Giesfeldt

ROY F. WESTON, INC.
THREE HAWTHORN PARKWAY, SUITE 400
VERNON HILLS, ILLINOIS 60061
® 708-918-4000

28 February 1992

241378280

OFFICE COPY

Ms. Betty Lavis
Remedial Project Manager (HSRW-6J)
U.S. Environmental Protection Agency
77 West Jackson Boulevard
Chicago, Illinois 60604-3590

Subject: Response to Comments on the Draft Interim Quality Assurance Project Plan
Moss-American Site, Milwaukee, Wisconsin

Dear Ms. Lavis:

Roy F. Weston, Inc. (WESTON), on behalf of the settling defendant Kerr-McGee Chemical Corporation (KMCC), is hereby responding to Agency comments dated 24 December 1991, on the Draft Interim Quality Assurance Project Plan (QAPP) and appended Field Sampling Plan (FSP) submitted to the U.S. Environmental Protection Agency (U.S. EPA) on 18 November 1991. A response to each comment from the U.S. EPA, the Wisconsin Department of Natural Resources (WDNR), and other related parties is addressed herein. Those portions of the above-referenced documents which have been revised as a result of these comments are also being transmitted herein.

KMCC and WESTON have recently undertaken a method performance study to further evaluate the use of Method 8270 SIMS in achieving the data quality objectives of this background determination study. This study is expected to be completed in mid- to late-March. Until this study is completed we are resubmitting this QAPP/FSP as a partially complete draft. Specifically, we have omitted any further reference to method precision and accuracy until this study is complete. In the interest of continuing to work with the Agency in finalizing the document, we have resubmitted this draft utilizing a redlining and strike-over format to assist in your review of document revisions.

Upon completion of the method performance study, we will submit a final QAPP/FSP which incorporates findings of the study and any additional comments received from the Agency on this transmittal.

In addition, we will forward under separate cover an illustration of our proposed background sampling locations for U.S. EPA and WDNR review and approval.

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Should you require further clarification of this transmittal, please contact the undersigned. We look forward to receiving approval of this QAPP/FSP in order that we may mobilize for associated field work this summer.

Very truly yours,

ROY F. WESTON, INC.

Gary J. Deigan
Senior Project Manager

Kurt S. Stimpson
Project Director

GJD:KSS/slr
Enclosures

cc: Mr. Mark Krippel, Project Manager
Kerr-McGee Chemical Corporation
798 W. Factory St.
West Chicago, IL 60186

Mr. George B. Rice
Kerr-McGee Chemical Corporation
P.O. Box 25861
Oklahoma City, Oklahoma 73125

Mr. Richard Meserve
Covington & Burling
1201 Pennsylvania Avenue N.W.
P.O. Box 7566
Washington, D.C. 20044

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Regional Counsel (1 copy)
Attn: Moss-American Site Coordinator (5CS)
U.S. Environmental Protection Agency
77 West Jackson Boulevard
Chicago, IL 60604

Assistant Attorney General (1 copy)
Environment and Natural Resources Division
U.S. Department of Justice
P.O. Box 7611
Ben Franklin Station
Washington, D.C. 20044
Ref. D.J. #90-11-2-590

Section Chief (3 copies)
Environmental Response and Repair Section
Bureau of Solid and Hazardous Waste Management
Department of Natural Resources
101 S. Webster Street
P.O. Box 7921
Madison, WI 53707-7921

Mr. Jim Schmidt (2 copies)
Department of Natural Resources
Southeast District Office
P.O. Box 12436
Milwaukee, WI 53212

Attachment 1

Agency Comments on 18 November 1991
Draft QAPP/FSP

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:

HSRW-6J

December 24, 1991

Mr. Kurt Stimpson
Weston, Inc.
Three Hawthorn Parkway, Suite 400
Vernon Hills, Illinois 60061

Dear Mr. Stimpson:

EPA has completed its review of the Draft Interim Quality Assurance Plan (QAPP) and appended Field Sampling Plan (FSP) submitted November 18, 1991 by Weston on behalf of Kerr-McGee. Attached to this letter are the comments that must be addressed in order to receive formal approval on the above documents. These include those submitted by the Quality Assurance Section (QAS) that I faxed to Weston on 12/20, some of which are marked with an asterisk(*). Please note my hand written instructions as to how to deal with these comments. In general, the comments submitted by QAS, while numerous, do not appear to be particularly difficult to address. Most are simply clarifications.

I have attached the comments received from the State of Wisconsin as received, separately from the above comments. Please address them or provide a brief (if appropriate) written rationale explaining why they were not addressed.

In order to facilitate review of the revised QAPP/FSP, please submit:

1) A list of the revisions made and the page/section number;

2) One set of revised pages only for QAS. QAS will comment on new areas if they receive the entire QAPP/FSP;

3) The designated number of copies of the revised QAPP/FSP for the remainder of the reviewers.

Please note that EPA has moved to another building. Our new address is on the letterhead. Feel free to call me if you have any questions or want to discuss the comments with me.

Sincerely,

Betty G. Lavis
Project Manager
312/886-4784

Attachment

cc: Mark Krippel
Gary Edelstein

EPA'S COMMENTS

Comments on the QAPP:

1. The introduction of the QAPP should be expanded (and contracted) to relate more of the site history and previous investigative actions relevant to the project and less of the topography, geology, and hydrogeology. The specific objective reads clearly, but the project objective, initial statement, and site history are weak. In the site history, no mention is made of the RI/FS or summaries of its findings. After reading the introduction the reviewer does not have a good understanding of the QAPP's contents and project objectives. The purpose of the QAPP and the areas the QAPP addresses could be outlined more clearly.

2. As PAH compounds are sensitive to UV light, precautions must be taken to preclude UV irradiation during the specified cleanup steps.

3. One reviewer noted that if the GC/MS is tuned with perfluorophenanthrene as opposed to decaflourotriphenylphosphine, the resulting data may not be comparable to data generated during the RI. Let's discuss this potential problem.

4. There is a discrepancy between the levels or units used for calibration standards used in MDL study (20, 50, 200, 500 and 2,000 ng/ml) and those reported under Preparation of Calibration Standards (20, 50, 200, 500 and 2,000 ug/ml). Given the operation conditions, the GC/MS could not detect standards prepared at ng/ml concentrations.

5. Section 3-1 - Edelstine should be spelled Edelstein. Same for Figure 3-1.

Comments on the FSP:

1. Section 2 - The MPB statistical approach assumes the validity of the following assumption: soil and vegetative cover are directly related to CPAH deposition and that the data will therefore be normally distributed. This assumption must be tested and the QAPP must define the test of the assumption.

2. Page 2-2 - The source document for clean up and removal standards must be referenced.

3. Section 3.3.1 and 3.3.2 - Frequency of field duplicates, 1 per 20 should be 1 per 10. Why are no field blank samples being submitted?

4. Section 3.4 - What will be the protocol if samples appear uniform before they are mixed?

5. Section 4 - What system will be used to conceal the identity of field duplicates in the lab? Is that covered in 3.3.2.?

6. Section 5.2 - Time of collection must be included in minimum requirements for sample labels.

7. Table 3-1 of the FSP, Step 3 instructs samplers to rinse equipment with isopropanol and "retain drippings." The table must state what is to be done with the retained drippings.

rec'd 12/23/91 BL

State of Wisconsin \ DEPARTMENT OF NATURAL RESOURCES

Carroll D. Besadny
Secretary

101 South Webster Street
Box 7921
Madison, Wisconsin 53707
SOLID WASTE TELEFAX 608-267-2768
TELEFAX 608-267-3579
TDD 608-267-6897

December 19, 1991

IN REPLY REFER TO: FID #4137828
Milwaukee Co.
ER/SFND

Ms. Betty Lavis, RPM
U. S. EPA Region V, HSRW-6J
77 W. Jackson Blvd.
Chicago, Ill 60604

SUBJECT: Comments on the Draft Quality Assurance Project Plan (QAPP) and Appended Field Sampling Plan (FSP), Moss-American (Kerr-McGee) Superfund Site, Milwaukee, WI

Dear Ms. Lavis:

We have completed our review of the above-referenced submittal, prepared by Roy F. Weston, Inc., for Kerr-McGee Chemical Corp. Based on our review, we have the following comments:

1. Our chemist's comments are attached.

2. Comments from our District and Central Office Water Resource Program staff are attached.

3. Section 2.2 and 2.2.2 of the FSP - Based on the discussion at the September 26, 1991 meeting, we understand there was agreement that the downstream tributary sediment sampling could occur, but there hasn't been a decision on how that data will be used. We also agreed that the FSP would outline options for how downstream tributary data could be used, and you (in consultation with us) would then decide on data use. The plan should be revised to state that there hasn't been a final decision on downstream tributary sampling data use and outline data use options.

requested from Weston 12/17

4. Section 2.2 - At the September 26 meeting, our Southeast District (SED) staff agreed to attempt to provide suggested background soil sampling locations. To assist in this effort, Weston should provide information on the level of detail needed for describing the locations (i.e., ¼-¼ section, longitude/latitude, circled on a map of some sort, etc.) and provide SED staff, you and this office copies of detailed topographic maps showing land uses in the area that are based on recent aerial photographs or copies of recent aerial photos. We understand that Weston has flown the site and is preparing detailed maps. Also, the Southeastern Wisconsin Regional Planning

Commission (SEWRPC), located in Waukesha, has recent aerial photos for the area available for purchase. The maps provided in the FSP are not detailed enough for this purpose.

We recommend that Weston provide their suggested preliminary Phase I soil and sediment sampling locations on the detailed maps or photos described above. Justification for the selected locations should also be provided. This would be of considerable help to us, and would begin the process for determining the exact locations through communications with the agencies and Weston. We suggest that process begin now, given that the Phase I field work is scheduled for this April.

Thank you for the opportunity to comment. Should you have any questions, do not hesitate to contact this office.

Sincerely,

Gary A. Edelstein, P.E., Waste Management Engineer
Emergency & Remedial Response Section
Bureau of Solid & Hazardous Waste Management

Noted: _____
Doug Ballotti, Acting Unit Leader
Superfund Program Unit

Enc.

cc: Jim Schmidt - SED (w/enc.)
Will Wawrzyn - SED (w/enc.)
Tom Janisch - WR/2 (w/enc.)
Kurt Stimpson - Weston (w/enc.)
Mark Krippel - Kerr-McGee (w/enc.)

State of Wisconsin

CORRESPONDENCE/MEMORANDUM

DATE: December 11, 1991 FILE REF:

TO: Gary Edelstein - SW/3

FROM: Charlene Khazae - SW/3 CK

SUBJECT: Draft Interim Quality Assurance Project Plan Moss-American Site, Milwaukee, Wisconsin

There are a number of items that need to be called to your attention regarding the above QAPP and FSP.

QAPP:

1. Will the QA Section of Weston specifically request reanalyzing samples if necessary, as long as analytical holding times are not exceeded? This should be a corrective action.

2. Do FTL and SH&S Coordinator both have authority to halt project? If so, who resolves conflict?

3. Section 4-1. Should be specified that the field duplicates are a check on field sampling techniques. It is not a check on analytical reproducibility. MSD's and other lab duplicates are checks for analytical reproducibility.

4. Table 4-1. What does "frequency" mean? One sampling event?
Might better read:

Field Samples	Field Duplicates	Lab QC(MS/MSD)	Total
45	3	3	48*

*does not include MS/MSD since no extra volume required...

5. Section 4.2 Precision-Will MS/MSD be repeated if >20% RPD?

6. MS recoveries of 50-150% seems like a wide range even for soils.

7. Page 4-5. "existing data (if any)" Was there any? Be specific.

8. Where are H&S monitoring numbers recorded? Logbook? Separate form? No reference to H&S Plan at all.

9. 6.1.3 Yellow copy of CofC- retained under secure conditions? (Logbook secure?)

10. Page 6-7 Custody Seals- prenumbered with #'s recorded on CofC or signed and dated? Please specify.

11. 6.2.1 1st item-Temperature? This will affect sample integrity.
3rd item-Are batch numbers and tracking numbers the same? Please clarify.
5th item-CofC copies,kept in secure area?

12. 6.2.5 Typing errors on bottom of page. didn't see any - @ yes - 4th line

13. Pages 7-2,7-3 Needs to be more specific regarding frequency of continuing calibrations.

14. 9.3.2 Field duplicates are <u>not</u> a lab QC check.

15. Page 9-4 Analytical balances are not monitored. Do they not go through a calibration check?

16. 11.2 Typing errors. 1st line

FSP:

1. again- Table 2-1 "Frequency" ?

2. Table 3-1 Step 1- Phosphate-free detergent should be used.

3. Table 5-1 a.Do glass jars have teflon-lined lids?
b.Would be nice to have a footnote : "All sample containers have been cleaned according to EPA's highest standards..." or simply reference Appendix C.

4. 6.3 "preservative (if any)" can be eliminated since it has been established that none will be added.

5. Why not include examples of Weston's Custody Seals and Sample Labels?

CORRESPONDENCE/MEMORANDUM

State of Wisconsin

DATE: December 17, 1991 FILE REF: 3200

TO: Mark Giesfeldt - SW/3

FROM: Duane Schuettpelz - WR/2

SUBJECT: Comments on Draft Interim Quality Assurance Project Plan and Draft Interim Field Sampling Plan for the Moss-American Site, Dated November 18, 1991.

Section 2.2 Soil and Sediment Sampling Program

1. Page 2-2 of 14. It needs to be reiterated that in relationship to the sediment quality criteria (SQC), they are based on organic carbon normalized values. Any comparisons done between measured background sediment levels of CPAHs found during Phases I or II with the SQC for CPAHs must be done on a standardized basis that involves consideration of organic carbon normalized sediment values.

   Based on the need for standardization, total organic carbon (TOC) should be analyzed for in all background sediment samples. Appropriate analytical procedures for TOC analysis needs to be included in Section 8 of the Interim QAPP.

   To correct background CPAH concentrations at a given TOC value in the sample to compare with the standardized SQC value for CPAHs (3,000 µg/Kg at 3.4% TOC), the following formula should be used:

$$y = \frac{(x)(3.4\%)}{\%\ \text{TOC in background sediment sample}}$$

   where:

   x = Total CPAH concentration measured in background sediments (µg/kg)

   y = Corrected CPAH concentration based on TOC content, for direct comparison with SQC value of 3,000 µg/kg at 3.37 % TOC.

2. Page 2-14 of 14. The referenced figures in paragraph 3 should be figures 2-3 and 2-4, rather than 2-4 and 2-5.

Section 2.2.1. Soil Sampling Design

1. Page 2-10 of 14. The description of Phase II soil background study states that the Phase I work will describe the background upland broadleaf forest habitat. This habitat is not proposed for study in Phase I.

Section 2.2.2. Sediment Sampling Design

1. Page 2-14 of 14. The deferring of decisions on how downstream tributary sampling will be used in MPB determinations is noted. The considerations and methodology for integrating sampling results for above-site and tributaries is contained in the Schuettpelz memo of August 7, 1991.

General

1. It is recommended that analysis for outliers be performed on all background sampling site data if some samples appear to have relatively high concentrations.

2. Phase I soil background data should be subject to ANOVA to see if the stratification used (between habitats) is resulting in discernable differences between habitats.

3. We have read and strongly concur with the Wawrzyn WR/SEH Draft Review and comments on the Moss-American site QAPP and Interim Field Sampling Plan.

v:\9204\wr9mossa.tpj

cc: Will Wawrzyn WR/SEH

State of Wisconsin

CORRESPONDENCE/MEMORANDUM

Date: December 16, 1991 File Ref: 3200

To: Gary Edelstein SW/3

From: Will Wawrzyn WR/SEH

Subject: Review and Comments on Moss-American Site QAPP

Attached are my comments on the Moss-American Site QAPP. Please call if you have any questions.

p. 10-2 Final Results should be accompanied by blank and recovery results.

Appendix A, Section 2

p.2 The soil and sediment sampling design should identify the proposed soil/sediment sample depth. Background soil samples should be composited and extend vertically to depths at which contamination was observed on the former facility site. Background sediment samples should be composited and extend vertically to parent or post glacial material.

p. 2-12 The phase 1 background sediment sample reach should extend north of Brown Deer Rd. to Donges Bay Rd. This is consistent with the intent and rational to sample similar adjacent land uses, specifically agriculture, and for "selecting locations to avoid sampling obvious upstream point and nonpoint source discharges such as tank farms, major highways, and landfills." In addition, background soil samples should not be collected from any habitats downstream of the Moss-American site and within the Little Menomonee River 100-year floodplain.

p. 2-12 What is the purpose of conducting ANOVA for significant differences in CPAH concentrations between stream segments? Sediment data from the RI/FS should be sufficient for identifying significant differences between stream segment sediments. Is this test to be applied between stream segment tributary concentrations versus Little Menomonee River segments located

immediately upstream? The purpose of this test, dependent versus independent variables, and hypotheses must be clearly stated.

A more rigorous multiple comparison test (eg. Newman-Keuls test) may be appropriate. Unlike a single factor ANOVA which test the hypotheses Ho: u1=u2=...uk, the rejection of Ho does not imply that all k means are different from one another, and one would not know where the differences are located.

Section 3

p. 3-1 What is the rational for selecting a soil sample depth of 12 inches for background determination? (see comments on Section 2, p. 2 above). B. Lavis - this depth was mutually agreed upon at the meeting 12/26/91
Sediment samples from catch basins may be skewed by large particles (>62 um) of broken asphalt, plastic, etc.

cc: Tom Janisch WR/2
Jim Schmidt SW/SEH

ATTACHMENT

I. Approval Signature Page and Table of Contents

Please delete Charles Elly, Director, of the Region 5 CRL from and add Gary Edelstine, the Wisconsin DNR RPM, to the Approval Signature Page.

II. Project Description (Sect. 2)

A. All target compounds should be explicitly listed and a rationale given for their choice. If only selected PAHs known to be carcinogens are of interest, this should be stated in the project description section of the QAPjP and a reason given for not including other PAHs, BTEX, phenols, etc. in the target compound list (2.6, pg. 2-13).

B. Please reference Table 8-1 and any other locations where MDLs for target compounds are listed (2.6, pg. 2-13).

C.* Please list the parameters for health and safety monitoring or reference where this information can be found. Also, section 7.1, pg. 7-1, can be referenced for a discussion of the instruments to be used in the field for health and safety purposes.

D. Please delete CRL from the statement on U.S. EPA offices that must approve analytical procedures. QAS alone provides this approval, except for any review done as part of an external lab audit (3.2.1, pg. 3-5).

E. Please specify that the lab's QA section will routinely review a specified per centage of data packages. Section 10.2.2, pg. 10-2, of this QAPjP provides only incomplete information on this practice, but this can also be referenced (3.4.3, pg. 3-8).

F. Table 4-1 needs the following changes to meet the Region 5 requirement for frequency of field duplicates (1 field duplicate per 10 or fewer investigative samples collected): in line 1, 5 field duplicates; in line 2, 3 field duplicates; in line 3, 2 field duplicates; and in line 4, 4 field duplicates.

G. The specification of +/- 20% for precision control limits for MS/MSD contradicts the +/- 50% specification given in the SOP. Please correct one of these specifications(4.2, pg. 4-3).

H. Please correct the wording of the sentence on reporting of blank spike results when MS/MSD data does not meet control limits so that it is clear that MS/MSD results still must be reported and the sample data qualified in these circumstances (4.2, pg. 4-3).

I. Please delete the specification that sample results will be corrected for recovery if MS/MSD results are outside their control limits. Sample data should only be qualified in these circumstances (4.2, pg. 4-4).

J. Please correct the specification for completeness for lab tests - it should be 95% or better, not 90% or better (4.3, pg. 4-4).

IV. Sample Custody (Sect. 6)

A. Please reference the FSP for sample cooler packing information, including the specification that custody seals will be used (6.1.3, pg. 6-3).

B. Procedures for recording transfer of samples within the lab should be described or reference made to where this information can be found (6.2.2, pg. 6-9).

C.* It should be specified that samples will be held for at least 60 days, not 30 days, after analysis before they are disposed of (6.2.4, pg. 6-10).

D. Please specify the Weston office's geographic location where evidence files will be maintained (6.3, pg. 6-12).

V. Calibration Procedures (Sect. 7)

A. Please specify that the frequency of calibration verification will be at least every 12 hours, not necessarily just every shift (7.2, pg. 7-3).

B. Also, please reference the SOP in Appendix B for procedures for preparing calibration standards.

VI. Analytical Procedures (Sect. 8)

A. Comment II.A, above, will affect which analytes are to be included in the MS/MSD. See, also, comment XII.A, below (9.3.2, pg. 9-3).

B. The surrogate control limits of 20%-150% given in section 9.3.3 contradicts the SOP in Appendix B, which specifies 50%-150%. Please correct one of these specifications (pg. 9-3).

C. For information on GC/MS tuning and calibration please reference the SOP in Appendix B and section 7 of this QAPjP.

VII. Data Reduction, Validation, and Reporting (Sect. 10)

A. Please reference the SOP in Appendix B for information on formulas to be used in determining the concentration of contaminants in samples (10.2.1, pg. 10-1).

B. Please reference the SOP in Appendix B also for surrogate and MS/MSD control limits to be used in data review (10.2.2, pg. 10-2).

C. Please specify who will receive the final data report. This information must be available to the the U.S. EPA on request (10.2.3, pg. 10-3).

VIII. Preventive Maintenance (Sect. 12)

A brief description of routine, short-term preventive maintenance procedures for GC/MS - septa replacement, injection port cleaning, etc. - should be included in Table 12-1.

IX. Data Assessment (Sect. 13)

Instrument sensitivity verification should make use of the low concentration calibration standard, not the continuing calibration standard (13.2.4, pg. 13-2).

X. QA Reports (Sect. 15)

It is insufficient to specify that a QA report will be prepared if QA problems are encountered. It should be specified that the final project report will include QA information regardless of whether or not QA problems were observed (15, pg. 15-1).

XI. Field Sampling Plan (Appendix A)

A. Figure 2-5 should be included in the reference for floodplains and habitat information and figure 2-3 should be deleted (2.2, pg. 2-4).

B. The listing of 53 total sediment samples should be 58, but see comment III.F, above (3.2, pg. 3-1).

C. As noted in comment III.F, above, the frequency of field duplicates should be one per ten or fewer investigative samples collected. Please correct the text in section 3.3.1, accordingly (pg. 3-2).

D. Please explain how water in sediment samples will be handled - e.g. will it be decanted before sample mixing? (3.4, pg. 3-3).

E.* It should be specified that the isopropanol used in sampling equipment decontamination will not be allowed to drain into the river (pg. 3-4).

F. Please delete the description of procedures for filling more than one sample bottle for a sample. For the tests and matrices planned for this project, one bottle per sample is sufficient (4.2, pg. 4-3).

G. It needs to be specified that the contractor will assume the responsibility of assuring that sample bottles to be used are contaminant-free. Also, the procedures to be used for verifying that the sample bottles are contaminant-free need to be described. See Addendum 1 to this Attachment for the language to be inserted into this QAPjP that specifies that the contractor is assuming the responsibility of assuring that sample bottles are contaminant-free (Table 5-1).

XII. PAH SOP (Appendix B)

A. As was noted in the QAS comments made in August, this SOP and associated MDL study should be consistent with method 8270 and/or the CLP SOW. Thus, expanded lists of target, calibration, internal standard, surrogate, and matrix spike compounds; some different quantitation ions; and SPCCs - all these need to be corrected or provided for, or the restricted lists and changes from method 8270 specifications, as given in the existing SOP, need to be justified.

In this regard, and as was also noted in the August comments, the 7/15/91 version of the SOP, which included Tables 1-4, not just Tables 1 and 2, much more closely reflected the method 8270 specifications and is preferred as a starting point, rather than the existing SOP.

B. As was noted previously, it needs to be specified that the source of the PES's will be Kerr-McGee (9.2, pg. 7).

C. A preliminary screening of samples for concentration level is highly recommended, as is specified in section 7.5.1 of method 8270 (11, pg. 10).

D. Please specify that the GC/MS will be tuned at least every 12 hour shift to the manufacturer's specifications using PFK (11.1, pg. 10).

E. Please specify that, subsequently, DFTPP tuning criteria, as specified in Table 3 of method 8270, will be met (11.1, pg. 10).

F. Please specify that the techniques described in section 8.10 of method 8270, which confirm identification and prevent mis-identification of PAHs, will be used (12.2, pg. 11).

G. Please specify that two different source materials will be used for calibration and spiking standards (4.1, pg. 17).

H. The GC/MS operating conditions need to be specified as completely as in section 7.3 of method 8270. Notable omissions from the existing SOP are carrier flow rate and injector and transfer line temperatures (Table 2).

Addendum 1: Bottle Requirements Language

Bottle Requirements

The contaminant-free sample containers (bottles) used for analyzing CLP TCL and TAL analytes for this sampling effort will be prepared according to the procedures specified in U.S. EPA's "**Specifications and Guidance for Obtaining Contaminant-Free Sample Containers, April 1990**" attached document. It will be assured that the bottles used for the sampling activity do not contain target organic and inorganic contaminants exceeding the level specified in the above mentioned document. For non-CLP TCL and TAL types of analytes, bottles either should 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 the analytes of interest to insure that the contaminant levels of those analytes do not exceed approximately 1/3 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 continuing basis. This data will be maintained in the project evidence file (for a Fund-lead site-in a central ARCS' file) and will be available, if requested, for EPA review.

In addition, the data for field blanks, rinsate blanks, and trip blanks, etc., will be monitored for contamination, and 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 re-testing the representative bottle from a suspect lot, re-sampling the suspected samples, validating the data taking into account that the contaminants could be introduced by the laboratory (i.e., common lab solvents, sample handling artifacts, etc.) or could be 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.

For the Fund-lead projects, the corrective actions will be conducted in a comprehensive manner in order to avoid the use of identified contaminated lot(s) for other projects, and to insure that if the bottle supplier(s) is deemed unresponsive or unable to provide cleaned bottles as specified, other EPA projects are not negatively impacted by the use of non-compliant bottles.

Attachment 2

Response to Agency Comments

RESPONSE TO COMMENTS FROM THE U.S. EPA (B. LAVIS)

Comments on the QAPP

1. The Project Description of the QAPP (Section 2) has been revised to address the concerns raised in this comment.

2. The revised SOP for Method 8270 SIMS (Appendix B) outlines precautions to take during cleanup steps.

3. Decafluorotriphenylphosphine (DFTPP) is not an appropriate tuning compound for MS methods using the selected ion monitor (SIM) mode. DFTPP spectra acquired using SIM descriptors do not resemble spectra during full scan acquisition used for Method 8270. Meeting confirmation ion/quantification ion (C/Q) criteria given in the SOP for continuing calibration satisfies the objective of DFTPP tuning i.e., confirming that standard spectra are nondistorted thus ensuring that target analytes are properly identified.

4. The correct units are ng/ml. The units on page 17 of 20 in the CPAH SOP (Appendix B) will be corrected.

5. The spelling of Edelstein has been corrected in Subsection 3.1.2 and on Figure 3-1 of the QAPP.

Comments on the FSP

1. Testing of the statistical assumptions associated with the maximum probable background (MPB) approach is not a requirement of the Consent Decree. The Consent Decree states that calculation of MPB concentrations shall be conducted in accordance with the methods provided in Appendix J of the Feasibility Study (FS).

2. In Section 2.2, page 2-2, the source document for cleanup and removal standards (the Consent Decree) has been appropriately referenced.

3. The frequency of field duplicates will be changed to 1 per 10. This change shall be reflected in amendments made to Subsection 3.3.1 and Table 2-1 of the FSP, and Section 4.1 and Table 4-1 of the QAPP. The U.S. EPA Region V CRL discourages the use of aqueous field blanks for soil and/or sediment samples. This statement can be found in the QAPP, Section 4.1, the last three sentences of the first paragraph.

4. The protocols outlined in Section 3.4 of the FSP will be followed for all samples regardless of appearance, in order to ensure consistency. A sentence stating this point has been added to the FSP, Section 3.4 on page 3-3.

5. Field duplicate samples will not be identified as such on the sample paperwork. Only the field personnel (and not the laboratory personnel) will know how to interpret the sample nomenclature system. A sentence has been added to Section 4.1 of the FSP that states that all field duplicates will be submitted "blind" to the laboratory.

6. The time of sample collection will be included as a minimum requirement for sample labels. A statement to this effect has been included in Section 5.2 of the FSP.

7. A statement has been added at the end of Table 3-1 indicating that the 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.

RESPONSE TO COMMENTS BY THE U.S. EPA QUALITY ASSURANCE SECTION (QAS)

Comments on the QAPP

I: Charles Elly will be deleted from the signature page; however, as stated in the Consent Decree, the WDNR does not have approval status, only the U.S. EPA. Therefore, Gary Edelstein will not be added to the approval signature page.

II-A: A rationale (consistent with the Consent Decree) has been provided in the new Subsection 2.5.3 for the eight target CPAH compounds.

II-B: Table 8-1 is referenced in Table 2-1 which is referred to in the original Section 2.6 of the QAPP. A sentence has been added to the last paragraph of the new Subsection 2.5.3, page 2-16, that refers directly to Table 8-1.

II-C: All health and safety issues associated with the field program for the Moss-American site will be addressed in the Site Health and Safety Plan. Appropriate references to this plan have been incorporated into the QAPP.

II-D: The U.S. EPA Region V CRL has been deleted from Subsection 3.2.7, page 3-5.

II-E: It has been specified that the WESTON laboratory's QA section will review 10 percent of the data packages. This statement will be placed in Subsection 3.4.3, page 3-8, and in the third paragraph of Subsection 10.2.2 on page 10-2 of the QAPP.

II-F: The frequency of field duplicate collection has been changed to 1 per 10 or fewer investigative samples collected. The number of field duplicate samples on each line of Table 4-1 of the QAPP will be changed to meet this requirement.

II-G: All references to quality assurance objectives for precision and accuracy in Section 4.2 of the QAPP have been omitted until completion of a method performance study. This method performance study has been designed to evaluate method performance with an off-site, Milwaukee area background soil and sediment matrix. Results of this method performance study will be utilized to specify appropriate ranges of precision and accuracy.

II-H: See comment II-G above.

II-I: See comment II-G above.

II-J: In Section 4.3, page 4-5, the completeness factor has been changed to 95 percent or better as requested.

IV-A: A statement has been added to Subsection 6.1.3, page 6-3, of the QAPP specifying the use of custody seals and referring to the FSP as requested.

IV-B: The last two paragraphs of Subsection 6.2.2, page 6-9 of the QAPP, discuss procedures for transferring samples within the laboratory including record keeping requirements.

IV-C: Subsection 6.2.4, page 6-10, of the QAPP, has been amended as requested.

IV-D: It has been stated in Section 6.3, that the WESTON, Vernon Hills, Illinois office will house the project evidence file.

V-A: Section 7.2, page 7-3, has been amended as requested.

V-B: A sentence referring procedures for preparing calibration standards to the SOP in Appendix B has been added to Section 7.2, page 7-3, as requested.

VI-A: This comment is not applicable. See our response to comment II.A.

VI-B: See our response to comment II-G.

VI-C: A sentence has been added to the end of Section 8.1, page 8-3 of the QAPP, that states the requested information.

VII-A: This comment has been addressed as requested in a sentence added to the last paragraph of Subsection 10.2.1, page 10-1.

VII-B: This comment has been addressed as requested in a sentence added to the end of the first paragraph of Subsection 10.2.2 on page 10-2.

VII-C: A sentence will be added to the last paragraph of Subsection 10.2.3 on page 10-3 of the QAPP, that states that 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.

VIII: Table 12-1 of the QAPP has been revised to incorporate the requested information.

IX: Subsection 13.2.4 has been revised to reflect this requested change.

X: Section 15 has been amended as requested.

Comments on the FSP

XI-A: Due to a typographical error in the last paragraph of Section 2.2 on page 2-4 of the FSP, reference to Figure 2-3 was inadvertently omitted. The reference in this paragraph should be Figures 2-3 and 2-4 instead of Figures 2-4 and 2-5. This error has been amended and should clear up the misunderstanding that prompted this comment.

XI-B: The listing of 53 total sediment samples in Section 3.2, page 3-1 of the FSP has been changed to 61 total sediment samples.

XI-C: The frequency of field duplicate sample collection has been changed to 1 per 10 or fewer investigative samples collected and the text in Subsection 3.3.1 of the FSP amended accordingly. Table 2-1 of the FSP also reflects this change.

XI-D: Any water that is collected with a sediment sample will not be decanted prior to undergoing sample homogenization. This statement will be added to Section 3.4, page 3-3, of the FSP.

XI-E: Step 3 in Table 3-1, page 3-4, of the FSP states that the isopropanol drippings will be retained; therefore, they will not be allowed to drain into the river. A sentence has been added that states that the drippings will be containerized in a drum or equivalent vessel, staged on site with RI wastes, and properly disposed following the completion of all predesign field activities.

XI-F: The information identified in this comment has been deleted from Section 4.2, page 4-3 of the FSP.

XI-G: Language discussing protocols associated with assuring that sample containers to be used are contaminant-free has been included in Section 8 of the FSP.

Comments on Appendix B - SOP for CPAHs

XII-A: The method is designed for low level detection and quantification of a selected list of CPAH's which was determined in the Consent Decree. The SOP as written contains all the elements of method 8270 except for the DFTPP tuning which is addressed below.

Note that system performance criteria are included in Section 8.2 of the SOP. Also the Table 1 in the current SOP contains all the information given in the original Tables 1-4.

XII-B: We will use a separate source of standards for calibration and spiking.

XII-C: The use of a screening method is discretionary; however, we have added a QC step (Section 9.5 of the SOP) to assure that "carryover" from dirty samples does not cause false positives.

XII-D: See SOP Section 11.1 which states that mass calibration will be performed each 12 hour period.

XII-E: DFTPP is not an appropriate tuning compound for MS methods using the selected ion monitor (SIM) mode. DFTPP spectra acquired using SIM descriptors do not resemble spectra during full scan acquisition used for method 8270. Meeting confirmation ion/quantification ion (C/Q) criteria given in the SOP for continuing calibration satisfies the objectives of DFTPP tuning i.e., confirming that standard spectra are nondistorted thus assuring that target analytes are properly identified.

XII-F: Field duplicates are described in the QAPP; confirmation techniques given in 8.10 are not relevant to the SIM method. The laboratory will analyze a standard reference material.

XII-G: Two sources of standards will be used - one for calibration and one for spiking..

XII-H: See revised Table 2. Also please note our response to QAS comment II.G.

RESPONSE TO COMMENTS BY WDNR (DUANE SCHUETTPELZ)

Comments on the FSP Section 2.2 Soil and Sediment Sampling Program

1. WESTON and KMCC acknowledge that background sediment CPAH levels may be normalized to a standard organic carbon content. As defined in the ROD, compliance with Wisconsin Sediment Quality Criteria (SQC) is to be considered, however is not an ARAR. Our present understanding is that the requirements for the calculation of background sediment CPAH concentrations are contained within Appendix J of the FS.

2. The referenced figures in paragraph 3 on page 2-4 have been corrected.

Comments on the FSP Subsection 2.2.1 Soil Sampling Design

1. In Subsection 2.2.1, page 2-10 of 14, the phrase "upland broadleaf forest" will be changed to "non-wetland, non-forested upland."

Comments on the FSP Subsection 2.2.2 Sediment Sampling Design

1. As agreed upon in the 26 September 1991 meeting of U.S. EPA, WDNR, WESTON, and KMCC, a decision by U.S. EPA and WDNR as to the use of downstream tributary sediment data has been deferred at this time. However, we wish to point out that the Statement of Work's (SOW) description of Predesign Task 2 specifically states: "... the Settling Defendant will develop a sampling and analysis plan to determine the background concentrations of CPAHs in sediments of the Little Menomonee River, including background concentrations upstream of the former wood preserving plant and in relevant downstream tributaries." Deferring or limiting the use of downstream tributary sediment data is inconsistent with the SOW requirements.

General

1. As stated in the Consent Decree, the calculation of MPB background concentrations shall be conducted in accordance with the methods provided in Appendix J of the FS.

2. Phase I soil background data may be subjected to appropriate statistical tests (ANOVA, Newman-Keuls, Tukey's) to determine the usefulness of stratification if the background values are above the risk-based cleanup standards. This statement has been added to Subsection 2.2.1, page 2-8 of the FSP.

3. Response not appropriate.

RESPONSE TO COMMENTS BY WDNR (WILL WAWRZYN)

Comments on the QAPP

p. 10-2 The final analytical data report will include blank and recovery results.

Comments on the FSP (Appendix A) Section 2

p. 2 As agreed upon in the 26 September 1991 meeting of U.S. EPA, WDNR, WESTON, and KMCC, background soil samples will be collected to a depth of 12 inches and composited. Background sediment samples will be collected to the depth of the "hardpan" (which may occur between 6 inches to 4 feet) and composited.

p. 2-12 The background sediment samples will be collected upstream of the former wood preserving plant (north of Brown Deer Road) in the main channel of the Little Menomonee River, as stated on page 2-10. Background soil samples will not be collected downstream of the Moss-American site within the Little Menomonee River 100-year floodplain.

p. 2-12 Appropriate statistical tests (ANOVA, Newman-Keuls, Tukey's) on Phase II sediment data is deferred until the appropriate use of this data has been agreed upon.

p. 3-1 See response to comment p. 2 above.

RESPONSE TO COMMENTS BY WDNR (GARY EDELSTEIN)

1. Response not appropriate.

2. Response not appropriate.

3. See our response to comment 1-FSP Subsection 2.2.2 by Duane Schuettpelz.

4. WESTON and KMCC will provide an illustration of proposed background sampling locations for review by WDNR and U.S. EPA.

RESPONSE TO COMMENTS BY WDNR (CHARLENE KHAZAE)

Comments on the QAPP

1. It is stated in the second to last paragraph in Section 14.2, page 14-3, of the QAPP and on Figure 14-3 that the reanalysis of samples is one of the corrective actions that will be considered. The need for such an action will be evaluated on a case by case basis and holding times will be one of the criteria that will be examined.

2. In Section 3.3 on page 3-6 of the QAPP, the responsibilities of both the Field Team Leader and the Site Health and Safety Coordinator are specified. On page 14-2, the last paragraph of Section 14.1 states that work may be stopped by the Field Team Leader following instructions from specific management persons.

3. The word "Field" has been removed from the second sentence of Section 4.1, page 4-1.

4. The format of Table 4-1 is required by the U.S. EPA Region V Quality Assurance Section and is utilized in all Region V QAPPs. Frequency indicates the number of rounds of sampling in a particular phase for each compound of interest.

5. All references to quality assurance objectives of precision and accuracy in Section 4.2 of the QAPP have been omitted until the completion of a method performance Study. The method performance study has been designed to evaluate method performance with an off-site Milwaukee area background soil and sediment matrix. The results of the method performance study will be utilized to specify appropriate ranges for precision and accuracy.

6. Matrix spike recoveries at 50 to 150 percent are based on the following published criteria or experimentally determined recoveries for the PAH target analytes or similar compounds:

   a. p-terphenyl d_{14} surrogate criteria recovery range in U.S. EPA-CLP 33 to 141 percent in waters and 18 to 137 percent in soils.

   b. QC acceptance criteria (Table 6 of SW 846 Method 8270, Revision 2. November 1990) benz(a)anthracene (33 to 143 percent), benzo(b)fluoranthene (24 to 159 percent), benzo(k)fluoranthene (11 to 162 percent), benzo(a)pyrene (17 to 163 percent), benzo(ghi)perylene D-219 where D is detected greater than zero, indeno(1,2,3-cd)pyrene (D-171 percent) and pyrene (54 to 120 percent).

c. Soil matrix spike advisory QC limits for U.S. EPA-CLP semivolatiles - acenaphthene (31 to 137 percent) and pyrene (35 to 142 percent).

In addition, please see comment 5 above.

7. The words "existing data (if any)" have been deleted from Section 4.3, page 4-5 of the QAPP.

8. The Health and Safety Plan is a separate, stand alone document that will not be cross-referenced to the QAPP. All health and safety monitoring issues will be discussed in the Health and Safety Plan.

9. It is stated on page 6-3 (second paragraph), and page 6-5, (first full paragraph), that sample paperwork (including the yellow copy of the chain-of-custody form) is the responsibility of the Field Sample Manager. The basis for custody is summarized at the beginning of Section 6, page 6-1. All project documentation (which automatically becomes part of the evidence file) will be under custody if one or all of the custody requirements is fulfilled, and as such, is considered to be secure.

10. All custody seals will be prenumbered and the numbers recorded on the chain-of-custody form. A statement to this effect has been added to Section 6.3, page 6-2 of the FSP where sample documentation completion requirements are specified and also to Subsection 6.1.3, page 6-3 of the QAPP.

11. The right hand column of the WESTON chain-of-custody form includes an area where the temperature of the samples is noted upon receipt at the laboratory. Temperature will be added to the third bullet on page 6-7, Section 6.2 of the QAPP. Batch numbers and tracking numbers are the same - this has been clarified in Subsection 6.2.1, page 6-8, third bullet. All sample paperwork received by the laboratory becomes part of the evidence file for the project, and, as such will be managed as specified in Section 6.3, page 6-12 of the QAPP.

12. The typographical error in Subsection 6.2.5 on page 6-10 of the QAPP has been corrected and is shown on page 6-12 of the revised section.

13. Additional information on frequency of continuing calibration can be found in Section 8.3 of the CPAH SOP in Appendix B. A sentence will be added to the last paragraph of Section 7.2 on page 7-3 of the QAPP referencing the SOP.

14. The word "field" has been removed from the first sentence in Subsection 9.3.2, page 9-3 of the QAPP.

15. Balance calibration for all analytical balances is checked daily per WESTON OP21-06-102, "Daily Balance Check." A sentence stating this point has been added to page 9-4, second to last paragraph.

16. The typographical error on page 11-1, Section 11.2 of the QAPP, first sentence, has been corrected.

Comments on the FSP

1. The format of Table 2-1 is required by the U.S. EPA Region V Quality Assurance Section and is utilized in all Region V QAPPs. Frequency indicates the number of rounds of sampling in a particular phase for each compound of interest.

2. Phosphate-free detergent has been specified in Table 3-1, page 3-4 of the FSP.

3. The glass jars to be utilized during sample collection will have teflon-lined lids. A reference to the U.S. EPA guidelines in Appendix C has been placed as a footnote on Table 5-1, page 5-2 of the FSP.

4. Ice is a form of preservation, as indicated by the word "cool" in Table 5-1 of the FSP.

5. Examples of WESTON's custody seals and sample container labels have been included in Section 6.1 of the QAPP as Figures 6-2 and 6-3, respectively.

Attachment 3

Revised Pages of QAPP/FSP

QUALITY ASSURANCE PROJECT PLAN

FOR

THE MOSS-AMERICAN SITE
MILWAUKEE, WISCONSIN

Prepared by

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

26 February 1992

QUALITY ASSURANCE PROJECT PLAN
FOR
THE MOSS-AMERICAN SITE
MILWAUKEE, WISCONSIN

26 FEBRUARY 1992

Prepared and
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Senior Project Manager

Approved By:

Kurt S. Stimpson, WESTON
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Approved By:

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Betty Lavis, U.S. EPA
Remedial Project Manager

Approved By:

Valerie J. Jones, U.S. EPA
Regional Quality Assurance Manager

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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.

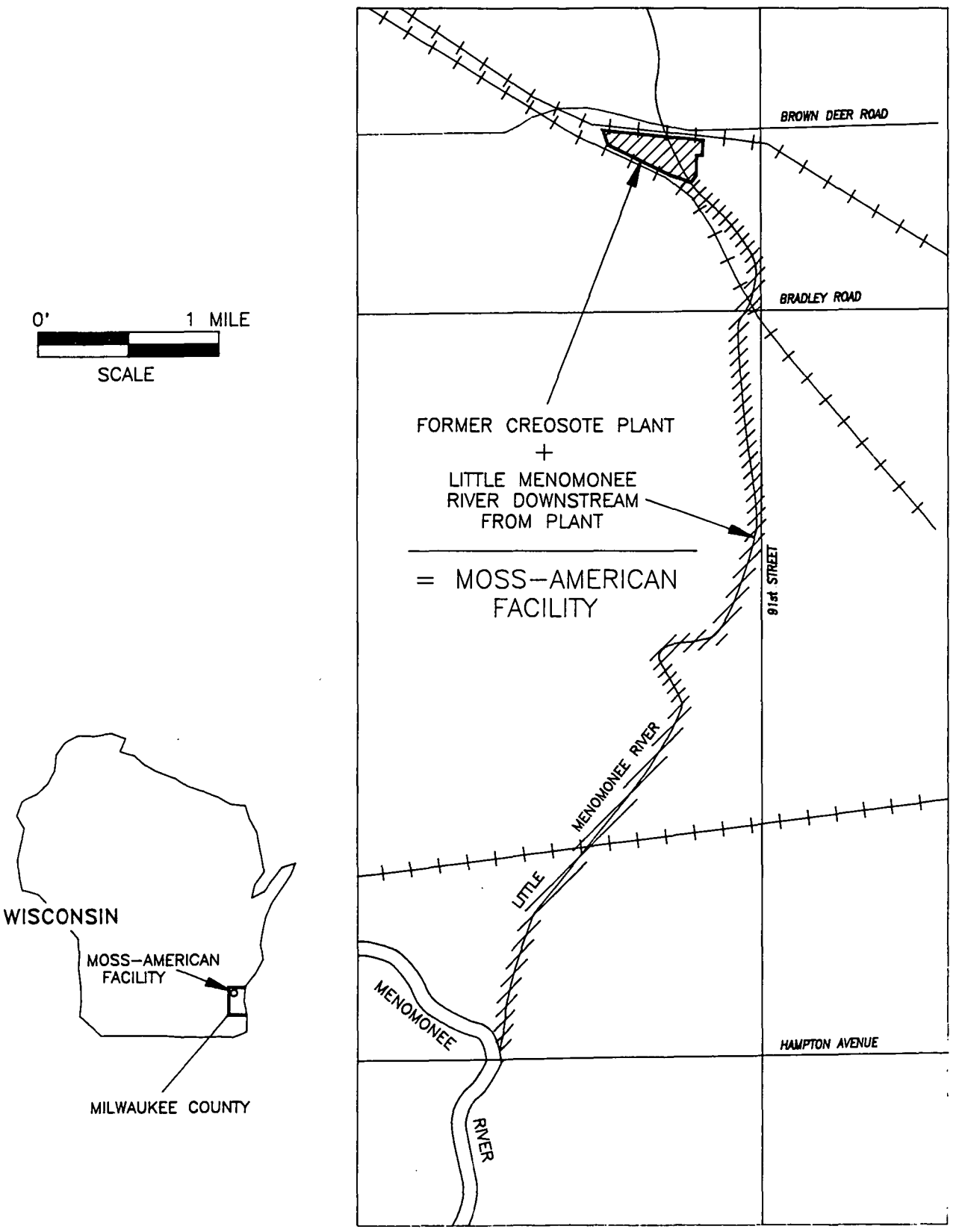
2.2 SITE SETTING

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."

~~The Moss-American site area topography as interpreted by the U.S. EPA in the Remedial Investigation (RI) report for the Moss-American Site (U.S. EPA, 9 January 1990) is as follows:~~

> ~~"The Milwaukee area is part of the Great Lakes section of the Central Lowlands physiographic province. The area is characterized by topographic features resulting primarily from glacial processes. Local relief in the area is generally less than 100 feet giving rise to the flat to rolling topography characteristic of glaciated areas.~~
>
> ~~"The climate for the area is typical for the upper Midwest, with warm summers and cold winters. The average daily temperature range for January and February is 8° to 32°F; for July and August it is 55° to 83°F. The average annual precipitation is~~



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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~~between 29 and 30 inches (water equivalent) with monthly averages ranging from 1.1 inches in February to 3.8 inches in June and in July (SEWRPC, 1976).~~

~~"The Little Menomonee River is tributary to the Menomonee River, which discharges to the Milwaukee Harbor Estuary about 0.9 mile from Lake Michigan. The Menomonee River watershed includes approximately 137 square miles with 10 square miles or 15 percent tributary to the Little Menomonee River. There are approximately 69 miles of perennial stream in the Menomonee River watershed of which 11.9 miles, or 17 percent, are within the Little Menomonee River Watershed.~~

"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.

"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.

~~"Access to the undeveloped county park property is not restricted, although it is limited by railroad tracks on the north and south, and the fenced automobile storage lot on the west. Access from the east is by an undeveloped lot and the river, which limits access to the west half of the park property. The county property west of the river is posted 'No Trespassing–Hazardous Chemicals May Be Present.'~~

~~"Elevations at the site range from 714 to 750 feet. The river drains the entire site. The land slopes up to 3 percent west of the river and up to 10 percent east of the river.~~

"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 topography east of the river has not been modified except for an extensive cut in the field in the far eastern part of the site, which was used for fill material, and levees along the river in a clearing south of the C&NW Railroad.~~

~~"It is not known whether the material cut from the hillside was used on the site or elsewhere.~~

"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 GEOLOGY AND HYDROGEOLOGY~~

~~The following summary of the site geology and hydrogeology is according to the Moss-American RI Report (U.S. EPA, 9 January 1990):~~

~~"The site overlies a surficial water-bearing unit and confining bed. The water-bearing unit consists of a thin mantle of fill, alluvium, and weathered till. This thin layer of material would not yield sufficient water to wells to classify it a true aquifer. The confining bed is the unweathered Oak Creek Formation, which is predominantly a dense silty clay till. On the cross section ([RI report] Figure 2-4), the top two units (F and Aw) constitute the water-bearing unit. The confining bed is labelled 'OC'."~~

~~**Surficial Unit**~~

~~"The surficial unit comprises everything above the confining bed. It includes extensive fill deposits, alluvial deposits along the river, and the weathered upper few feet of the Oak Creek Formation.~~

~~"The fill is highly variable and has been added to the site at different times for different reasons. The most recent fill was added in the western portion of the site to provide a level area for parking in the automobile transfer area. Fill thickness is as great as 10 feet beneath the railroad sidings, decreasing to the south. Approximately 1 foot of fill covers the [former] process area.~~

~~"Alluvial deposits are associated with the Little Menomonee River. They consist of sand and gravel channel deposits and silt and clay flood deposits.~~

~~"The till is part of the Oak Creek Formation, which consists of glacial till, lacustrine clay, silt and sand, and some glaciofluvial sand and gravel. The till is fine grained, commonly containing 80 to 90 percent silt and clay. The till was generally weathered to a depth of 2 to 10 feet, as evidenced by standard penetration test results and color. The weathered till is generally brown, whereas the unweathered till is gray. Penetration resistance was two to four times greater in the unweathered till.~~

~~Hydraulic conductivities from tests on shallow wells completed in the alluvium and weathered Oak Creek Formation ranged from 10^{-3} to 10^{-4} cm/s. Hydraulic properties of the fill are probably comparable, except that more variability would be expected. The saturated thickness of the surficial material averaged above 10 feet in July 1988.~~

~~"The water table as measured in July 1988 is shown in [RI report] Figure 2-5. Groundwater flowed toward the low-lying areas adjacent to the river. These areas are typically marshy wetlands but they were dry at the time of the study because of the drought that summer. Groundwater discharged to these areas either migrates downriver through alluvial sands, or is lost to the atmosphere by evapotranspiration.~~

~~Discharge to the river was apparent only in the vicinity of MW07. Downstream from MW07, the Little Menomonee River was a losing stream at the time of the study.~~

~~"During wetter conditions, the Little Menomonee River is probably a gaining stream (groundwater discharges to the river). At the beginning of the field investigation, before the monitoring wells were installed, ponded water in the wetland between MW11 and MW12 flowed into the river. In addition, groundwater levels dropped as much as 1 foot during a 2 week period in July alone, indicating normal groundwater levels are significantly higher than the measured water levels.~~

~~"Therefore, based on the observations discussed above, the surface-groundwater relationship appears to be seasonal, with groundwater discharging to the river in spring and the river discharging to the groundwater in summer. However, because of the extreme dry conditions at the time of the study, it cannot be assumed that the seasonal fluctuation is representative of normal conditions.~~

~~"Flow volumes across the 715-foot groundwater contour west of the river were calculated for the site ([RI report] Appendix I, Table I-1). The calculations are based on hydraulic properties and the aquifer geometry measured in July 1988. The total lateral groundwater flow volume for the western part of the site was 1,700 gallons per day. Because of the drought conditions this estimate is much lower than the anticipated normal discharge. Normal groundwater discharge is estimated to be between 3,000 and 14,000 gallons per day. The estimates are based on the average and maximum hydraulic conductivities measured in the shallow wells on-site, using a saturated thickness 2 feet less than the thickness of the surficial material ([RI report] Appendix I, Table I-2).~~

~~**Confining Bed**~~

~~"The unweathered part of the Oak Creek Formation consists of a confining bed between the surficial water-bearing unit and underlying regional aquifers. The formation is a dense, silty clay till with interbedded lacustrine units. Below the site, the glacial deposits are approximately 150 feet thick and underlain by the dolomite aquifer (SEWRPC 1976). Sand and gravel lenses or beds of the sand and gravel aquifer were not encountered below the site during the soils investigation, in which soil samples were collected to a depth of 60 feet.~~

~~"The minimum thickness of the confining bed below the site is at least 40 feet. The maximum thickness, if no sand and gravel beds are present, could be about 120 feet.~~

~~The minimum thickness is based on the extent of the investigation (60 feet) minus the overburden thickness (about 20 feet). The maximum thickness is based on SEWRPC information (see [RI report] Figure 2-1).~~

~~"Slug tests conducted on the Oak Creek Formation in the deep and intermediate wells indicate average hydraulic conductivities in the screened zones of 10^{-5} to 10^{-6} cm/s. The screened zones are completed in sandy layers or in the zone believed to be most permeable. Therefore, the hydraulic conductivity of the entire unit is probably less than the values reported. Vertical hydraulic conductivity should be considerably less because of the anisotropy associated with the laminated and thinly bedded lacustrine silts, sands, and clays.~~

~~"Regionally, vertical percolation through the till is a source of recharge for the sand and gravel aquifer and the dolomite aquifer. Regional estimates for deep percolation through the till range from 48,000 to 191,000 gallons per day per square mile (Milwaukee Metropolitan Sewerage District Report), or 6,600 to 26,000 gallons per day for the 88 acre Moss American site [former creosoting plant]."~~

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 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

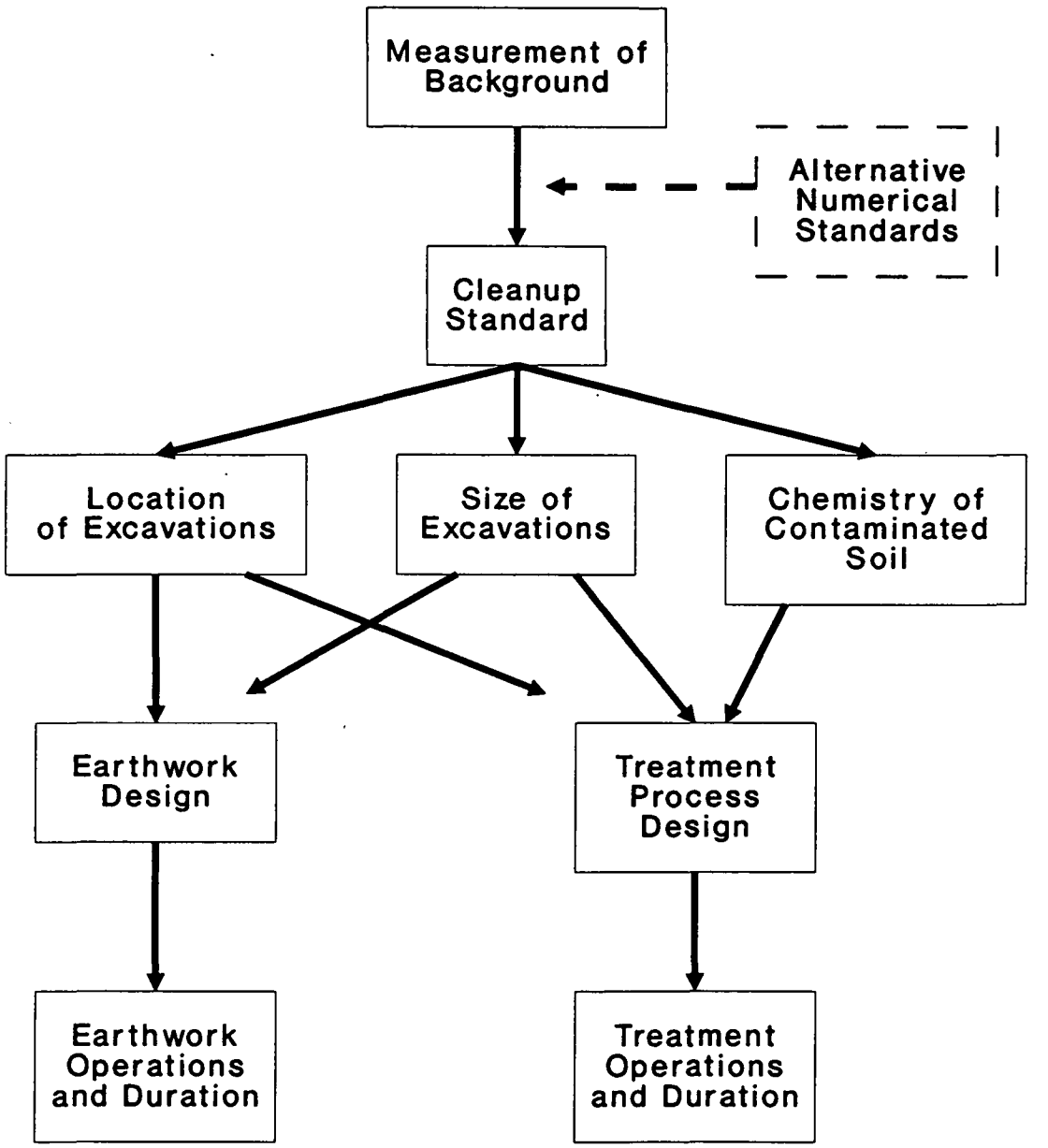

Figure 2-2

Consequences of Determination of Background

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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.

Table 2-1

Data Quality Objectives Summary
Moss-American Site
Milwaukee, Wisconsin

Activity	Matrix	Analytical Parameter	Data Use	Analytical Level
Background sampling	Soil	"A"	BC, HS	V, I
	Sediment	"A"	BC, HS	V, I

Parameter A: Low detection limit CPAH analysis per Appendix B. See Table 8-1 for contaminants of concern.

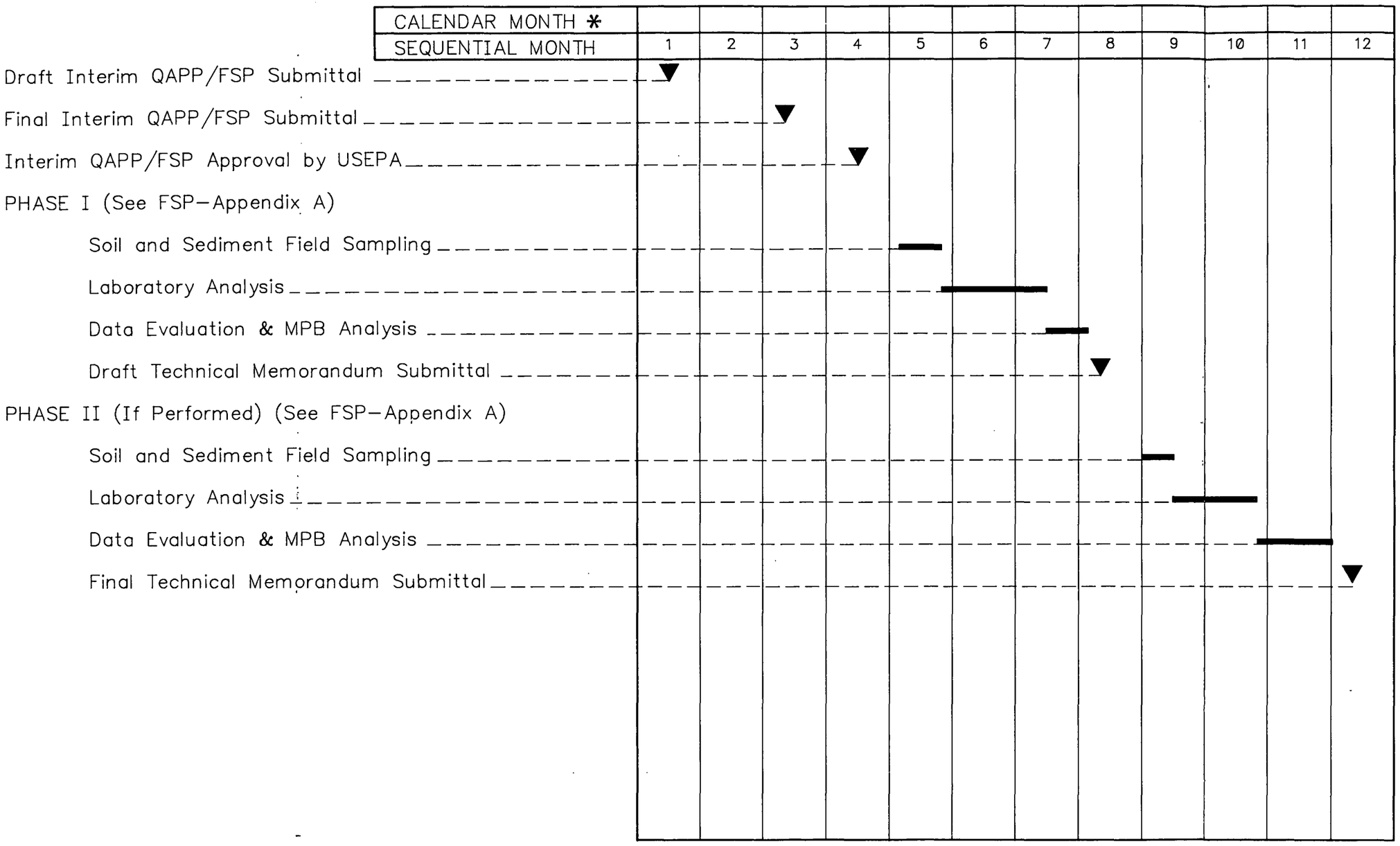
BC - Background characterization.

HS - Health and safety monitoring.

Level V - Analysis by laboratory using non-standard method.

Level I - Qualitative screening using field instruments.

Note: Organization based on "Data Quality Objectives for Remedial Response Activities: Example Scenario" (U.S. EPA/540/G-87/004).



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

68591 D

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,

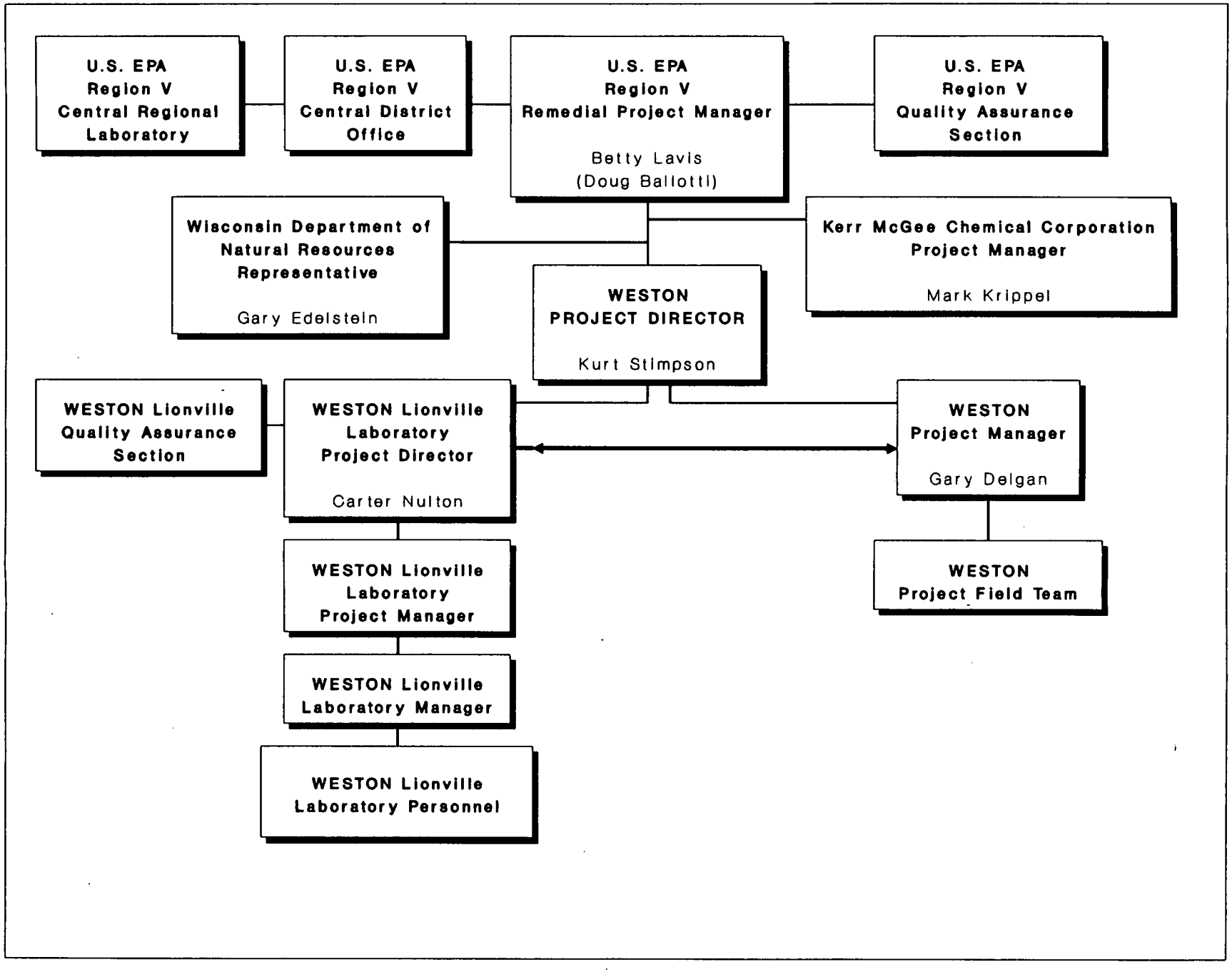


FIGURE 3-1

Project Organization Chart
Predesign Task 2 Activities Moss-American Site

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 CRL and/or~~ the U.S. EPA Region V QAS must review and approve analytical procedures.
- Internally, the KMCC Project Manager will review and approve analytical procedures.

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.

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. The RPD of the MS/MSD will be recorded and evaluated by statistically-generated control limits. ~~For the Moss-American site project, a control limit of ±20 percent will be targeted for precision criteria.~~ For the Moss-American Site project, QA objectives for precision have been omitted until the completion of a method performance study. The method performance study has been designed to evaluate method performance with an off-site Milwaukee-area background soil and sediment matrix. Results of this method performance study will be utilized to specify appropriate ranges for precision.

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 the mid-range of the calibration curve. 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. Results for blank spike analysis will only be reported when matrix spike results are questionable (i.e., inappropriate spike level or matrix effects).~~

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

~~The spike recoveries will be utilized to determine the need for analytical data adjustment. When spike recoveries are within a range of 80 to 120 percent, all associated data will be reported unadjusted for recovery. When spike recoveries fall outside of the 80 to 120 percent range but within the QC limit range of 50 to 150 percent, the associated analytical data will be adjusted according to the level of recovery. If spike recoveries are outside the QC limits for recovery (50 to 150 percent), corrective action measures (outlined in Section 14) that include re-analysis will be implemented in an attempt to bring recovery within the stated QC limits. If after re-analysis matrix spike recoveries are outside of the QC limits, the data will be adjusted to the QC limits based on the level of recovery.~~

As discussed previously for precision, the QA objectives for accuracy will be determined based on the results of the method performance study.

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 95 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.

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.

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 ANALYTICS DIVISION
208 WELSH POOL ROAD
PICKERING CREEK IND. PARK
LIONVILLE, PA 19353

SITE NAME	**DATE**
ANALYSIS	**TIME**
PRESERVATIVE	

SPECIALTY CLEANED CONTAINER

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

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.

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 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.

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 the SOP in Appendix B, Section 8.3. 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.

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.

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.

Blank spikes are performed in duplicate for each preparation batch of 20 or fewer samples. Duplicate analysis allows precision and accuracy data to be generated.

9.3.2 Matrix QC Indicators

Matrix QC indicators include ~~field~~ 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. For Method 8270-modified, the surrogate compounds to be used are: chrysene-d12 and dibenzo (a, h) anthracene-d14. Surrogate recoveries below 20 percent or above 150 percent will be re-extracted and reanalyzed.

Three internal standards will be added to the sample prior to analysis but after sample preparation. Internal standards are indicators of instrument performance and are used to quantitate analyte concentrations in the samples. For Method 8270, the internal standards to be used are: pyrene-dio, 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 ~~analytical balances and~~ 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 will contain control limits for surrogates and MS/MSDs to be used in laboratory data review following completion of the method performance study.

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 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 the 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

Table 12-1

Equipment Maintenance Summary
WESTON Lionville Laboratory

INSTRUMENT	PROCEDURE	FREQUENCY
Finnigan GC/MS	Change column Change injector sleeve Change septa	As needed; depends on ability to meet performance criteria
	Clean ionizer source	Quarterly or as needed
	Change filament	Quarterly or as needed Clean
	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

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 ~~continuing~~ the low concentration calibration standards.

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.

DRAFT INTERIM FIELD SAMPLING PLAN
MOSS-AMERICAN SITE
MILWAUKEE, WISCONSIN

Prepared for

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

Prepared by

Roy F. Weston, Inc.
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Vernon Hills, Illinois 60061

26 February 1992

APPENDIX A

DRAFT INTERIM FIELD SAMPLING PLAN

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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.

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

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

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 ~~upland broadleaf forest~~ 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]			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.

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:

XXX = A consecutively assigned sample number unique to a specified field sampling point. ~~Because of preservation and volume requirements for requested analytes, a sample from one field sampling point may arrive in more than one bottle. In this case, each bottle from the same sampling point will be assigned the same number.~~

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.

- 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 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.

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

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DRAFT NUMBER: 02/25/92

1.0 PURPOSE/APPLICATION

1.1 This method is designed for the determination of polynuclear aromatic hydrocarbons (PAH) in soil and sediment. Table 1 lists the analytes determined by this method.

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 REFERENCE

2.1 EPA SW846, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods", 3rd Edition, Method 8270.

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. 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 (SP No. 21-20-015) 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 coextracted 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 to eliminate false positives and achieve the PQL listed above.

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 5 mL Disposable serological pipets.

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 3/4" 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). Teflon tubing connection to trap 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: Glass, wide mouthed.

6.1.13 Heating mantle

6.1.14 Analytical balance capable of accurately weighing ±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.

6.2 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 spitless 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 um 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 (i.e.; RF, RRT, amount detected - see Sect. 13).

7.0 REAGENTS

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

7.2 Alumina: neutral, 80/200 mesh (Woelm-Super A).

7.3 Silica Gel: high purity grade, 100/200 mesh.

7.4 Sodium Hydroxide Solution: 0.5 N.

7.5 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.

7.6 Acetone, Methylene Chloride, Hexane: pesticide quality or equivalent.

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 are listed in Table 1. All solutions should be discarded six months after the date prepared.

8.2 Initial Calibration: Using a 2 µL injection, analyze each calibration standard according to Section 11.1. Tabulate area responses against concentration for each compound and internal standard. Calculate RRFs for each compound.

$$RRF = \frac{[A_s]\,[C_{is}]}{[A_{is}]\,[C_s]}$$

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 (≤25% RSD), the RRF can be assumed to be invariant and the RFs for the middle concentration will be used for calculations for the remainder of the 12-hour period.

If the RSD is greater than 25% 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 the measurement of the middle level calibration standard. If resulting RRFs vary from RRFs of initial calibration by more than ± 30% or if any RRF is less than 0.25, the test must be repeated using a new calibration standard. Alternatively, a new initial calibration must be analyzed.

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 as a safeguard against laboratory contamination.

A laboratory "method blank" must be run along with each extraction batch (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 for soil and sediment matrices.

9.2 The laboratory will analyze performance evaluation samples as provided by the client. 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 can resume.

9.3 Each sample will be dosed with two surrogates (Table 1) just prior to the extraction process. Surrogates are used to assess method performance, ~~and any sample with a surrogate recovery of less than 20% or greater than 150% will require re-extraction and re-analysis~~.

9.4 Matrix spikes and matrix spike duplicates will be analyzed at a rate of one per 20 samples of the same matrix. QC limits for spike recoveries are ~~50% - 150%~~ and all analytes will be spiked. Reproduceability between MS and MSD recoveries will have acceptance limits of ~~50-150%~~.

9.5 ~~Any time a peak is found that exceeds the instrument calibration a solvent blank will be analyzed to verify there has been no system contamination.~~

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 2/3 full with DCM. Add a few boiling chips. Stopper the bottom of a Soxhlet with glass wool and attach the Soxhlet to the roundbottom flask. Place the sample into the Soxhlet and label properly. Add 100 µL of surrogate to each sample and add 100 µL of spike solution to BS, MS, and MSD. Reflux the system for a minimum of 4 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 quantitative transfer. Add a few boiling chips and a 3-ball Snyder column to the K-D and concentrate the extract on a 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 clean-up procedure (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:

$$\frac{\text{Weight of dry sample (g)}}{\text{Weight of "Wet" sample (g)}} \times 100\% = \% \text{ Solids}$$

$$\% \text{ moisture (reported on Form 1)} = 100\% - \% \text{ Solids}$$

Percent solids should be determined at the time samples are weighed for extraction to ensure an accurate representation of the sample being analyzed.

10.3 Clean-up 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 will be taken during column chromatography to avoid UV irradiation ie., columns will be covered with foil or dark glass columns will be employed.

10.3.1 Transfer the extract from concentrator tube (in DCM) to a 16 mL vial (A) using a disposable pipet. Rinse the concentrator tube with DCM and add to vial A to ensure 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. 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 the 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 (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 2 mL. Dispose of remaining base fraction.

10.3.4 ~~Combined Silica/Alumina Column: The column is prepared as follows: Pack a 5 mL disposable pipet with a glass wool plug, 1.0 mL of neutral alumina, 3.0 mL silica gel, and 0.3 mL anhydrous Na_2SO_4 in sequence (i.e., wool at the tip/bottom and Na_2SO_4 at the top). The column need not be activated. Secure the column vertically with a clamp, tip down.~~

~~Load the extract from 10.3.3 onto the column. Use two 2 mL washes of DCM to rinse vial B and ensure quantitative transfer. Elute the PAH with DCM until about 11 mL of eluate has been collected in a 12 mL vial (C). Blow it down to 1.0 mL.~~

~~Dispose of the used column in a fiber waste drum.~~

NOTE: Cleanup procedure is being evaluated currently.

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 (FC5311) 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 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 Extracts will be diluted if peaks outside of the calibration range are encountered to bring the largest peak to within the calibration range. After dilution, additional internal standard mix will be added to the extract at the amount described in 11.3. The extract will be re-analyzed in order to quantify large peaks. 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 peak will be identified as positive if it meets the following criteria:

12.1 The calculated RT relative to the appropriate internal standard must be within $\pm$ 0.005 RRT units when compared to the continuing calibration standard (or the middle standard of an initial calibration for samples analyzed in the same 12-hour period as the ICAL).

12.2 Peaks with proper 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 confirmation mass does not meet 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 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

13.1 Concentrations are calculated according to the equation:

$$\text{Conc.} = \frac{[A_c]\,[Q_{is}]\,[V]}{[A_{is}]\,[RRF]\,[W]\,[P]}$$

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.

Results are reported in ng/g.

13.2 Detection limits will be determined during method validation.

13.3 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.

13.4 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.

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

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

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 (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 deliverable requirements.

Appendix A

EPA CONTRACT LABORATORY PROGRAM GLASSWARE CLEANING - ORGANICS WESTON SP NO 21-20-015

1.0 PURPOSE

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

2.0 PROCEDURE

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 prepared in Class A volumetric flasks.

1.0 Preparation of Internal Standard Solution

1.1 Purchase the following mixture:

Cambridge Isotope Laboratories (CIL) Catalog No. ES-2044
Deuterium Labeled PAH Surrogate Cocktail
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 (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 IS Stock by 20x with methylene chloride to make an Internal Standard Working Solution:

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

1.4 Add the 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 the following compound as a pure solid:

Dibenz[a,h]anthracene-d_{14}
(CIL Cat. No. DLM-677, D_{14} = 97%)

and purchase the following solution:

1000 µg/mL Chrysene-d_{12} in methylene chloride

(EPA - NOTE: future purchases will probably be Supelco Cat. No. 4-8416M at 2000 µg/mL and will require different dilutions to make a Working Standard)

2.2 Weigh approximately 10 mg of the dibenz[a,h]anthracene-d_{14} to the nearest .1 mg in a 10 mL Class A volumetric flask and dilute to volume with methylene chloride (final conc. = 1000 µg/mL)

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

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

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

2.4 Dilute the SS stock by 100x with methylene chloride to make a Surrogate Standard Spiking Solution:

example: $1\ mL \times \frac{100\ \mu g}{mL} \times \frac{1}{100\ mL} = \frac{1\ \mu g}{mL}$

2.5 Add the SS spike solution to all samples and blanks before extraction at a rate of 100 µL per sample. This results in 100 ng of each SS added to each of the samples (10 g).

2.6 Other convenient dilutions may be used to reach the final SS spike solution concentration of 1 µg/mL.

3.0 Preparation of Matrix/Blank Spiking Solution.

3.1 Purchase the following mixture:

Supelco Cat. No. 4-8905 Polyneuclear Aromatic Hydrocarbon Mix (or equivalent - adjust concentrations and dilutions as necessary). Contains all analytes (See Table 1) at 2000 µg/mL in methylene chloride/benzene (1:1).

3.2 Dilute 1 mL of the above to 10 mL with methylene chloride to make an Analyte Stock Solution:

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

3.3 Dilute the analyte stock by 200x with methylene chloride to make a Matrix/Blank Spiking Solution:

example: $$0.5\ mL \times \frac{200\ \mu g}{mL} \times \frac{1}{10\ mL} = \frac{1\ \mu g}{mL}$$

3.4 Add the Matrix/Blank Spiking Solution to the required samples and/or blanks at a rate of 100 µL per sample. This results in 100 ng of each spike compound (i.e., each analyte) added to the appropriate samples and blanks.

3.5 Other convenient dilutions may be used to reach the final analyte concentration of 1 µg/mL in the Matrix/Blank Spiking Solution.

4.0 Preparation of Calibration Standards

4.1 Calibration Standard Solutions will be prepared from the IS, SS, and Analyte Stocks prepared in 1.2, 2.3, and 3.2 above, respectively.

4.2 Any convenient serial dilutions may be used to make the solutions below. If a particularly 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 so as to allow a given component (IS, SS, or analyte) to be changed and easily checked verses the other components.

4.4 Prepare five solutions of surrogate compounds and five of analytes 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:

20, 50, 200, 500, 2000 ng/mL

(These may, at times, be refered to as CC1...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.

TABLE 1
PAH COMPOUNDS

SIM DESC	QUAN REF	COMPOUND		CAS #	QUAN MASS	CONF. MASS	C/Q RATIO	SPIKE AMOUNT
H4	IS#1	Pyrene-d10	Int. Std. #1	1718-52-1	212.14	NA	NA	NA
H4	IS#1	Chrysene-d12	Surr. Std. #1	1719-03-5	240.17	NA	NA	100 ng
H4	IS#1	Benzo(a)Anthrancene		56-55-3	228.09	226.09	.12-.50	100 ng
H4	IS#1	Chrysene		218-01-9	228.09	226.09	.13-.52	100 ng
H5	IS#2	Benzo(b)Fluoranthene		205-99-2	252.09	126.05	.04-.16	100 ng
H5	IS#2	Benzo(k)Fluoranthene		207-08-9	252.09	126.05	0.5-.18	100 ng
H5	IS#2	Benzo(a)Pyrene-d12	Int. Std. #2	63466-71-7	264.17	NA	NA	NA
H5	IS#2	Benzo(a)Pyrene		50-32-8	252.09	126.05	.04-.17	100 ng
H6	IS#3	Indeno(1,2,3-cd)Pyrene		193-39-5	276.09	274.09	.11-.46	100 ng
H6	IS#3	Dibenz(a,h)Anthracene-d14	Surr. Std. #2	13250-98-1	292.17	NA	NA	100 ng
H6	IS#3	Dibenz(a,h)Anthracene		53-70-3	278.09	279.09	.12-.50	100 ng
H6	IS#3	Benzo(g,h,i)Perylene-d12	Int. Std. #1	93951-66-7	288.32	NA	NA	NA
H6	IS#3	Benzo(g,h,i)Perylene		191-24-2	276.09	274.09	.11-.46	100 ng

NA = Not Applicable
ND = Not Determined (to be established during method validation)
~~PQL = Practical Quantitation Limit based on 10 g sample before %solids factored in. (Target Limit is 1 ng/g)~~

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 min.)

Injection Volume: 2 μL

Injector Temperature: 280°C

Transfer Line Temperature: 250-300°C

Column Oven Temperature: 60°C for 1 min
60°C to 240°C at 10°C/min
240° to 300°C at 15°C/min
Hold at 300°C for the duration of the analysis (approx. 5 min)

Total Analysis Time: Approx. 27 min

ORGANIC ANALYSIS PROTOCOL POLYNUCLEAR AROMATIC HYDROCARBONS (PAH) BY CAPILLARY COLUMN GC/MS (SIM) TECHNIQUES FOR MOSS-AMERICAN SITE

These Approval Signatures Are Kept on File with WESTON's Analytics Division QA Standard Practice Records

DRAFT NUMBER: 02/25/92

	Printed Name:	Signature/Date:
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QA Section Manager _____

Historical File: Revision 00: 10/24/91

Reasons for Change, Draft 02/25/92:

- revised Appendix B, 4.4 and 4.5
- revised Table 1 PAH Compounds

ORGANIC ANALYSIS PROTOCOL POLYNUCLEAR AROMATIC HYDROCARBONS (PAH) BY CAPILLARY COLUMN GC/MS (SIM) TECHNIQUES FOR MOSS-AMERICAN SITE

DRAFT NUMBER: 02/25/92

I have read and understood this in-house SOP. I agree not to deviate from this in-house SOP without my supervisor's approval. Any approved deviations will be documented with the raw data and co-signed by my supervisor; with copy to the appropriate client file and the laboratory manager.

Printed Name:	Signature/Date: