceed to a depth where the "hardpan" layer is first encountered. This sediment sampling depth may be 3 to 4 feet or as little as a few inches, depending on sampling location within the river. A composite sample will be collected from the entire depth of sediment core.

Sediment sampling in streams, rivers, and ditches with flowing water will progress from downstream to upstream with the farthest downstream location sampled first and the most upstream location sampled last. This will minimize any cross-contamination between sediment locations that could result from the disturbance of the sediment.

The processes of sample homogenization and equipment decontamination are described in Subsections 3.4 and 3.5, respectively.

3.3 FIELD QUALITY CONTROL SAMPLES

Two types of quality control (QC) samples will be collected during the pre-design background sampling activities:

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

The purpose behind each QC sample is explained in Subsection 4.1 of the QAPP. The specific level of QC effort for the Moss-American Site activities is presented in Table 2-1, and the sample collection procedures for each QC sample are detailed below in Subsections 3.3.1 and 3.3.2.

3.3.1 Field Duplicate Samples

Field duplicate samples will be collected at select locations during soil and sediment sampling on a 1 per 10 sample (or less) basis for each sample matrix using procedures identical to those for the investigative samples of the same matrix. Field duplicate samples will be analyzed for the same parameters as the investigative samples. At the location where a field duplicate sample will be collected, the field sampler will collect sufficient sample material for both the investigative and duplicate sample. After the entire volume of material has been collected and homogenized as described in Subsection 3.4, the field sampler will alternately fill sample bottles for the investigative sample and the duplicate sample until all sample containers for each sample are filled.

3.3.2 Matrix Spike/Matrix Spike Duplicate Samples

Matrix spike/matrix spike duplicate (MS/MSD) samples will be collected on a 1 per 20 sample (or less) basis for both soil and sediment samples. They are not additional samples, but instead investigative samples assigned for MS/MSD analysis. Therefore, all sample collection procedures are identical to those for other investigative samples of the same matrix (i.e., soil and sediment). No additional sample volume is required for either

MS/MSD soil or sediment samples. Each MS/MSD sample will be identified as such on the sample chain-of-custody form and will be shipped to the analytical laboratory for all scheduled analyses.

3.4 SAMPLE HOMOGENIZATION PROCEDURES

The homogenizing procedure is designed to increase the probability that the relatively small sample aliquot is representative of the relatively large soil/sediment volume removed from the sample location, thereby enhancing the representativeness and reproducibility of the soil sample. The soil will be placed in a decontaminated stainless steel bowl or tray, and a decontaminated stainless steel spoon or spatula will be used to break up the soil into pieces approximately 1/2 inch or less in diameter. The soil pieces will then be stirred using decontaminated spoons or spatulas so that all of the soil at the bottom of the tray or bowl is displaced to the top and vice versa. This action will be repeated at least three times. The homogenizing process will be considered complete when the texture and color of the soil appear uniform throughout. The homogenization procedure will be followed for all samples, regardless of appearance, in order to ensure consistency unless stated elsewhere in this document. Any water that is collected with a sediment sample will not be decanted prior to undergoing sample homogenization.

3.5 DECONTAMINATION REQUIREMENTS

All reusable digging and sampling equipment, including the shovel, pickaxe, core sampler, Ponar sampler, Ekman grab, stainless steel spatulas, spoons, bowls and trays, and other sediment sampling equipment, will be decontaminated between collection of each soil/sediment sample according to the procedures outlined in Table 3-1.

3.6 ANALYTICAL METHODS

Section 8 of the QAPP discusses the analytical methodology by which Moss-American background soil and sediments will be analyzed. Table 2-1 summarizes the sampling effort for all investigative and QC samples.

Table 3-1

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

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

Note: The water-based drippings from decontamination will be left to fall on the ground (because there is no reason to expect contamination in the background samples) unless otherwise directed by the U.S. EPA or WDNR.

* Any retained drippings will be containerized in a drum or other equivalent storage vessel, staged on site with the RI wastes and properly disposed at an appropriate disposal facility following the completion of all predesign field work.

SECTION 4

SAMPLE NUMBERING SYSTEM

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

Two identification numbers will be used for each background soil and sediment sample; these are a WESTON project sample number and an analytical laboratory sample identifier.

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

4.1 PROJECT SAMPLE NUMBERING SYSTEM

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

- Project Identifier. A three-character designation will be used to identify the facility for which the samples will be collected. For this project, it will be MA1. MA stands for Moss-American Site, and the numerical designation (1, 2, 3...) refers to the phase of the project.

- Sample Type and Location. A two-character type code (SS for soil and SD for sediment) followed by a one-character, two-digit locus code followed by a four-digit coordinate code will indicate sample type and location. For QC samples, the four-digit coordinate code will be followed by "D" for field duplicate sample and by "M" for matrix spike/matrix spike duplicate sample. (It should be noted that all field duplicate samples will be submitted "blind" to the laboratory. Only field personnel will be acquainted with the sample nomenclature system.)

- Sequence. For soil and sediment samples, a two-digit number will be used to indicate the first, second, third, etc., sample collected at a given location during a particular phase of the project. Some examples of the project sampling number system are as follows:

Soil

- MA1-SSG15-0304-01 reads as
  - Moss-American Site.
  - Phase I Predesign Study.
  - Surface Soil Grid Number 15.
  - Grid coordinates X=03, Y=04.
  - First sample at this location.
- MA1-SSG15-0304D-01 reads as
  - Duplicate of first sample example.
- MA1-SSG15-0304M-01 would be the sample identifier if the first sample example was an MS/MSD sample.

Sediment

- MA1-SDL04-0019-01 reads as
  - Moss-American Site.
  - Phase I Predesign Study.
  - Sediment Locus Number 4.
  - Location number 19 of n candidate locations.
  - First sample at this location.

4.2 LABORATORY SAMPLE IDENTIFIER

The laboratory identifier will be an eleven-digit number in the following format: YYMMLBBB-XXX, where YYMMLBBB is the batch number, and

YYMM	=	Year/month (e.g., 9104).
L	=	Laboratory identifier (e.g., L = lab name).
BBB	=	A computer-assigned consecutive batch number which rolls over after 999 to 001.

XXX = A consecutively assigned sample number unique to a specified field sampling point.

Upon arrival at the laboratory, the WESTON batch number will be recorded by the laboratory custodian/sample log-in person on the chain-of-custody form and on the bottle label using a permanent marker.

SECTION 5

SAMPLE HANDLING

5.1 SAMPLE CONTAINERS AND SAMPLE PRESERVATION

All soil and sediment samples are expected to be low hazard level. Table 5-1 lists the required sample containers, sample volumes, sample preservation requirements, and holding times associated with all parameters and media applicable to the Moss-American Site predesign background sampling activities.

5.2 SAMPLE PACKAGING AND SHIPMENT

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

- Each sample container will be checked to ensure that the container lid is securely tightened.

- Each sample container will be checked to ensure that the sample label has been securely affixed to the container and completely/correctly filled out with the appropriate sample ID number, sample date, sample time of collection, and analytical parameters as a minimum requirement.

- Each container will be placed in a separate zip-lock bag and the bag securely closed (eliminating most of the air from within the bag).

- The low concentration samples will be placed in a cooler lined with a large polyethylene bag. Enough vermiculite or equivalent absorbent material will be packed around the samples to minimize the possibility of container breakage. The temperature will be maintained at 4° C with cold packs or ice, sealed in plastic bags. The remaining space in the cooler will be filled with additional packing material and the large polyethylene bag sealed.

Table 5-1

Required Sample Containers, Volumes, Preservation, and Holding Times
Moss-American Site
Milwaukee, Wisconsin

Material Type	Analysis	Sample Concentration Level	Number of Containers	Required Sample Volume	Sample Container Type	Sample Preservation	Sample Holding Time[b]
Soil/sediment	CPAH[a]	Low	1	8 oz.	8-oz. wide mouth mouth glass jar	Cool, 4 degrees C	14 days until extraction; analysis within 40 days

[a]CPAH - Carcinogenic polycyclic aromatic hydrocarbons. See Appendix B for the standard operating procedure for this analysis.
[b]The holding times are calculated from the date of sample collection.

All sample containers will meet or exceed the criteria specified in the U.S. EPA guidelines contained herein Appendix C.

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

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

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

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

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

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

- Site name.
- Number of samples shipped.
- Number of coolers shipped.
- Date samples were shipped.

- Date samples should be received.
- Shipment airbill number(s).

SECTION 6

SAMPLE DOCUMENTATION AND TRACKING

6.1 FIELD RECORDS

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

6.2 FIELD CHAIN-OF-CUSTODY PROCEDURES

Field chain-of-custody procedures are discussed in Subsection 6.1 of the QAPP.

6.3 SAMPLE DOCUMENTATION FORMS

The main sample documentation form for the Moss-American Site background sampling activity is the WESTON chain-of-custody form (also called the custody transfer record/lab work request form). In addition, as previously mentioned, chain-of-custody seals and sample container labels will be utilized. The important protocols associated with each of these is summarized below:

Chain-of-Custody Form

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

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

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

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

- The Field Team Leader or his designee will sign and date the chain-of-custody form as relinquisher of the samples.

Custody Seals

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

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

- The seal numbers will be recorded on the enclosed chain-of-custody form(s).

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

Sample Bottle Labels

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

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

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

Once the Field Sample Manager has turned over the sample paperwork to the Field Team Leader, it is the responsibility of the Field Team Leader to maintain all the paperwork and to be able to account for all forms at the end of field work.

SECTION 7

SAMPLING TEAM ORGANIZATION

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

SECTION 8

SAMPLE CONTAINER PROCUREMENT

All sample containers to be used during the Moss-American Site sampling program will be purchased by WESTON from a reputable supplier capable of providing the bottle quantity and type that meet or exceed the strict quality control requirements set forth by the U.S. EPA in OSWER Directive No. 9240.0-05, Specifications and Guidance for Obtaining Contaminant-Free Sample Containers, April 1990 (Appendix C). A written and/or verbal Invitation For Bid will be presented to suppliers such as Eagle Picher that will include a copy of the above-mentioned specification document. The supplier capable of providing all bottle supplies according to the specifications requested in a timely and cost-effective manner will be chosen to provide the Moss-American Site sampling containers. Alternatively, the sample containers will be procured from the analytical laboratory. Sample containers will be purchased on an as-needed basis and will be stored at the WESTON warehouse prior to the commencement of field work. WESTON's oversight personnel will record the bottle lot numbers associated with each sample collected during the Moss-American Site field sampling program.

It will be assured that the sample containers used for the Moss-American Site Predesign Task 2 sampling activities do not contain target organic and inorganic contaminants exceeding the levels specified in the abovementioned document. For analytes not contained in the U.S. EPA guidance document, the bottles will either be cleaned in the same way as for the similar types of analytes or it will be negotiated with the bottle supplier(s) to clean and test the bottles for analytes of interest to ensure that the contaminant levels of these analytes do not exceed approximately one-third of the required quantitation limits. Specifications for the bottles will be verified by checking the supplier's certified statement and analytical results for each bottle lot, and will be documented on a continuing basis. This data will be maintained in the project evidence file and will be available, if requested, for U.S. EPA review.

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

APPENDIX B

STANDARD OPERATING PROCEDURES FOR LOW CONCENTRATION ANALYSIS OF CARCINOGENIC POLYNUCLEAR AROMATIC HYDROCARBONS (INCLUDES METHOD VALIDATION REPORT)

ORGANIC ANALYSIS PROTOCOL POLYNUCLEAR AROMATIC HYDROCARBONS (PAH) IN SOIL/SEDIMENT BY CAPILLARY COLUMN GC/MS SELECTED ION MONITORING (SIM) TECHNIQUES FOR MOSS-AMERICAN SITE (KERR-McGEE)

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QA Standard Practice Records

REVISION NUMBER: 00

1.0 PURPOSE/APPLICATION

1.1 This method is designed for the determination of polynuclear aromatic hydrocarbons (PAH) in soil and sediment. The ensuing table lists the analytes determined by this method.

COMPOUND	CAS No.
benzo(a)anthrancene	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
dibenz(a,h)anthracene	53-70-3
benzo(g,h,i)perylene	191-24-2

1.2 The practical quantitation limit (PQL) of this method for the determination of an individual compound is 2 ng/g for soil and sediment. PQLs for a

specific sample may be different from that listed depending upon the nature of interferences in the sample matrix, percent moisture, and dilutions required for analysis.

2.0 REFERENCES

2.1 EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, November 1986:

Method 3540, Soxhlet Extraction,
Method 3611, Alumina Column Cleanup and Separation of Petroleum Wastes,
Method 8270, GC/MS for Semivolatile Organics: Capillary Column Technique
Method 8280, The Analysis of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans

2.2 EPA Method 1625 Revision B, "Semivolatile Organic Compounds by Isotope Dilution GC/MS", January, 1985.

3.0 SUMMARY OF METHOD

3.1 A measured amount of sample (10 g for soil and sediments) is extracted with methylene chloride using a Soxhlet extractor. The methylene chloride extract is concentrated to a volume of 1 mL. Internal standards are then added and a 2 μL aliquot is injected for GC/MS analysis.

3.2 The method provides selected column chromatographic cleanup procedures to aid in the elimination of interferences that may be encountered.

3.3 The method specifies the use of a capillary column gas chromatograph (GC) interfaced to a mass spectrometer (MS) operated in selected ion monitoring (SIM) mode. Data is acquired utilizing SIM descriptors which are switched in sequence according to retention time data derived from a calibration standard.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated backgrounds at the masses (m/z) monitored. All of these

materials must be routinely demonstrated to be free from interferences under the conditions of the analyses by running laboratory reagent blanks.

4.1.1 Glassware must be scrupulously cleaned to ensure low detection limits. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Refer to Appendix A, Operating Practice (OP) No. 21-16-0001, for detailed cleaning instructions.

4.1.2 After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

NOTE: Volumetric glassware should not be heated in a kiln.

4.1.3 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedures in Section 10.3 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches (i.e., EPA Method 3630 Silica Gel Cleanup and/or Gel-Permeation Cleanup as per WESTON OP No. 21-16-3640.1) to eliminate false positives and achieve the PQL listed in Section 1.2.

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDS) should also be made available to all personnel involved in the chemical analysis.

5.2 A fully fastened lab coat, latex gloves, and safety glasses should be worn whenever working with samples, extracts, or standards. All chemical containers should be properly labeled according to "Right-To-Know" guidelines.

5.3 The following analytes covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: benzo(a)anthracene, benzo(a)-pyrene, and dibenzo(a,h)anthracene. Primary standards of all toxic compounds should be prepared in a hood.

6.0 APPARATUS AND MATERIALS

6.1 Glassware and Supplies

6.1.1 Soxhlet Continuous Extraction Device.

6.1.2 40 mL VOA vials with teflon lined caps.

6.1.3 Evaporative Flask, Kuderna-Danish: 500 mL.

6.1.4 Concentrator Tubes, Kuderna-Danish: 10 mL. Attach to K-D flask with plastic clips.

6.1.5 Snyder Column, Kuderna-Danish: three ball.

6.1.6 Vials: 12 and 16 mL with Teflon®-lined screw cap.

6.1.7 Disposable Pipets: 5 ¾" pasteur.

6.1.8 Teflon® Boiling Chips: wash with methylene chloride prior to use.

6.1.9 Nitrogen Blowdown Apparatus: N-Evap® Analytical Evaporator Model 111, Organomation Associates Inc., Northborough, Massachusetts or equivalent. Tygon® tubing or equivalent and gas regulator is required.

6.1.10 Filter Paper: Whatman No. 41, or equivalent.

6.1.11 Water bath: heated, with concentric ring cover, capable of maintaining temperature 60-100°C. The bath must be used in a well ventilated hood.

6.1.12 Glass Funnels: wide mouthed.

6.1.13 Heating Mantle.

6.1.14 Analytical Balance: capable of accurately weighing $\pm$0.01 g.

6.1.15 Glass wool: baked at 400°C for a minimum of 4 hours before use.

6.1.16 Assorted Class A Volumetric Flasks: including 5, 10, and 100 mL.

6.1.17 Chromatography column: 300 mm X 10 mm ID, with pyrex glass wool at bottom and teflon stopcock.

6.1.18 250 mL beakers.

6.1.19 250 mL Erlenmeyer flasks.

6.1.20 Aluminum weighing dish.

6.1.21 500 mL flat bottom flasks.

6.2 Gas Chromatograph/Mass Spectrometer (GC/MS) System

6.2.1 Gas Chromatograph: An analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gasses. The injection port must be designed for splitless injection onto capillary columns. The column should be inserted directly into the source of the MS.

6.2.2 Capillary Column: 30 m long x 0.32 mm ID fused silica DB-5 with 0.25 μm film thickness. Refer to Table 2 for complete operating conditions.

6.2.3 Mass Spectrometer: Low resolution mass spectrometer capable of scanning masses up to 500 amu with a cycle time of 1 second or less in the electron impact mode. The MS must be equipped with a 70 eV (nominal) ion source and be capable of acquiring m/z abundance data in real time selected ion monitoring for groups of two or more masses with cycle time of 1 second or less.

6.2.4 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all data obtained for the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. The SIM data acquired during the chromatographic program is defined as the Selected Ion Current Profile (SICP). Software must also be available that allows integrating the abundances in any SICP between specified time or scan-number limits, as well as performing routine calculations such as: response factor (RF), relative retention time (RRT), amount detected (see Section 13).

7.0 REAGENTS

7.1 Sodium Sulfate: granular, anhydrous. Purify by heating at 400°C for 4 hours in a shallow tray.

7.2 Alumina: neutral, 80/200 mesh (Woelm-Super A or equivalent). Dry Alumina overnight at 130°C prior to use.

7.3 Sodium Hydroxide Solution: 0.5 N.

7.4 Stock Standard Solutions: Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions. Methylene chloride (dichloromethane; DCM) is used as solvent for all solutions. Refer to Appendix B for standard preparation.

7.5 Methanol, Acetone, Methylene Chloride, Hexane: pesticide quality or equivalent.

7.6 Prepurified nitrogen gas.

8.0 CALIBRATION

8.1 Using stock standards, prepare calibration standards that will allow measurement of relative response factors (RRFs) for five concentration ratios of each analyte of interest relative to internal standards. Internal standards, surrogates and analytes are listed in Table 1. All solutions should be discarded six months after the date prepared.

8.2 Initial Calibration (ICAL): Tune the instrument and establish operating conditions as described in Section 11.1. Using a 2 μL injection, analyze each calibration standard. Tabulate area responses against concentration for each compound and internal standard. Calculate RRFs for each analyte and surrogate.

$$RRF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

A_s = Area of quantitation ion for compound of interest.
A_{is} = Area of quantitation ion for internal standard.
C_{is} = Concentration of the internal standard, ng/mL.
C_s = Concentration of the compound of interest, ng/mL.

If the RRF value over the working range is a constant ($\leq$25% RSD), the RRF can be assumed to be invariant and the RRFs for the middle concentration will be used for calculations for the remainder of the 12-hour period.

If the RSD is greater than 25.0% or if any RRF is less than 0.25, the calibration may not be used.

8.3 Continuing Calibration: The RRFs must be verified on each working day by measurement of the middle level calibration standard. If the resulting RRFs vary from the average RRF determined for the initial calibration for the corresponding compound by more than $\pm$ 30.0% difference (%D) or if the daily RRF for any single compound is less than 0.25, the test must be repeated using a freshly prepared calibration standard. If %D criteria still fail, a new initial calibration must be analyzed. If minimum RRF criteria still fail, the instrument or GC column requires service.

8.4 The injection of the first initial calibration standard or the continuing calibration standard initiates a 12-hour analytical period. The instrument is considered calibrated for 12 hours from the time of this first injection, and data for any samples injected during this period will be considered valid.

9.0 QUALITY CONTROL

9.1 Before processing any sample, the analyst must demonstrate through the analysis of a method blank that all glassware and reagents are interferant-free

at the method detection limit of the matrix of interest. Each time a set of samples is extracted, or there is a change in reagents, a method blank must be processed to provide an indicator for potential laboratory contamination.

A laboratory method blank must be run along with each extraction batch of 20 or fewer samples. A method blank is performed by executing all of the specified extraction and cleanup steps, except for the introduction of a sample. The method blank is also dosed with a surrogate solution (see Section 9.3). Sodium sulfate will be used as the method blank medium for soil and sediment matrices.

Similarly, a spiked blank will be prepared with each extraction batch as an internal control to help identify matrix versus procedural/instrumental causes for poor recoveries in spiked samples. If acceptable recoveries are demonstrated by the matrix spike/matrix spike duplicate then the blank spike will not be reported. The blank spike will only be used as needed for diagnostic purposes.

9.2 The laboratory will analyze performance evaluation samples as provided by Kerr/McGee. Additional sample analysis will not be permitted if the performance criteria are not achieved. Corrective action must be taken and acceptable performance must be demonstrated before sample analyses may resume.

9.3 Each sample will be dosed with two surrogates (Table 1) just prior to the extraction process. Surrogate recoveries are used to assess method performance; samples with surrogate recoveries of less than 50% or greater than 120% will be re-extracted and re-analyzed if it is determined that the outliers are not due to matrix effects. Data will be flagged to indicate that accompanying QC did not meet criteria.

9.4 Matrix spikes (MS) and matrix spike duplicates (MSD) will be analyzed at a rate of one per 20 samples of the same matrix. All analytes will be spiked at a level of 50 ng/g. If higher background levels are consistently encountered, the spike level will be adjusted. The QC limits for the MS/MSD recoveries are 50-150%. The precision goal, expressed as relative percent difference (RPD) is 50%. Recovery and/or RPD outliers will be evaluated and flagged on a case by case basis. If it is determined that the outliers are a result of lab error, the batch will be re-extracted and re-analyzed.

9.5 Individual chromatographic runs will be evaluated on a case by case basis for evidence of carryover. Corrective action (e.g., re-analysis, insertion of blanks, etc.) will be performed as appropriate.

10.0 EXTRACTION AND CLEAN-UP PROCEDURES

10.1 Extraction of Soil and Sediment: Record all extraction information in a bound logbook and label glassware accordingly. Rinse all glassware with acetone and DCM and dispose of washes properly. Decant any obvious liquid layer and stir the sample to ensure homogeneity. Dispose of the liquid in a safe manner. Weigh 10 grams of sample into a tared glass jar and record the weight to the nearest tenth of a gram. Add an equivalent amount of granular anhydrous sodium sulfate, or enough to give the sample a dry consistency.

Fill a 500 mL roundbottom flask approximately two thirds (⅔) full with DCM. Add a few boiling chips. Stopper the bottom of a Soxhlet extractor with glass wool and attach the Soxhlet to the roundbottom flask. Place the sample into the Soxhlet and label properly. Include an extra method blank to be spiked (blank spike), and a sample in triplicate at a 5% frequency for MS/MSD spiking. Add 100 μL of surrogate to each investigative and QC sample, and add 100 μL of spike solution to the blank spike (BS), MS, and MSD. Reflux the system for a minimum of 16 hours.

After the system has refluxed and cooled, quantitatively transfer the extract into a K-D through a glass funnel lined with filter paper containing sodium sulfate. Rinse the roundbottom flask with DCM to insure a quantitative transfer. Add a few boiling chips and a 3-ball Snyder column to the K-D. Concentrate the extract on a water bath at 90°to 100°C to an apparent volume of 10 mL.

Dispose of the remaining soil in a fiber waste drum.

Proceed to the cleanup procedures in Section 10.3.

10.2 Determination of Percent Solids:

Decant any obvious liquid layer and stir the sample to ensure homogeneity. Dispose of the liquid in a safe manner. Determine the weight of an aluminum weighing dish to the nearest tenth of a gram and record it in a

bound notebook. Add approximately 10 grams of sample to the dish and record the pan + sample weight (again, to the nearest tenth of a gram). Subtracting the weight of the pan will give the wet weight of the sample. Place the dish in an oven (in a hood!) at 105°C for a minimum of 12 hours. Re-weigh and, subtracting the weight of the pan as before, determine the dry weight of the sample.

Calculation: $\frac{\text{Weight of dry sample (g)}}{\text{Weight of wet sample (g)}} \times 100\% = \%\ \text{Solids}$

% moisture (reported on Form 1) = 100% - %Solids

10.3 Cleanup Procedures:

For maximum PAH recovery, the samples must be extracted with DCM and boiled down without solvent exchange in the K-D apparatus. Solvent exchange is to be performed in a 16 mL vial without much heating. Care must be taken during column chromatography to avoid UV irradiation. This can be accomplished by covering columns with foil, or dark glass columns can be employed.

10.3.1 Transfer the extract from the concentrator tube (in DCM) to a 16 mL vial (vial A) using a disposable pipet. Rinse the concentrator tube with DCM and add to vial A to ensure a quantitative transfer.

10.3.2 Solvent exchange to hexane: Rinse the tip of the blow down apparatus with DCM before use. Blow down the extract to about 3 mL with nitrogen. The Reacti-Therm heater setting must never exceed 3.5 on low! Add about 4 mL of hexane to the 16 mL vial A and mix it well. Again blow down to about 3 mL.

10.3.3 Base wash: Bring up the volume of the extract to about 5 mL by adding hexane. Add 2.5 mL of 0.5 N NaOH to vial A. Cap and shake the vial vigorously for 1 minute. Wait for the two layers to separate. Transfer the top layer to a clean 12 mL vial (vial B). A centrifuge may be necessary to assist in separating layers. Add 3 mL of hexane to vial A. Cap and shake for 30 seconds. Again, transfer the top layer to vial B. Blow the extract in vial B down to about 1 mL with nitrogen. Dispose of the remaining base fraction.

10.3.4 Alumina Column Cleanup

Pack a chromatography column with a glass wool plug. Prewash the chromatography column with methanol, acetone, DCM, and hexane. Fill the glass chromatographic column to about 20 cm with hexane. Weigh out 10.0 g of alumina and add the alumina to the column. Gently tap the column to distribute the alumina evenly to minimize chromatographic voids. Alternatively, a slurry of alumina in hexane may be used to pack the column. Allow the alumina to settle and then add 1.0 g of anhydrous sodium sulfate on top of the alumina. Elute the column with 50 mL of hexane. Let the solvent flow through the column until the head of the liquid in the column is just above the sodium sulfate layer. Discard the eluate. Close the stopcock to stop the solvent flow. Transfer 1.0 mL of sample extract onto the column. Rinse out the extract vial with 1 mL hexane and add it to the column immediately. To avoid overloading the column, it is suggested that no more than 300 mg of extractable organics be placed on the column. Just prior to exposure of the sodium sulfate to the air, elute the column with a total of 15 mL of hexane. If the extract is in 1 mL of hexane, and if 1 mL of hexane was used as a rinse, then 13 mL of additional hexane should be used. Collect this fraction in a 16 mL vial, label as prewash and save. Next, elute the column with 100 mL of methylene chloride and collect the eluate in a 250-mL flask. Label this fraction PAHs. Elute the column with 40 mL DCM and collect the eluate in a 40 mL VOA vial. Label this postwash and save. Concentrate the PAH extracts from the 250-mL flask using standard K-D and N_2 blowdown techniques to a volume of 1.0 mL.

NOTE: Some extracts (viscous or very dark) may be difficult to blow down to 1 mL. In these cases, a final volume of 5 or 10 mL (as appropriate) may be used.

CAUTION: Never blow down the extract to less than 0.5 mL at any stage of cleanup, as analytes may be lost!

11.0 GC/MS ANALYSIS

11.1 The mass spectrometer will be calibrated with perfluorophenanthrene (FC-5311) before each 12 hour analytical period to ensure correct mass assignment. Establish proper selected ion monitoring (SIM) windows by analyzing a calibration standard to determine retention times of analytes and

standards. Refer to Table 1 for SIM conditions and Table 2 for GC conditions.

11.2 Establish acceptable calibration according to Section 8.2 or 8.3.

11.3 Add internal standard mix (IS) to the sample extract prior to injection onto the GC/MS. Add 100 µL of internal standard solution for each 1 mL of sample extract. Refer to Appendix B for IS preparation.

11.4 If peaks above the calibration range are encountered, the extracts will be diluted to bring the largest peak within the calibration range. After dilution, additional internal standard mix will be added to the extract at the amount described in Section 11.3. The diluted extract will be analyzed in order to quantify large peaks (i.e., those that are above the calibration range). One result for each compound will be reported, with a maximum of two analyses per sample reported. Details of how the dilutions are prepared will be documented in the instrument run log.

12.0 IDENTIFICATION CRITERIA

A chromatographic peak will be identified as positive if it meets the following criteria:

12.1 The calculated retention time (RT) relative to the appropriate internal standard must be within ± 0.005 RRT units when compared to the respective target compound in the continuing calibration standard (or the middle standard of an initial calibration when the samples are analyzed within the same 12-hour period as the ICAL).

12.2 Peaks with proper relative retention time (RRT) occurring at masses monitored for a given compound must maximize simultaneously (±2 scans) and produce a signal at least 2.5 times background. If the peak at the confirmation mass is not 2.5 times background but meets all other criteria and, in the judgement of the GC/MS analyst the peak is positive, the compound can be quantified and reported as positive with an explanation written on the chromatogram, and a suitable flag qualifying any tabulated results (i.e., Form 1 and the data summary or spreadsheet).

The ratio between the quantitation and the confirmation mass (see Table 1) is used to assist the analyst in determining levels of interference. Confirmation masses are not used for quantitation purposes. If the confirmation to quantitation ratio is not within the range specified in Table 1 but, in the judgement of the GC/MS analyst the peak is positive, the compound can be quantified and reported as positive with a suitable flag qualifying any tabulated results (i.e., Form 1 and the data summary or spreadsheet).

13.0 CALCULATIONS

The abbreviations described for the equation in Section 13.1 carry through to Sections 13.2 - 13.3, as appropriate.

13.1 Concentrations are calculated according to the equation:

$$\text{PAH (ng/g)} = \frac{(A_c)(Q_{is})\,(V)}{(A_{is})(RRF)(W)(P)}$$

where:
- A_c = Area of target compound quantitation ion.
- A_{is} = Area of internal standard quantitation ion.
- P = % Solids ÷ 100
- Q_{is} = Amount (ng) of internal standard added.
- RRF = Relative Response Factor (Section 8.2)
- V = Volume of extract in mL (= dilution factor)
- W = Sample amount in grams.

13.2 Surrogate recoveries are calculated according to the equation:

$$\%\ \text{Rec} = \frac{(A_s)\,Q_{is}}{(A_{is})(RRF)(Q_s)} \times 100\%$$

where:
- A_s = Area of surrogate compound quantitation ion.
- Q_s = Amount (ng) of surrogate added.

13.3 Spike recoveries are calculated according to the equation:

$$\%\ \text{Rec} = \frac{(A_{sp})\,Q_{is}}{(A_{is})(RRF)(Q_{sp})} \times 100\%$$

where:
- A_{sp} = Area of spike compound quantitation ion.
- Q_{sp} = Amount (ng) of spike added.

13.4 The relative percent difference between MS and MSD analyses is calculated according to the following equation:

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

where: S = First Sample value (MS value)
D = Second sample value (MSD value)

14.0 DATA REPORTING

Quantitation reports from the GC/MS system will be transferred to WESTON's Laboratory Information Management System (LIMS) where calculations will be performed and final reports generated.

Typical semivolatile EPA CLP-type forms will be provided (i.e., Forms 1 through 7) in addition to a data summary and case narrative. Raw data (i.e., Selected Ion Current Profiles and Quantitation Reports) for all samples and standards will be included as per typical CLP deliverables requirements. Any problems encountered (e.g., poor surrogate or spike recoveries, interferences or unusual ion profiles) will be discussed in the case narrative.

TABLE 1
PAH COMPOUNDS

SIM DESC	QUAN REF	COMPOUND	CAS #	QUAN MASS	CONF. MASS	C/Q RATIO	SPIKE AMT. (ng)
H4	IS#1	Pyrene-d10 IS #1	1718-52-1	212.14	NA	NA	NA
H4	IS#1	Chrysene-d12 SS #1	1719-03-5	240.17	NA	NA	500
H4	IS#1	Benzo(a)Anthrancene	56-55-3	228.09	226.09	0.12-0.50	500
H4	IS#1	Chrysene	218-01-9	228.09	226.09	0.13-0.52	500
H5	IS#2	Benzo(b)Fluoranthene	205-99-2	252.09	126.05	0.04-0.18	500
H5	IS#2	Benzo(k)Fluoranthene	207-08-9	252.09	126.05	0.04-0.18	500
H5	IS#2	Benzo(a)Pyrene-d12 IS #2	63466-71-7	264.17	NA	NA	NA
H5	IS#2	Benzo(a)Pyrene	50-32-8	252.09	126.05	0.04-0.17	500
H6	IS#3	Indeno(1,2,3-cd)Pyrene	193-39-5	276.09	274.09	0.11-0.46	500
H6	IS#3	Dibenz(a,h)Anthracene-d14 SS #2	13250-98-1	292.17	NA	NA	500
H6	IS#3	Dibenz(a,h)Anthracene	53-70-3	278.09	279.09	0.12-0.50	500
H6	IS#3	Benzo(g,h,i)Perylene-d12 IS #3	93951-66-7	288.32	NA	NA	NA
H6	IS#3	Benzo(g,h,i)Perylene	191-24-2	276.09	274.09	0.11-0.46	500

IS = Internal Standard
SS = Surrogate Standard
NA = Not Applicable

TABLE 2
GC/MS OPERATING CONDITIONS

Mass Spec.:	0.75 sec/scan SIM acquisition (See Table 1)
Column:	30 m x 0.32 mm ID x 0.25 μm df DB-5 (J&W Scientific)
Carrier Gas:	Helium
Column Head Pressure:	13 psi
Injection:	Splitless (splitter opened after 1 minute)
Injection Volume:	2 μL
Injector Temperature:	280°C
Transfer Line Temperature:	250-300°C
Column Oven Temperature:	60°C for 1 minute 60°C to 240°C at 10°C/minute 240° to 300°C at 15°C/minute Hold at 300°C for the duration of the analysis (approximately 5 minutes)
Total Analysis Time:	approximately 27 minutes

APPENDIX A
ORGANIC ANALYSIS PROTOCOL
GLASSWARE CLEANING - ORGANICS

WESTON OP No. 21-16-0001
Revision Number: 01

1.0 PURPOSE

Establish procedures for cleaning analytical glassware to ensure that sample integrity is not violated by contaminated glassware.

2.0 PROCEDURE: EXTRACTABLES AND GENERAL PURPOSE GLASSWARE

2.1 Wash glassware with a phosphate-free detergent (e.g., Alconox). Rinse with tap water five (5) times and deionized water five (5) times.

2.2 Rinse with acetone (once). If the glassware still appears dirty, consult the Section Supervisor.

2.3 Rinse with hexane (once).

2.4 Kiln dry at 450°C for a minimum of four (4) hours.

APPENDIX B
PREPARATION OF STANDARDS

NOTE: All solutions are prepared in Class A volumetric flasks.

1.0 PREPARATION OF INTERNAL STANDARD SOLUTION

1.1 Purchase the following deuterium labeled PAH surrogate cocktail mixture: Cambridge Isotope Laboratories (CIL) Catalog No. ES-2044.

This mixture contains the following at 200 µg/mL in dichloromethane-d_2/ methanol-d_4 (1:1):

Pyrene-d_{10}	(0.98%)
Benzo(a)pyrene-d_{12}	(0.98%)
Benzo(g,h,i)perylene-d_{12}	(0.98%)

1.2 Dilute 1 mL of CIL solution ES-2044 to 10 mL with methylene chloride to make an Internal Standard (IS) Stock Solution at 20 µg/mL.

$$1\ mL \times \frac{200\ \mu g}{mL} \times \frac{1}{10\ mL} = \frac{20\ \mu g}{mL}$$

1.3 Dilute the 20 µg/mL IS Stock by 20x with methylene chloride to make an Internal Standard Working Solution at 1 µg/L.

example: $0.5\ mL \times \frac{20\ \mu g}{mL} \times \frac{1}{10\ mL} = \frac{1\ \mu g}{mL}$

1.4 Add the 1 µg/mL IS Working Solution to all sample extracts and standards at a rate of 100 µL per 1 mL of extract/standard. This results in 100 ng of each IS added to 1 mL.

1.5 Other convenient dilutions may be used to reach the final Working Solution concentration of 1 µg/mL.

2.0 PREPARATION OF SURROGATE SPIKING SOLUTION

2.1 Purchase dibenz(a,h)anthracene-d_{14} (CIL Cat. No. DLM-677, D_{14} = 97%) as a pure solid. Weigh approximately 10 mg of the dibenz(a,h)anthracene-d_{14} to the nearest 0.1 mg in a 10 mL Class A volumetric flask and dilute to volume with methylene chloride (final concentration = 1000 µg/mL).

2.2 Purchase chrysene-d_{12} as a 1000 µg/mL solution in methylene chloride.

NOTE: Future purchases of chrysene-d_{12} solution will probably be Supelco Cat. No. 4-8416M at 2000 µg/mL and will require different dilutions to make a Working Standard.

2.3 Dilute 1 mL of each solution above (Sections 2.1 and 2.2) to 10 mL with methylene chloride to make a Surrogate Standard (SS) Stock Standard at 100 µg/mL:

$$1\ \text{mL} \times \frac{1000\ \mu\text{g}}{\text{mL}} \times \frac{1}{10\ \text{mL}} = \frac{100\ \mu\text{g}}{\text{mL}}$$

NOTE: If the solution in Section 2.2 is not exactly 1000 µg/mL, adjust the volume used accordingly.

2.4 Dilute the 100 µg/mL SS Stock by 20x with methylene chloride to make a Surrogate Standard Spiking Solution at 5 µg/mL:

example: $1.25\ \text{mL} \times \frac{100\ \mu\text{g}}{\text{mL}} \times \frac{1}{25\ \text{mL}} = \frac{5\ \mu\text{g}}{\text{mL}}$

2.5 Add the 5 µg/mL SS spike solution to all samples and blanks before extraction at a rate of 100 µL per sample. This results in 500 ng of each SS added to each 10 g sample aliquot.

2.6 Other convenient dilutions may be used to reach the final SS spike solution concentration of 5 µg/mL.

3.0 PREPARATION OF MATRIX/BLANK SPIKING SOLUTION

3.1 Purchase Supelco Polynuclear Aromatic Hydrocarbon Mix, Catalog No. 4-8905 or equivalent. This mix contains all analytes of interest at 2000 µg/mL in methylene chloride/benzene (1:1). If equivalent mixes are not at this concentration, adjust the ensuing directions/dilutions as necessary.

COMPOUND*	CAS No.	Concentration
benzo(a)anthrancene	56-55-3	2000 µg/mL
chrysene	218-01-9	2000 µg/mL
benzo(b)fluoranthene	205-99-2	2000 µg/mL
benzo(k)fluoranthene	207-08-9	2000 µg/mL
benzo(a)pyrene	50-32-8	2000 µg/mL
indeno(1,2,3-cd)pyrene	193-39-5	2000 µg/mL
dibenz(a,h)anthracene	53-70-3	2000 µg/mL
benzo(g,h,i)perylene	191-24-2	2000 µg/mL

* additional PAH compounds are also present

3.2 Dilute 1 mL of the above 2000 µg/mL solution to 10 mL with methylene chloride to make an Analyte Stock Solution at 200 µg/mL:

$$1\ \text{mL} \times \frac{2000\ \mu\text{g}}{\text{mL}} \times \frac{1}{10\ \text{mL}} = \frac{200\ \mu\text{g}}{\text{mL}}$$

3.3 Dilute the 200 µg/mL Analyte Stock by 40x with methylene chloride to make a Matrix/Blank Spiking Solution at 5 µg/mL:

example: $$0.25\ \text{mL} \times \frac{200\ \mu\text{g}}{\text{mL}} \times \frac{1}{10\ \text{mL}} = \frac{5\ \mu\text{g}}{\text{mL}}$$

3.4 Add the 5 µg/mL Matrix/Blank Spiking Solution to the required samples and/or blanks at a rate of 100 µL per sample. This results in 500 ng of each spike compound (i.e., each analyte) added to the designated MS/MSD sample aliquots and BS designated blanks.

3.5 Other convenient dilutions may be used to reach the final analyte concentration of 5 µg/mL in the Matrix/Blank Spiking Solution.

4.0 PREPARATION OF CALIBRATION STANDARDS

4.1 Calibration Standard will be prepared from the IS and SS Stocks (prepared in Sections 1.2 and 2.3) and an Analyte Stock (Section 3.2) different than the stock used for the preparation of the Matrix/Blank Spiking Solution.

4.2 Any convenient serial dilutions may be used to make the solutions below. If a particular direct series is adopted, it will be documented. Otherwise, refer to the Standard Prep Log ID Number to determine the exact sequence used for a particular stock.

4.3 A "modular" approach is used to prepare the calibration standards to allow a given component (IS, SS, or analyte) to be changed and easily checked verses the other components.

4.4 Prepare a series of five solutions of surrogate compounds and a separate series of five of solutions of analyte mix at the following concentrations in methylene chloride: 40, 100, 400, 1000, 4000 ng/mL.

4.5 Add the corresponding surrogate and analyte solutions together at a 1:1 ratio to make the Calibration Standards at the following concentrations:

Add 1 Part Surrogate Solution (ng/mL)	plus 1 Part Analyte Solution (ng/mL)	Resultant Calibration Standard (ng/mL)	Shorthand Calibration Standard ID
40	40	20	CC1
100	100	50	CC2
400	400	200	CC3
1000	1000	500	CC4
4000	4000	2000	CC5

The 200 ng/mL solution will be used as a Continuing Calibration Standard.

4.6 Before analysis, 100 μL of IS Working Solution (1 μg/mL) will be added to 1 mL of each Calibration Standard (or other similar ratio such as 10 μL to 100 μL, etc.). This will simulate the addition of 100 μL IS Working Solution to a 1 mL sample extract.

APPENDIX C

SPECIFICATIONS AND GUIDANCE FOR OBTAINING CONTAMINANT-FREE SAMPLE CONTAINERS

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MAY 2 [illegible]

MEMORANDUM

SUBJECT: Revision of "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers"

FROM: Joan F. Fisk, Chief,
Analytical Methods Implementation Section,
Analytical Operations Branch,
Hazardous Site Evaluation Division (OS-230)

TO: Addressees

In September, 1989 you received OSWER Directive #9240.0-05 from Henry Longest II with the memorandum titled "Decentralization of the Superfund Bottle Repository Functions". The purpose of this transmittal is to provide you with a revised version of the "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers" that addresses problems brought up once the original document was put into use. This revised version has been through extensive review provided by the Regions through the Contract Laboratory Technical Project Officers who circulated the draft for comments.

The Analytical Operations Branch plans to transmit this document formally with an amended directive number, but since we have had so many urgent requests for it, we decided that this early distribution to you would be of great assistance in your procuring of bottles. We would appreciate any comments that you have as soon as possible, so that if we have overlooked any deficiencies we can remedy them prior to the transmittal as a directive.

Addressees:

Contract Laboratory Program Technical Project Officers
Regional Sample Control Centers
Superfund Branch Chiefs

cc:

Director, Waste Management Division
Regions I, IV, V, VII, VIII
Director, Emergency and Remedial Response Division
Region II
Director, Hazardous Waste Management Division
Regions III, VI
Director, Toxic and Waste Management Division
Region IX
Director, Hazardous Waste Division
Region X
Director, Environmental Services Division
Regions I-X
Joan Barnes, AOB
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Bill Topping, PCMD
Lloyd Guerci, OWPE
Susan Bromm, OWPE
Russ Wyer, HSCD
Hans Crump-Wiesner, ERD
Penny Hansen, SAB

SPECIFICATIONS

AND

GUIDANCE

FOR OBTAINING

CONTAMINANT-FREE SAMPLE CONTAINERS

APRIL, 1990

TABLE OF CONTENTS

SECTION I

INTRODUCTION

In August 1989, the Environmental Protection Agency's (EPA) Office of Emergency and Remedial Response (OERR) decentralized Superfund's Sample Container Repository program (OSWER Directive #9240.0-05). In conjunction with the decentralization of Superfund's bottle program, OERR issued initial "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers" (August 1989) to assist the Regions in obtaining appropriate sample containers from commercially available suppliers.

This document revises the initial specifications and provides a single source of standardized specifications and guidance on appropriate cleaning procedures for preparing contaminant-free sample containers that meet all Contract Laboratory Program (CLP) detection/quantitation limits, including those for newly established low concentration analyses. Although the specifications and guidance procedures contained in this document are based on CLP low concentration requirements, they also are suitable for use in other analytical programs. Specific needs of EPA Regions will dictate which cleaning procedures are used by the designated bottle preparer.

Major revisions in this document include:

- Allowing the use of polypropylene closures as an alternative to phenolic closures;
- Specifying the use of CLP Inorganic Low Concentration Contract Required Detection Limits (CRDL);
- Specifying the use of CLP Organic Low Concentration Contract Required Quantitation Limits (CRQL);
- Including procedures for the cleaning of containers for fluoride and nitrate/nitrite analyses;
- Including procedures for the quality control analysis of fluoride and nitrate/nitrite; and
- Specifying the use of CLP Inorganic and Organic Low Concentration analytical methods for quality control analyses.

OERR and the EPA Regions decided to use the most stringent CLP requirements available to set the specifications for obtaining contaminant-free sample containers. As a result, the CLP Inorganic and Organic Low Concentration Statement of Work (SOW) requirements were selected as the basis for these specifications. Major factors in this decision included the desire to have a single set of bottle cleaning specifications that met or exceeded all analytical requirements and the related need to avoid potential misuse of cleaned bottles (e.g., using a container cleaned by a multi-concentration

procedure for a low concentration sample). OERR will reevaluate this decision if the low concentration requirements are deemed to be too stringent.

Specifications and guidance for preparing contaminant-free sample containers are provided in the sections that follow and are intended to describe one approach for obtaining cleaned, contaminant-free sample containers for use by groups performing sample collection activities under Superfund and other hazardous waste programs. Although other cleaning procedures may be used, sample containers must meet the criteria specified in Section II. In certain instances, the user of the sample containers may require exact adherence to the cleaning procedures and/or quality control analysis described in this document. In other instances, the user may require additional or different cleaning procedures and/or quality control analysis of the sample containers. The specific needs of the bottle user will determine the requirements for the cleaning and quality control analysis of the sample containers.

Most environmental sampling and analytical applications offer numerous opportunities for sample contamination. For this reason, contamination is a common source of error in environmental measurements. The sample container itself represents one such source of sample contamination. Hence, it is vital that sample containers used within the Superfund program meet strict specifications established to minimize contamination which could affect subsequent analytical determinations. Superfund sampling and analysis activities require all component materials (caps, liners, septa, packaging materials, etc.) provided by the bottle preparer to meet or exceed the criteria limits of the bottle specifications listed within Section II.

Section III provides guidance on cleaning procedures for preparing contaminant-free sample containers that meet the specifications contained in Section II. The procedures provided in this section are intended to provide sample containers that meet all current CLP Low Concentration Inorganic and Organic detection/quantitation levels.

In selecting cleaning procedures for sample containers, it is important to consider all of the parameters of interest. Although a given cleaning procedure may be effective for one parameter or type of analysis, it may be ineffective for another. When multiple determinations are performed on a single sample or on a subsample from a single container, a cleaning procedure may actually be a source of contamination for some analytes while minimizing contamination in others. It should be the responsibility of the bottle supplier to verify that the cleaning procedures actually used satisfy the quality control requirements set forth in Section IV.

Two aspects of quality assurance (i.e., quality control and quality assessment) must be applied to sample containers as well as to the analytical measurements. Quality control includes the application of good laboratory practices and standard operating procedures especially designed for the cleaning of sample containers. The cleaning operation should be based on protocols especially designed for specific contaminant problems. Strict adherence to these cleaning protocols is imperative.

Quality assessment of the cleaning process depends largely on monitoring for adherence to the respective protocols. Because of their critical role in the quality assessment of the cleaning operation, protocols must be carefully designed and followed.

Guidance is provided in Section IV on design and implementation of quality assurance and quality control protocols.

SECTION II

SAMPLE CONTAINER AND COMPONENT MATERIAL SPECIFICATIONS

This Section identifies sample containers commonly used in the Superfund program and provides specifications for contaminant-free sample containers for each bottle type.

A. CONTAINER MATERIAL

A variety of factors affect the choice of containers and cap material. These include resistance to breakage, size, weight, interferences with analytes of interest, cost, and availability.

Container types A through L (Figure 1, pages 6-7) are designated as the type of sample containers that have been used successfully in the past. Kimax or Pyrex brand borosilicate glass is inert to most materials and is recommended where glass containers are used (i.e., pesticides and other organics). Conventional polyethylene is recommended when plastic is acceptable because of its lower cost and lower adsorption of metal ions. The specific sampling situation will determine the use of plastic or glass.

While the sample containers shown in Figure 1 are utilized primarily for Superfund sampling activities, they also may be used for sampling activities under other programs, such as the Resource Conservation and Recovery Act (RCRA).

B. MAXIMUM CONTAMINANT LEVEL SPECIFICATIONS FOR SAMPLE CONTAINERS

The CLP, through a series of technical caucuses, has established inorganic Contract Required Detection Limits (CRDL) and organic Contract Required Quantitation Limits (CRQL) which represent the minimum detectable quantities needed to support the hazardous substance identification and monitoring requirements necessary for remedial and other actions at hazardous waste sites.

The philosophy used for determining the maximum permissible amount of contamination in a sample container was to consider the number of aliquots of sample that are available in the container and assume that the contamination present would be uniformly distributed in all of the aliquots. This assumption, and the assumption that there should be no more than one-half the CRDL or CRQL contributed by the container, resulted in the establishment of contamination limits by container type.

For inorganic sample containers, the CRDLs listed in Table 1, page 8, are the specifications for maximum trace metal contamination. Concentration at or above these limits on any parameter should preclude these containers from use in collecting inorganic samples.

The CRQL specifications for organic sample containers are listed in Table 2, pages 9-13. When the CRQL in Table 2 is multiplied by the appropriate factor listed below, the resulting value then represents the maximum concentration allowed for particular sample containers based on organic CLP sample sizes for routine analyses.

Container type	Multiple of CRQL
A	1.0
B	0.5
D	10.0
E	8.0
F	4.0
G	2.0
H	0.5
J	0.5
K	2.0

C. GROSS CONTAMINATION

Gross contamination is defined as greater than two hundred times the acceptable concentration values in Tables 1 or 2, unless the cleaning procedure is successful in reducing the amount of contamination to within specifications. If this is not achieved, the grossly contaminated materials should be discarded and replaced to prevent cross contamination with other batches of containers.

The bottle preparer should inspect all materials to ensure conformance with the required specifications.

FIGURE 1

SAMPLE CONTAINER
SPECIFICATIONS

Container Type	Specifications
A	Container: 80-oz amber glass, ring handle bottle/jug, 38-mm neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 38-430 size; 0.015-mm teflon liner. Total Weight: 2.45 lbs.
B	Container: 40-mL glass vial, 24-mm neck finish. Closure: white polypropylene or black phenolic, open-top, screw cap, 15-cm opening, 24-400 size. Septum: 24-mm disc of 0.005-in teflon bonded to 0.120-in silicon for total thickness of 0.125-in. Total Weight: 0.72 oz.
C	Container: 1-L high-density polyethylene, cylinder-round bottle, 28-mm neck finish. Closure: white polyethylene cap, white ribbed, 28-410 size; F217 polyethylene liner. Total Weight: 1.89 oz.
D	Container: 120-mL wide mouth, glass vial, 48-mm neck finish. Closure: white polypropylene cap, 48-400 size; 0.015-mm teflon liner. Total Weight: 4.41 oz.
E	Container: 16-oz tall, wide mouth, straight-sided, flint glass jar, 63-mm neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 63-400 size; 0.015-mm teflon liner. Total Weight: 9.95 oz.
F	Container: 8-oz short, wide mouth, straight-sided, flint glass jar, 70-mm neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 58-400 size; 0.030-mm teflon liner. Total Weight: 7.55 oz.

FIGURE 1

SAMPLE CONTAINER SPECIFICATIONS (Continued)

Container Type	Specifications
G	Container: 4-oz tall, wide mouth, straight-sided, flint glass jar, 48-mm neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.015-mm teflon liner. Total Weight: 4.70 oz.
H	Container: 1-L amber, Boston round, glass bottle, 33-mm pour-out neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 33-430 size; 0.015-mm teflon liner. Total Weight: 1.11 lbs.
J	Container: 32-oz tall, wide mouth, straight-sided, flint glass jar, 89-mm neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 89-400 size; 0.015-mm teflon liner. Total Weight: 1.06 lbs.
K	Container: 4-L amber glass, ring handle bottle/jug, 38-mm neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 38-430 size; 0.015-mm teflon liner. Total Weight: 2.88 lbs.
L	Container: 500-mL high-density polyethylene, cylinder-round bottle, 28-mm neck finish. Closure: white polypropylene cap, white ribbed, 28-410 size; F217 polyethylene liner. Total Weight: 1.20 oz.

TABLE 1

INORGANIC ANALYTE SPECIFICATIONS

	Analyte	Contract Required Detection Limits[1] (µg/L)
1.	Aluminum	100
2.	Antimony	5
3.	Arsenic	2
4.	Barium	20
5.	Beryllium	1
6.	Cadmium	1
7.	Calcium	500
8.	Chromium	10
9.	Cobalt	10
10.	Copper	10
11.	Iron	500
12.	Lead	2
13.	Magnesium	500
14.	Manganese	10
15.	Mercury	0.2
16.	Nickel	20
17.	Potassium	750
18.	Selenium	3
19.	Silver	10
20.	Sodium	500
21.	Thallium	10
22.	Vanadium	10
23.	Zinc	20
24.	Cyanide	10
25.	Fluoride	200
26.	Nitrate/Nitrite	100

1 CRDLs are based on the CLP Inorganic Low Concentration SOW (1990)

TABLE 2

ORGANIC COMPOUND
SPECIFICATIONS

	Volatiles	CAS Number	Contract Required Quantitation Limits[1] (µg/L)
1.	Chloromethane	74-87-3	1
2.	Bromomethane	74-83-9	1
3.	Vinyl Chloride	75-01-4	1
4.	Chloroethane	75-00-3	1
5.	Methylene Chloride	75-09-2	2
6.	Acetone	67-64-1	5
7.	Carbon Disulfide	75-15-0	1
8.	1,1-Dichloroethene	75-35-4	1
9.	1,1-Dichloroethane	75-34-3	1
10.	cis-1,2-Dichloroethene	156-59-4	1
11.	trans-1,2-Dichloroethene	156-60-5	1
12.	Chloroform	67-66-3	1
13.	1,2-Dichloroethane	107-06-2	1
14.	2-Butanone	78-93-3	5
15.	Bromochloromethane	74-97-5	1
16.	1,1,1-Trichloroethane	71-55-6	1
17.	Carbon Tetrachloride	56-23-5	1
18.	Bromodichloromethane	75-27-4	1
19.	1,2-Dichloropropane	78-87-5	1
20.	cis-1,3-Dichloropropene	10061-01-5	1
21.	Trichloroethene	79-01-6	1
22.	Dibromochloromethane	124-48-1	1
23.	1,1,2-Trichloroethane	79-00-5	1
24.	Benzene	71-43-2	1
25.	trans-1,3-Dichloropropene	10061-02-6	1
26.	Bromoform	75-25-2	1
27.	4-Methyl-2-pentanone	108-10-1	5
28.	2-Hexanone	591-78-6	5
29.	Tetrachloroethene	127-18-4	1
30.	1,1,2,2-Tetrachloroethane	79-34-5	1

1 CRQLs are based on the CLP Organic Low Concentration SOW (1990)

TABLE 2

ORGANIC COMPOUND
SPECIFICATIONS
(Continued)

Volatiles	CAS Number	Contract Required Quantitation Limits[1] (µg/L)
31. 1,2-Dibromoethane	106-93-4	1
32. Toluene	108-88-3	1
33. Chlorobenzene	108-90-7	1
34. Ethylbenzene	100-41-4	1
35. Styrene	100-42-5	1
36. Xylenes (total)	1330-20-7	1
37. 1,3-Dichlorobenzene	541-73-1	1
38. 1,4-Dichlorobenzene	106-46-7	1
39. 1,2-Dichlorobenzene	95-50-1	1
40. 1,2-Dibromo-3-chloropropane	96-12-8	1

1 CRQLs are based on the CLP Organic Low Concentration SOW (1990)

TABLE 2

ORGANIC COMPOUND
SPECIFICATIONS
(Continued)

	Semivolatiles	CAS Number	Contract Required Quantitation Limits[1] (µg/L)
1.	Phenol	108-95-2	5
2.	bis-(2-Chlorethyl)ether	111-44-4	5
3.	2-Chlorophenol	95-57-8	5
4.	2-Methylphenol	95-48-7	5
5.	2,2'-oxybis-(1-Chloropropane)	108-60-1	5
6.	4-Methylphenol	106-44-5	5
7.	N-Nitroso-di-n-dipropylamine	621-64-7	5
8.	Hexachloroethane	67-72-1	5
9.	Nitrobenzene	98-95-3	5
10.	Isophorone	78-59-1	5
11.	2-Nitrophenol	88-75-5	5
12.	2,4-Dimethylphenol	105-67-9	5
13.	bis-(2-Chloroethoxy)methane	111-91-1	5
14.	2,4-Dichlorophenol	120-83-2	5
15.	1,2,4-Trichlorobenzene	120-82-1	5
16.	Naphthalene	91-20-3	5
17.	4-Chloroaniline	106-47-8	5
18	Hexachlorobutadiene	87-68-3	5
19.	4-Chloro-3-methylphenol	59-50-7	5
20.	2-Methylnaphthalene	91-57-6	5
21.	Hexachlorocyclopentadiene	77-47-4	5
22.	2,4,6-Trichlorophenol	88-06-2	5
23.	2,4,5-Trichlorophenol	95-95-4	20
24.	2-Chloronaphthalene	91-58-7	5
25.	2-Nitroaniline	88-74-4	20
26.	Dimethylphthalate	131-11-3	5
27.	Acenaphthylene	208-96-8	5
28.	2,6-Dinitrotoluene	606-20-2	5
29.	3-Nitroaniline	99-09-2	20
30.	Acenaphthene	83-32-9	5

1 CRQLs are based on the CLP Organic Low Concentration SOW (1990)

TABLE 2

ORGANIC COMPOUND
SPECIFICATIONS
(Continued)

	Semivolatiles	CAS Number	Contract Required Quantitation Limits[1] (µg/L)
31.	2,4-Dinitrophenol	51-28-5	20
32.	4-Nitrophenol	100-02-7	20
33.	Dibenzofuran	132-64-9	5
34.	2,4-Dinitrotoluene	121-14-2	5
35.	Diethylphthalate	84-66-2	5
36.	4-Chlorophenyl-phenylether	7005-72-3	5
37.	Fluorene	86-73-7	5
38.	4-Nitroaniline	100-01-6	20
39.	4,6-Dinitro-2-methylphenol	534-52-1	20
40.	N-Nitrosodiphenylamine	86-30-6	5
41.	4-Bromophenyl-phenylether	101-55-3	5
42.	Hexachlorobenzene	118-74-1	5
43.	Pentachlorophenol	87-86-5	20
44.	Phenanthrene	85-01-8	5
45.	Anthracene	120-12-7	5
46.	Di-n-butylphthalate	84-74-2	5
47.	Fluoranthene	206-44-0	5
48	Pyrene	129-00-0	5
49.	Butylbenzylphthalate	85-68-7	5
50.	3,3'-Dichlorobenzidine	91-94-1	5
51.	Benz[a]anthracene	56-55-3	5
52.	Chyrsene	218-01-9	5
53.	bis-(2-Ethylhexyl)phthalate	117-81-7	5
54.	Di-n-octylphthalate	117-84-0	5
55.	Benzo[b]fluoranthene	205-99-2	5
56.	Benzo[k]fluoranthene	207-08-9	5
57.	Benzo[a]pyrene	50-32-8	5
58.	Indeno(1,2,3-cd)pyrene	193-39-5	5
59.	Dibenz[a,h]anthracene	53-70-3	5
60.	Benzo[g,h,i]perylene	191-24-2	5

1 CRQLs are based on the CLP Organic Low Concentration SOW (1990)

TABLE 2

ORGANIC COMPOUND
SPECIFICATIONS
(Continued)

	Pesticides/PCBs	CAS Number	Contract Required Quantitation Limits[1] (µg/L)
1.	alpha-BHC	319-84-6	0.01
2.	beta-BHC	319-85-7	0.01
3.	delta-BHC	319-86-8	0.01
4.	gamma-BHC (Lindane)	58-89-9	0.01
5.	Heptachlor	76-44-8	0.01
6.	Aldrin	309-00-2	0.01
7.	Heptachlor epoxide	1024-57-3	0.01
8.	Endosulfan I	959-98-8	0.01
9.	Dieldrin	60-57-1	0.02
10.	4,4'-DDE	72-55-9	0.02
11.	Endrin	72-20-8	0.02
12.	Endosulfan II	33213-65-9	0.02
13.	4,4'-DDD	72-54-8	0.02
14.	Endosulfan sulfate	1031-07-8	0.02
15.	4,4'-DDT	50-29-3	0.02
16.	Methoxychlor	72-43-5	0.10
17.	Endrin ketone	53494-70-5	0.02
18	Endrin aldehyde	7421-36-3	0.02
19.	alpha-Chlordane	5103-71-9	0.01
20.	gamma-Chlordane	5103-74-2	0.01
21.	Toxaphene	8001-35-2	1.0
22.	Aroclor-1016	12674-11-2	0.20
23.	Aroclor-1221	11104-28-2	0.20
24.	Aroclor-1232	11141-16-5	0.40
25.	Aroclor-1242	53469-21-9	0.20
26.	Aroclor-1248	12672-29-6	0.20
27.	Aroclor-1254	11097-69-1	0.20
28.	Aroclor-1260	11096-82-5	0.20

1 CRQLs are based on the CLP Organic Low Concentration SOW (1990)

SECTION III

SAMPLE CONTAINER PREPARATION AND CLEANING PROCEDURES

This Section is provided as guidance for the preparation of sample containers that meet the contaminant-free specifications contained in Section II. There are various procedures for cleaning sample containers depending upon the analyses to be performed on the sample. The following cleaning procedures are modeled after those specified for the Superfund Sample Container Repository program.

A. Cleaning Procedure for Container Types: A, E, F, G, H, J, and K

1. Sample Type: Semivolatile Organics, Pesticides, Metals, Cyanide, and Fluoride in Soils and Water.

   a. Wash glass bottles, teflon liners, and caps with hot tap water using laboratory grade nonphosphate detergent.

   b. Rinse three times with copious amounts of tap water to remove detergent.

   c. Rinse with 1:1 nitric acid (reagent grade HNO_3, diluted with ASTM Type I deionized water).

   d. Rinse three times with ASTM Type I organic free water.

   e. Oven dry bottles, liners and caps at 105-125°C for one hour.

   f. Allow bottles, liners, and caps to cool to room temperature in an enclosed contaminant-free environment.

   g. Rinse bottles with pesticide grade hexane (for pesticide determinations) or pesticide grade methylene chloride (for semivolatile organics determinations) using 20 mL for 1/2 gallon containers; 10 mL for 32-oz and 16-oz containers; and 5 mL for 8-oz and 4-oz containers.

   h. Oven dry bottles, liners, and caps at 105-125°C for one hour.

   i. Allow bottles, liners, and caps to cool to room temperature in an enclosed contaminant-free environment.

   j. Place liners in lids and cap containers.

   k. Label each container with lot number and pack in case.

   l. Label exterior of each case with lot number.

   m. Store in contaminant-free area.

2. Sample Type: Nitrate/Nitrite in Soils and Water.

   a. Substitute reagent grade sulfuric acid (H_2SO_4) for nitric acid in step A.1.c.

   b. Follow all other steps in the cleaning procedure described in part A.1 above.

B. Cleaning Procedure for Container Types: B, D

1. Sample Type: Purgeable (Volatile) Organics in Soils and Water.

   a. Wash glass vials, teflon-backed septa, teflon liners, and caps in hot water using laboratory grade nonphosphate detergent.

   b. Rinse three times with copious amounts of tap water to remove detergent.

   c. Rinse three times with ASTM Type I organic-free water.

   d. Oven dry vials, caps, septa, and liners at 105-125°C for one hour.

   e. Allow vials, caps, septa and liners to cool to room temperature in an enclosed contaminant-free environment.

   f. Seal 40-mL vials with septa (teflon side down) and cap.

   g. Place liners in lids and cap 120-mL vials.

   h. Label each vial with lot number and pack in case.

   i. Label exterior of each case with lot number.

   j. Store in contaminant-free area.

C. Cleaning Procedure for Container Types: C, L

1. Sample Type: Metals, Cyanide, and Fluoride in Soils and Water.

   a. Wash polyethylene bottles and caps in hot tap water using laboratory-grade nonphosphate detergent.

   b. Rinse three times with copious amounts of tap water to remove detergent.

   c. Rinse with 1:1 nitric acid (reagent grade HNO_3, diluted with ASTM Type I deionized water).

   d. Rinse three times with ASTM Type I deionized water.

e. Invert and air dry in contaminant-free environment.

f. Cap bottles.

g. Label each container with lot number and pack in case.

h. Label exterior of each case with lot number.

i. Store in contaminant-free area.

2. Sample Type: Nitrate/Nitrite in Soils and Water.

a. Substitute reagent grade sulfuric acid (H_2SO_4) for nitric acid in step C.1.c.

b. Follow all other steps in the cleaning procedure described in part C.1 above.

SECTION IV

SAMPLE CONTAINER QUALITY ASSURANCE AND QUALITY CONTROL REQUIREMENTS

A. Quality Assurance

The objectives of this Section are to: (1) present procedures for evaluating quality assurance (QA) information to ensure that specifications identified in Section II have been met; and (2) discuss techniques for the quality control (QC) analysis of sample containers to be used in conjunction with the cleaning procedures contained in Section III.

Major QA/QC activities should include the inspection of all incoming materials, QC analysis of cleaned lots of containers, and monitoring of the containers' storage area. Complete documentation of all QC inspection results (acknowledging acceptance or rejection) should be kept as part of the permanent bottle preparation files. QA/QC records (e.g., preparation/QC logs, analytical data, data tapes, storage log) should also be stored in a central location within the facility.

Documentation indicating that the container lot has passed all QA/QC requirements should be provided by the bottle vendor to the bottle purchaser with each container lot. Documentation should include a signed and dated cover statement affirming that all QA/QC criteria were met or exceeded and copies of raw data from applicable analyses of the QC containers. Minimum documentation that should be provided with each lot of containers follows:

- A statement that "Sample container lot _____ meets or exceeds all QA/QC criteria established in 'Specifications and Guidance for Obtaining Contaminant-Free Sample Containers'";
- Reconstructed Ion Chromatographs (RICs) from volatile and semivolatile organics determinations;
- GC chromatographs from pesticides determinations;
- ICP, hydride-ICP, or ICP-MS instrument readouts from metals determinations;
- AA raw data sheets and instrument readouts from metals determinations; and
- Cyanide, fluoride, and nitrate/nitrite raw data sheets and instrument readouts from these detrminations.

1. Incoming Materials Inspection:

A representative item from each case of containers should be checked for conformance with specifications provided in Section II. Any deviation should be considered unacceptable. A log of incoming shipments should be maintained to identify material type, purchase order number, and delivery date. The date

of incoming inspection and acceptance or rejection of the material should also be recorded on this log.

2. Quality Control Inspection of Cleaned Lots of Containers:

Following container cleaning and labeling, two containers should be selected from each container lot to be used for QC purposes. The two categories of QC containers should be as follows:

a. Analysis QC Containers:

One QC container per lot should be designated as the analysis QC container. The sample container preparer should analyze the analysis QC container(s) to check for contamination prior to releasing the associated container lot for shipment. The QC analyses procedures specified in the Quality Control Analysis part of this Section for determining the presence of semivolatile and volatile organics, pesticides, metals, cyanide, fluoride, and nitrate/nitrite should be utilized.

For each representative analysis QC container(s), the appropriate QC number should be assigned to the related lot of containers. For example, the QC number could be a six-digit number sequentially assigned to each lot that has undergone QC analysis. Under this numbering scheme, the first alphabetical character would be the container type letter from Figure 1, the next four digits would be assigned sequentially in numerical order starting with "0001" for the first lot to undergo QC analyses, and the last character would be either a "C" to indicate clearance or an "R" to indicate rejection.

If the representative analysis QC container(s) passes QC inspection, the related lot of containers should be released, and the appropriate QC number should be entered in the preparation/QC log to indicate clearance of the lot for shipment.

If the analysis QC container(s) are found to be contaminated per the specified QC analysis procedures, the appropriate QC rejection number should be assigned and entered in the preparation/QC log. Any container labels should be removed and the entire lot returned for reprocessing under a new lot number. Excessive QC rejection for a particular container type should be noted for future reference.

A laboratory standard and a blank should be run with each QC analysis. All QC analysis results should be kept in chronological order by QC report number in a central QC file. The QC numbers assigned should be documented in the preparation/QC log, indicating acceptance or rejection and date of analysis.

A container lot should not be released for shipment prior to QC analysis and clearance. Once the containers have passed QC inspection, the containers should be stored in a contaminant-free area until packaging and shipment.

b. Storage QC Containers:

One QC container per lot should be designated as the storage QC container. The storage QC container should be separated from the lot after cleaning and labeling and should be stored in a designated contaminant-free area for one year. The date the container is placed in the storage area should be recorded in the storage QC container log.

If contamination of the particular container lot comes into question at any time following shipment, the storage QC container should be removed from the storage area and analyzed using the QC analysis procedures for that container type (see Quality Control Analysis, this Section). Upon removal, containers should be logged out of the storage area.

The designated storage area should be monitored continuously for volatile contaminants. A precleaned, 40-mL vial that has passed a QC inspection should be filled with ASTM Type I organic-free water and be placed in the storage area. This vial should be changed at one-week intervals. The removed vial should be subjected to analysis for volatile organics as described in the Quality Control Analysis part of this Section. Any peaks indicate contamination. Identify contaminants, if present, and include the results in a report to all clients who purchased bottles in the past month from the affected lot(s).

B. Quality Control Analysis

The types of QC analyses correlate with the types of containers being analyzed and their future use in sample collection. The QC analyses are intended for the determination of:

- Semivolatile organics and pesticides;
- Volatile organics;
- Metals;
- Cyanide;
- Fluoride; and
- Nitrate/Nitrite.

QC analyses should be performed according to the container type and related sample type and utilize the specific method(s) described below.

1. Determination of Semivolatile Organics and Pesticides:

Container Types: A, E, F, G, H, J, and K

a. Sample Preparation:

- Add 60 mL of pesticide-grade methylene chloride to the container, and shake for two minutes.

- Transfer the solvent to a Kuderna-Danish (KD) apparatus equipped with a three-ball Snyder column. Concentrate to less than 10 mL on a steam bath. Split the solvent into two 5 mL fractions for semivolatile and pesticide determinations.

- Add 50 mL of pesticide-grade hexane (for pesticide determinations only) to the KD apparatus by slowly pouring down through the Snyder column. Concentrate to less than 10 mL to effect solvent replacement of hexane for methylene chloride.

- Concentrate the solvent to 1 mL using a micro-Snyder column.

- Prepare a solvent blank by adding 60 mL of the rinse solvent used in step "g" of the cleaning procedure for container types A, E, F, G, H, J, and K (Section III page 14) directly to a KD apparatus, and proceed as above.

b. Semivolatile Organics Sample Analysis:

- Instrument calibration should be performed as described in the current CLP Low Concentration Organics SOW with the following exceptions:

  (1) If problems are encountered meeting the %RSD criteria on the initial calibration for semivolatiles, the high concentration point should be deleted and a four-point calibration used.

  (2) The low concentration standard should be used for the continuing calibration standard for semivolatile analyses.

  (3) The percent difference window should be widened to ± 30 percent for all compounds.

- Inject 1 µL of solvent into a gas chromatograph/mass spectrometer (GC/MS).

- GC/MS operating conditions are listed in Figure 3 (page 28).

- Any peaks found in the container solvent that are not found in the solvent blank or with peak heights or areas not within + 50 percent of the blank peak height or area should be cause for rejection.

- Identify and quantitate any contaminant(s) that cause rejection of a container lot.

- A standard mixture of the 9 semivolatile organic compounds listed in Table 3 (page 27) with concentrations in the 5-20 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits.

- A solvent blank should be run with each analysis.

c. Pesticides Sample Analysis:

- Instrument calibration should be performed as described in the current CLP Low Concentration Organics SOW.

- Inject 1 μL of solvent into a gas chromatograph (GC) equipped with an electron capture detector (ECD).

- GC/ECD operating conditions are listed in Figure 4 (page 29).

- Any peaks found in the container solvent that are not found in the solvent blank or with peak heights or areas not within + 50 percent of the blank peak height or area should be cause for rejection.

- A standard mixture of the 7 pesticide compounds listed in Table 3 (page 27) with concentrations in the 0.01 to 1 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits.

- A solvent blank should be run with each analysis.

2. Determination of Volatile Organics:

Container Types: B and D

a. Sample Preparation:

- Fill the container with ASTM Type I organic-free water.

b. Sample Analysis:

- Instrument calibration should be performed as described in the current CLP Low Concentration Organics SOW with the following exceptions:

(1) If problems are encountered meeting the %RSD criteria on the initial calibration for volatiles, the high concentration point should be deleted and a four-point calibration used.

(2) The low concentration standard should be used for the continuing calibration standard for volatile analyses.

(3) The percent difference window should be widened to ± 30 percent.

- GC/MS operating conditions are listed in Figure 5 (page 30).

- Any peaks not found in the blank or with peak heights or areas not within + 50 percent of the blank peak height or area should be cause for rejection.

- Identify and quantitate any contaminant(s) that cause rejection of a container lot.

- A standard mixture of the 5 volatile organic compounds listed in Table 3 (page 27) with concentrations in the 1-5 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits.

- A blank should be run by analyzing an aliquot of the ASTM Type I water used above.

3. Determination of Metals:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Add 50 mL of ASTM Type I deionized water to the container, and acidify with 0.5 mL reagent-grade HNO_3. Cap and shake well.

- Treat the sample as a dissolved metals sample. Analyze the undigested water using the current CLP Low Concentration Inorganics SOW.

b. Sample Analysis:

- Instruments used for the analysis of the samples should meet the contract required detection limits in Table 1.

- The rinse solution should be analyzed before use on the bottles that are designated for analysis to ensure that a contaminated solution is not used for rinsing the bottles.

- Calibration verification standards should be analyzed at the beginning, end, and every ten samples within an analysis run (a continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements). The verification standards should be three to five times the values in Table 1. The percent recovery factor for the

verification standards should be between 90 to 110 percent or ± 10 percent of the actual value of the verification standard.

- Calibration blanks should be analyzed at the beginning, end, and every ten samples within an analysis run. A calibration blank is a solution made up exactly like the sample preparation solution. The calibration blank should be below the values in Table 1.

- A set of standards in the expected working range should be analyzed with each analytical run. The acid matrix of the standards, blank, and quality control samples should match that of the samples.

- Concentrations at or above the detection limit for each parameter (listed in Table 1) should be cause for rejection of the lot of containers. NOTE: Sodium detection limit for container types A, E, F, G, H, J, and K is 5000 µg/L unless the containers will be used for low concentration analyses, then the detection limit is 500 µg/L.

4. Determination of Cyanide:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Place 250 mL of ASTM Type I deionized water in the container. Add 1.25 mL of 6N NaOH (for container types F and G use 100 mL ASTM Type I deionized water and 0.5 mL 6N NaOH). Cap the container and shake vigorously for two minutes.

b. Sample Analysis:

- Analyze an aliquot as described in the current CLP Low Concentration Inorganics SOW.

- The detection limit should be 10 µg/L or lower.

- A blank should be run by analyzing an aliquot of the ASTM Type I water used above.

- A set of standards in the expected working range, a quality control sample, and blank should be prepared exactly as the sample.

- The detection of contaminants of 10 µg/L cyanide (or greater) should be cause for rejection of the lot of containers. NOTE: Contamination could be due to the container, the cap, or the NaOH.

5. Determination of Fluoride:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Place 250 mL of ASTM Type I deionized water in the container (for container types F and G use 100 mL ASTM Type I deionized water). Cap the container and shake vigorously for two minutes.

b. Sample Analysis:

- Analyze an aliquot as described in the current CLP Low Concentration Inorganics SOW.

- The detection limit should be 200 µg/L or lower.

- A blank should be run by analyzing an aliquot of the ASTM Type I water used above.

- A set of standards in the expected working range, a quality control sample, and blank should be prepared exactly as the sample.

- The detection of contaminants of 200 µg/L (or greater) fluoride should be cause for rejection of the lot of containers. NOTE: Contamination could be due to the container or the cap.

6. Determination of Nitrate/Nitrite:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Place 250 mL of ASTM Type I deionized water in the container (for container types F and G use 100 mL ASTM Type I deionized water). Cap the container and shake vigorously for two minutes.

b. Sample Analysis:

- Analyze an aliquot as described in the current CLP Low Concentration Inorganics SOW.

- The detection limit should be 100 µg/L or lower.

- A blank should be run by analyzing an aliquot of the ASTM Type I water used above.

- A set of standards in the expected working range, a quality control sample, and blank should be prepared exactly as the sample.

- The detection of contaminants of 100 μg/L (or greater) nitrate/nitrite should be cause for rejection of the lot of containers. NOTE: Contamination could be due to the container or the cap.

C. Preparation and Labeling

Sampling for environmental specimens requires that sample containers be transported to field sites prior to sample collection. As a result, considerable time may elapse between the receipt of sample containers and collection of the samples. Because of the large number of samples taken at any one site, accounting for all sample containers can become extremely difficult. The following guidance on the identification and tracking of sample containers is based on procedures that have been used successfully in the CLP bottle program.

1. Each shipment should be inspected to verify that the requested number of cleaned and prepared sample containers have been supplied and meet the requirements specified in Section II (Tables 1 and 2). If any shipment fails to meet the required specifications, it should be discarded and replaced with a supply of sample containers that meet the required criteria.

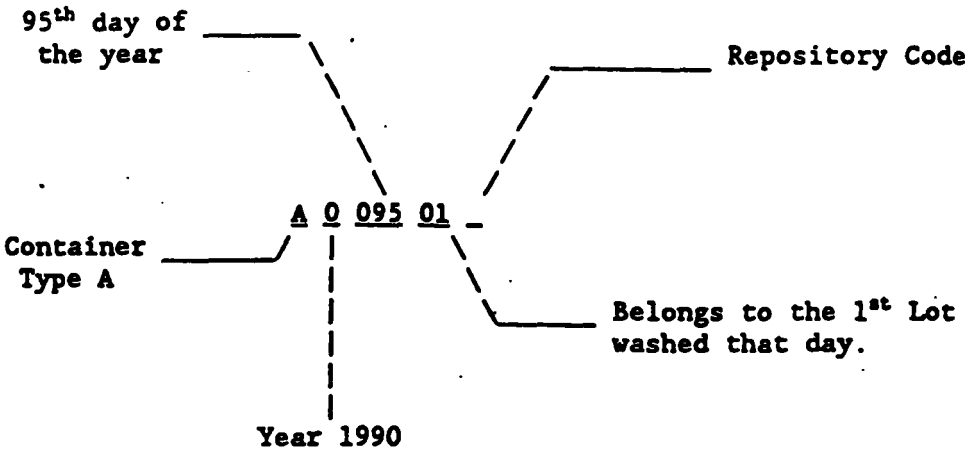
2. The sample containers should be removed and prepared in accordance with the methods designated below:

   a. Allocate the appropriate number of sample containers (Figure 1) to a designed container lot.

   b. Recommended lot size for each container should be based on the recommended number of items per case.

3. A permanent eight-digit lot number should be assigned to each lot of sample containers for identification and tracking purposes throughout the life of the containers. Figure 2 provides an example of a lot number sequence.

FIGURE 2



Lot Number Sequence

a. The first digit represents the container type in Section II (Figure 1).

b. The second digit represents the last digit of the calendar year.

c. The next three digits represents the day of the year on which the sample containers were washed.

d. The sixth and seventh digits represent the daily lot number.

e. The final digit represents the identification of the person who prepared the lot.

4. The lot number for each container should be entered, along with the date of washing, type of container, and number of containers per lot, into the preparation/QC log book.

5. Lot numbers printed with solvent resistant ink on a nonremovable label should remain with the corresponding containers throughout the cleaning procedure.

6. After sample container cleaning and drying, the label should be affixed to the containers in a permanent manner.

7. At least one face should be clearly marked, excluding the top and bottom faces, of each case of sample containers with the assigned lot numbers.

TABLE 3

STANDARD MIXTURES OF ORGANIC COMPOUNDS TO VERIFY SENSITIVITY

Volatiles	Semivolatiles	Pesticides
Methylene Chloride	Nitrobenzene	Gamma-BHC
Acetone	4-Chloroaniline	Heptachlor
2-Butanone	2,6-Dinitrotoluene	Aldrin
Trichloroethene	Diethylphthalate	Dieldrin
Toluene	4-Bromophenyl-phenylether	Endrin
	Hexachlorobenzene	4,4'-DDT
	Pentachlorophenol	Aroclor 1260
	Di-n-butylphthalate	
	bis(2-Ethylhexyl)phthalate	

FIGURE 3

GC/MS OPERATING CONDITIONS FOR SEMIVOLATILE ORGANICS QC ANALYSIS

OPERATOR: _____ DATE: _____

JOB NUMBER: _____ SAMPLE IDENTIFICATION: Container Lot number

SOLVENT: Methylene Chloride _____ ANALYTICAL METHOD: CLP Low Concentration SOW

Semivolatile Organics Fraction

COLUMN

Type Fused Silica Capillary or equiv.

Length 30 m

Diameter 0.25 mm or 0.32 mm ID

Liquid Phase (% wt)

J&W Scientific DB-5 or equivalent

Support N/A

Mesh N/A

CARRIER GAS Helium

Rotameter _____

Inlet Pressure, psig _____

Linear Velocity cm/sec 25-30

SCAVENGER GAS _____

SPLIT _____

FID GLASS

Hydrogen, mL/min N/A

Air, mL/min _____

CHART SPEED, cm/min _____

DETECTOR Mass Spectrometer

Range 35-500 a.m.u.

Attenuation _____

TEMPERATURE, °C

Detector _____

Injection Port 250-230°C

Column

Initial 40°/3 min

Program 10°/min

Final 290°C

INSTRUMENT _____

FIGURE 4

GC/ECD OPERATING CONDITIONS FOR PESTICIDES QC ANALYSIS

OPERATOR: _____ DATE: _____

JOB NUMBER: _____ SAMPLE IDENTIFICATION: Container Lot number

SOLVENT: Hexane _____ ANALYTICAL METHOD: CLP Low Concentration SOW

_____ Pesticide Fraction

COLUMNS (Two are required) FID GLASS

Type Fused Silica Capillary or equiv. Hydrogen, mL/min N/A

Length 30 m Air, mL/min N/A

Diameter 0.53 mm ID

Liquid Phase (% wt) CHART SPEED, cm/min 1 cm/min

J&W Scientific DB-1710 and DB-608 or equivalent

Support N/A DETECTOR Electron Capture

Mesh N/A Range _____

Attenuation 16

CARRIER GAS Helium or Hydrogen

Rotameter _____ TEMPERATURE, °C

Inlet Pressure, psig _____ Detector 350°C

Flow Rate, mL/min 5 Injection Port ≥ 200°C

Column

SCAVENGER GAS _____ Initial 150°/30sec

Program 5-6°/min

SPLIT _____ Final 275°C

INSTRUMENT _____

FIGURE 5

GC/MS OPERATING CONDITIONS FOR VOLATILES QC ANALYSIS

OPERATOR: _____ DATE: _____

JOB NUMBER: _____ SAMPLE IDENTIFICATION: Container Lot number

SOLVENT: Methanol ANALYTICAL METHOD: CLP Low Concentration SOW Volatile Organics Fraction

COLUMN

Type Fused Silica Capillary or equiv.

Length 30 m

Diameter 0.53 mm ID

Liquid Phase (% wt)

J&W Scientific DB-624, Suppelco VOCAL or equivalent

Support N/A

Mesh N/A

CARRIER GAS Helium or Nitrogen

Rotameter _____

Inlet Pressure, psig _____

Flow Rate, mL/min 15

SCAVENGER GAS _____

SPLIT _____

FID GLASS

Hydrogen, mL/min N/A

Air, mL/min N/A

CHART SPEED, cm/min _____

DETECTOR Mass Spectrometer

Range 35-300 a.m.u.

Attenuation _____

TEMPERATURE, °C

Detector _____

Injection Port _____

Column

Initial 10°/1-5 min

Program 6°/min

Final 160°C/all cmpds. elute

INSTRUMENT _____