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9 May 1997

EG&G Mound Applied Technologies, Inc.
Attn.: Mr. James Rigano
P.O. Box 3000, OSW-4
Miamisburg, Ohio 45343-3000

EG&G MOUND-04-04----9705290001

RE: ER Program, Mound Plant
BOA 24251, Task Order No. 68282
Methods Compendium - Update Packet 4

RFW WO# 05376-060-001 x07

Dear Mr. Rigano:

Roy F. Weston, Inc. (WESTON®) has completed Update Package 4 for the Compendium. This update contains three (3) new procedures (listed in the table below), 21 revised procedures, and four (4) revised Table of Contents. A complete list of attachments is shown on the Update Package 4 Content List.

Method	Title
S-001	General Instructions for Field Personnel
S-002	Soil Sampling with a Spade and Scoop
S-003	Soil and Rock Borehole Logging and Sampling

The three new procedures being issued in this update are revised versions of the ER Program SOPs that were originally included in Appendix A of the OU9 QAPP. The procedures were revised and issued as a part of the Compendium in response to concerns about the usefulness of the original procedures. The revised procedures are written to be simple and easy to follow.

The other revised procedures were changed primarily in response to USEPA comments on Update Package 2. A copy of the response to the USEPA comments is included as an attachment to this letter.

Please discard any previous versions of these methods and Table of Contents and insert the revisions attached to this letter. The new methods (S-001 to S-003) should be placed in Section 5 of the binder. When the next set of procedures for Section 5 are issued, a second binder will be

issued. No date has been set for the next update. If you have any questions, please call me or Craig Stoll at (513) 825-3440.

Very truly yours,

ROY F. WESTON, INC.

Gordon S. Horn, P.E., DEE
Alternate Project Manager

cas/vkb

Attachments

cc: Richard Christopher (EG&G)

Update Package 4 Content List
9 May 1997

Method Number	Revision	Description	Source
TOC 1	5.0	Table of Contents for the Entire Compendium	NA
TOC 2	4.0	Table of Contents for Section 1 (Analytical Methods)	NA
A-010	3.0	Explosives Analysis by USEPA Method 8330	QAPP
A-020	3.0	Volatiles Organic Analysis/EPA Method 8020	Compendium
A-021	3.0	Volatiles Organics Analysis (VOA)/EPA Method 601	Compendium
A-022	3.0	Gasoline Range Organic (GRO)/EPA Method 8015	Compendium
A-024	3.0	Diesel Range Organic (DRO)/EPA Method 8015	Compendium
Q-012	2.0	Electronic Data Deliverable Format Specification - MEIMS Non-CLP Standard Format	Compendium
TOC 3	3.0	Table of Contents for Section 4 - Data Validation Methods	NA
DV-002	2.0	Volatile Organic Method 8021 Data Validation	QAPP
DV-003	2.0	CLP Semi-Volatile Organic Analysis (SVOA) Data Validation	QAPP
DV-005	2.0	CLP Inorganic (Metals) Data Validation	QAPP
DV-006	2.0	Cyanide Data Validation	QAPP
DV-008	2.0	Total Dissolved Solids (TDS) and Total Suspended Solids (TSS) Data Validation	QAPP
DV-010	2.0	Explosives Data Validation	QAPP
DV-014	2.0	Tritium Data Validation	QAPP
DV-015	2.0	Gamma Spectrometry Data Validation	QAPP
DV-018	2.0	Volatile Organic Analysis (VOA)/Method SW8030 Data Validation	QAPP
DV-019	2.0	Hexavalent Chromium Data Validation	Compendium
DV-020	2.0	Volatile Organic Analysis (VOA)/Method SW8020 Data Validation	Compendium
DV-021	2.0	Volatile Organic Analysis (VOA) by EPA 601 Data Validation	Compendium
DV-022	2.0	Gasoline Range Organic (GRO) Analysis by SW8015 Data Validation	Compendium
DV-023	2.0	Total Petroleum Hydrocarbons (TPH) by EPA 418.1 Data Validation	Compendium
DV-024	2.0	Diesel Range Organic (DRO) Analysis by SW8015 for Data Validation	Compendium
TOC 4	1.0	Table of Contents for Section 5 - Field SOP Methods	NA
S-001	1.0	General Instructions for Field Personnel	Compendium
S-002	1.0	Soil Sampling with a Spade and Scoop	Compendium
S-003	1.0	Soil and Rock Borehole Logging and Sampling	Compendium

Response to United States EPA Comments
on
the Mound Plant Methods Compendium

1. Cover Letter, Reason for Re-issue, Method A-006: The cover letter states that Method A-006 was re-issued to include LCS requirements in Table 4.3. There is no Table 4.3 in Method A-006. It appears that it was Table 4.2 that was revised to include LCS requirements, please revise.

   The correct reference should have been Table 4.2.

2. Method A-020 - Volatiles (BTEX) Organics/EPA Method 8020: Page 5 of 6, Table 6.2.1, Parameters Column: Remove the reference to the SW8020 method, this method is SW8240/SW8260.

   Table 6.2.1 has been revised to remove the reference to SW8020.

3. Method A-020 - Volatiles (BTEX) Organics/EPA Method 8020: Page 5 of 6, Table 6.2.2, System Monitoring Compounds. Toluene is listed twice under acceptance criteria; should the second toluene be deleted and replaced with xylene?

   Yes, the error was identified after sending out the method. The error has been corrected and the revised method is enclosed with this response.

4. Method A-020 - Volatiles (BTEX) Organics/EPA Method 8020: Page 6 of 6, Table 6.2.3, Spike Concentrations. It is unclear what the "*" is, please define.

   The '' is a carry-over from the QAPP. The '*' indicates that the laboratory will select a spiking concentration which will result in a mid-range response based on the expected contaminant levels in the sample. The methods which use the undefined '*' include: A-10, A-20, A-021, A-022, and A-023. These methods have been corrected and are being re-issued with this response.*

5. Method A-024 - Petroleum Hydrocarbons/EPA Method 8015 DRO Title Page: The title page should be changed to Total Petroleum Hydrocarbons instead of Volatile Organic Analysis.

   The title page was revised to Diesel Range Organics (DRO) instead of Total Petroleum Hydrocarbons. Total Petroleum Hydrocarbons was not used to eliminate any confusion between DRO and method 418.1.

6. Method A-024 - Petroleum Hydrocarbons/EPA Method 8015 DRO Page 1 of 4, Section 1.1, Description: Method A-022 for GRO is referenced, change this to Method A-024 for DRO.

The reference to method A-022 was intentional. The reference was included for a novice user who might need to select between DRO and GRO. Method A-022 includes a reference to A-024.

7. Method A-024 - Petroleum Hydrocarbons/EPA Method 8015 DRO Page 3 of 4, Table 4.2/4.3, LCS Acceptance Criteria: Table 4.2 references Table 4.3 for LCS criteria, but LCS criteria is not listed in Table 4.3.

   Table 4.3 was revised to include a reference for LCS. The same omission was observed in method A-022 and was corrected.

8. Method DV-001 - CLP Volatile Organic Data Validation Page 4 of 6, Section 3.4, CRQL Table, Last Row: Please explain the NA for the CRQL for Iodomethane in water.

   In the OU9 QAPP, iodomethane in water was not a target analyte. Therefore, iodomethane is not a target analyte in method A-001 or DV-001.

9. Method DV-002 - Volatile Organic Method 8021 Data Validation Page 4 of 7, Section 3.4, Calibration: In the third sentence of the first paragraph, it should read "the results associated with the non-compliant standard must be estimated (J, UJ)."

   The text matches the text in the OU9 QAPP. Based on the intended data uses at Mound, the OU9 QAPP authors and reviewers decided that non-detect results associated with non-compliant calibration criteria did not require qualification and were usable as reported.

10. Method DV-002 - Volatile Organic Method 8021 Data Validation Page 4 of 7, Section 3.4, Surrogates: The sentence states that if surrogate recoveries are still outside limits, then positive sample results should be estimated (J). However, if surrogate recoveries are below QC limits and >10%, then shouldn't the non-detected results also be qualified as estimated (UJ)?

    The text matches the text in the OU9 QAPP. Based on the intended data uses at Mound, the QAPP authors and reviewers decided that non-detect results associated with surrogate outlier recoveries greater than 10 percent and less than the lower surrogate recovery limit would be used without qualification.

11. Method DV-002 - Volatile Organic Method 8021 Data Validation Page 5 of 7, Section 3.4, Field Duplicate: The last sentence should read, "If one of the sample results is >PQL and the other is non-detected, then both results are estimated (J, UJ).

    The text matches the text in OU9 QAPP. Per your request, the procedure has been revised to clarify that both the non-detect and the detected results should be qualified estimated (J,UJ).

12. Method DV-002 - Volatile Organic Method 8021 Data Validation Attachment I - Laboratory Completeness Checklist: Method 8021 is a GC method. Delete all GC/MS references and include applicable GC references.

    In order to limit the number of completeness checksheets, a separate checksheet was not created for method 8021 nor the other GC volatile methods. Per your request, we have revised the checklists to be more specific to GC analysis. The checksheets were added to DV-002, DV-018, DV-020, DV-021, DV-022, and DV-024.

13. Method DV-002 - Volatile Organic Method 8021 Data Validation Attachment III - Matrix Spike Recovery Outliers: Soil QC limits are listed in this table as 60%-140% (?30% RPD) for individual compounds. Method A-002 lists these limits as NA. Are the limits in this table advisory limits?

    Method 8021 has historically only been used at Mound for water analyses. Consequently, method A-002 only defines the limits for water. The soil limits on the table were simply retained from the original checklist model. For the purposes of Mound, the limits are advisory and will probably never be used.

14. Method DV-003 - CLP Semi-Volatile Organic Data Validation Attachment III - Holding Time Summary: Add a column for date extracted.

    The summary form was also used in DV-023. The form was corrected in both methods.

15. Method DV-005 - Inorganic (Metals) Data Validation Page 4 of 8, Section 3.4, Holding Time: Note that all TAL metals have a holding time of 180 days, with the exception of Mercury (28 days).

    The text matches the OU9 QAPP text. However, the method was revised to clarify the holding time for mercury is 28 days, as requested.

16. Method DV-005 - Inorganic (Metals) Data Validation Page 5 of 8, Section 3.4, Matrix Spike Recovery Table: The cells for Furnace AA concentrations of Lithium, Molybdenum, and Bismuth are blank: explain why or add NA if they are not applicable here.

    The table matches the table in the OU9 QAPP. The table was revised to include 'NA' in the empty cells.

17. Method DV-005 - Inorganic (Metals) Data Validation Page 7 of 8, Section 3.5.5, *Calibration Forms:* RF and RRF references are not applicable for inorganic methods.

    Section 3.5.5 was copied directly from the QAPP. The original text was intended to described Calibration Forms for all the data validation methods. Because the text was copied directly from the QAPP, it included references for some organic calibration

verification forms. The text was revised from "... outlying RF, RRF, RSD, or %D..." to "... outlying criteria."

18. Method DV-006 - Cyanide Data Validation Page 4 of 5, Section 3.4, Laboratory Control Sample: Delete references to Li, Mo, Sb, and Sn and replace with cyanide.

    The text was revised. References to Li, Mo, Sb, and Sn were replaced by cyanide.

19. Method DV-008 - Total Dissolved Solids (TDS) and Total Suspended Solids (TSS) Data Validation Attachment III - Data Summary Table: Is the second table for LCS? Please insert a title at the top of the table.

    Yes, the table has been titled.

20. Method DV-010 - Explosives Data Validation Attachment III - Matrix Spike Recovery Outliers Table: The RPDs listed for soil and water do not match those specified in Method A-010, Table 4.3.

    The limits were revised to match method A-010.

21. Method DV-010 - Explosives Data Validation Attachment III - Field Duplicate Table: The RPDs listed for soil and water in Section 3.4 (pg. 6 of 7) of this method (50% for soil 25% for water) do not match those specified in this table (35% for water).

    The text matches the text in the OU9 QAPP. The water limit in the method DV-010 was revised to 35%. The criterion for soil will remain unchanged. In the analytical procedure, no limit is listed for soil.

22. Method DV-014 - Tritium Data Validation Attachment III - Matrix Spike Summary Table: The QC limit according to the text in Section 3.4 of this method and Table 4.2 of Method A-014 is ±4SD. The QC limit listed on this table is listed as ±3SD. Please revise this.

    The criterion in section 3.4 has been revised to ±4SD.

23. Method DV-015 - Gamma Spectrometry Data Validation Page 2 of 5, Section 3.2, *Calculations:* Text appears to be missing at the end of this section.

    The line "... the validators verify:" was removed.

24. Method DV-015 - Gamma Spectrometry Data Validation Attachment III - Replicates Summary Table: The QC limit according to the text in Section 3.4 of this method and Table 4.2 of Method A-015 is ±4SD of the normalized range. The QC limit listed on this table is listed as ±SD. Please revise this.

The criterion has been revised to read ±4SD.

25. Method DV-015 - Gamma Spectrometry Data Validation Attachment III - Field Duplicate Summary Table: The QC limit according to the text in Section 3.4 of this method and Table 4.1 of Method A-015 is ±4SD of the normalized range. The QC limit listed on this table is listed as ±SD. Please revise this.

    The criterion has been revised to read ±4SD.

26. Method DV-018 - Volatile Organic Analysis by SW8030 Data Validation Page 5 of 7, Section 3.4, Field Duplicate: The last sentence should read, "If one of the sample results is >PQL and the other is non-detected, then both results are estimated (J, UJ)."

    The text matches the text in the OU9 QAPP. The sentence was revised to add (UJ).

27. Method DV-018 - Volatile Organic Analysis by SW8030 Data Validation Attachment I - Laboratory Completeness Checklist: Method 8030 is a GC Method. Delete all GC/MS references and include applicable GC references.

    See response 12.

28. Method DV-019 - Hexavalent Chromium Data Validation Attachment III - Data Summary Table: The initial calibration coefficient criteria is listed as >0.9995 in this table, but is listed as >0.995 in Section 3.4 of the text on this method. Please correct this inconsistency.

    The table was corrected to specify the limit as 0.995.

29. Method DV-020 - Volatile Organic Analysis by SW8020 Data Validation Attachment III - Surrogate Recovery Outliers for Method SW8240: The soil QC limits for surrogates are not consistent with the QC limits specified in Table 6.2.3 in Method A-020.

    The limits were revised to match method A-020 requirements for soil.

30. Method DV-021 - Volatile Organic Analysis by EPA601 Data Validation Attachment I - Laboratory Completeness Checklist: Method 601 is a GC Method. Delete all GC/MS references and include applicable GC references.

    See response 12.

31. Method DV-022 - Volatile Organic Analysis by SW8015 for Gasoline Range Organics Data Validation Page 4 of 6, Section 3.4, Surrogates: The sentence states that if surrogate recoveries are still outside limits, then positive sample results should be estimated (J). However, if surrogate recoveries are below QC limits and >10%, then shouldn't the non-detected results also be qualified as estimated (UJ)?

The qualification guidelines were based on the existing OU9 QAPP guidelines (see response 10). Based on these guidelines, non-detect results associated with surrogate recoveries greater than 10 percent and less than the lower surrogate recovery limit are not qualified.

32. Method DV-022 - Volatile Organic Analysis by SW8015 for Gasoline Range Organics Data Validation Page 4 of 6, Section 3.4, LCS: The sentence states that LCS recoveries should be verified with those specified in Method A-022. However, LCS recovery limits are not listed in Method A-022.

    See response 7.

33. Method DV-022 - Volatile Organic Analysis by SW8015 for Gasoline Range Organics Data Validation Page 5 of 6, Section 3.4, Field Duplicate: The last sentence should read, "If one of the sample results is >PQL and the other is non-detected, then both results are estimated (J, UJ)."

    Per your comment, the procedure has been revised to clarify that both the non-detect and the detected results should be qualified estimated (J,UJ).

34. Method DV-022 - Volatile Organic Analysis by SW8015 for Gasoline Range Organics Data Validation Attachment I - Laboratory Completeness Checklist: Method 601 is a GC Method. Delete all GC/MS references and include applicable GC references.

    See response 12.

35. Method DV-023 - Total Petroleum Hydrocarbons by EPA418.1 Data Validation Attachment I - Laboratory Completeness Checklist Method 418.1 is an infrared spectrometry method. This checklist is used for GC/MS methods. Please revise as necessary.

    The checklist was revised.

36. Method DV-023 - Total Petroleum Hydrocarbons by EPA418.1 Data Validation Attachment III - Data Summary Table: The Continuing Calibration criteria listed in Table 4.2 (pg. 3 of 4) of Method A-023 is 80-120% recovery. The criteria listed in this table is %D<15. Please revise as necessary.

    The criteria in DV-023 was revised to %D ≤20%.

37. Method DV-024 - Volatile Organic Analysis by SW8015 for Diesel Range Data Validation Page 4 of 6, Section 3.4, Surrogates: The sentence states that if surrogate recoveries are still outside limits, then positive sample results should be estimated (J). However, if surrogate recoveries are below QC limits and >10%, then shouldn't the non-detected results also be qualified as estimated (UJ)?

The qualification guidelines were based on the existing OU9 QAPP guidelines (see response 10). Based on these guidelines, non-detect results associated with surrogate recoveries greater than 10 percent and less than the lower surrogate recovery limit are not qualified.

38. Method DV-024 - Volatile Organic Analysis by SW8015 for Diesel Range Data Validation Page 4 of 6, Section 3.4, LCS: The sentence states that LCS recoveries should be verified with those specified in Method A-024. However, LCS recovery limits are not listed in Method A-024.

    See response 7.

39. Method DV-024 - Volatile Organic Analysis by SW8015 for Diesel Range Data Validation Page 5 of 6, Section 3.4, Field Duplicate: The last sentence should read, "If one of the sample results is >PQL and the other is non-detected, then both results are estimated (J, UJ)."

    Per your request, the procedure has been revised to clarify that both the non-detect and the detected results should be qualified estimated (J, UJ).

40. Method DV-024 - Volatile Organic Analysis by SW8015 for Diesel Range Data Validation Attachment I - Laboratory Completeness Checklist: Method 8015 is a GC Method. Delete all GC/MS references and include applicable GC references.

    See response 12.

Environmental Restoration Program

METHODS COMPENDIUM

Mound Plant
Miamisburg, Ohio

Department of Energy
Ohio Field Office
EG&G Mound Applied Technologies

Table of Contents

Introduction

Section 1 — Analytical Methods

Method Number	Method Description	Document
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A-002	Volatiles Organic Analysis/EPA Method 8021	QAPP
A-003	CLP Semi-Volatile Analysis/CLP SOW OLM01.8	QAPP
A-004	CLP Pesticide Analysis/CLP SOW OLM01.8	QAPP
A-005	CLP Metals/ILM03.0	QAPP
A-006	Cyanide/CLP SOW ILM03.0	QAPP
A-007	General Chemistry	QAPP
A-008	Total Dissolved Solids/Total Suspended Solids	QAPP
A-009	Total Organic Carbon	QAPP
A-010	Explosives Analysis by USEPA Method 8330	QAPP
A-011	Alkalinity	QAPP
A-012	Isotopic Uranium, Isotopic Plutonium, and Isotopic Thorium by Alpha Spectrometry	QAPP
A-013	Isotopic Americium^{241} in Water by Alpha Spectrometry	QAPP
A-014	Tritium	QAPP
A-015	Gamma Spectrometry	QAPP
A-016	Strontium^{90}	QAPP
A-017	Isotopic Radium^{226} in Water	QAPP
A-018	Volatiles Organic Analysis/EPA Method 8030	QAPP
A-019	Hexavalent Chromium/EPA Method SW7196A	PRS99/100
A-020	Volatiles Organics Analysis/EPA Method 8020	Compendium
A-021	Volatiles Organics Analysis/EPA Method 601	Compendium
A-022	Gasoline Range Organic (GRO)/EPA Method 8015	Compendium
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A-024	Diesel Range Organic (DRO)/EPA Method 8015	Compendium

Section 2 — Field Methods

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F-002	Gamma Spectrometry	Compendium
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Section 3 — Quality Assurance Methods

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Q-002	Chain-of-Custody Procedures	QAPP
Q-003	Documentation Requirements	QAPP
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Q-015	Compendium Methods Word Processing Format Specifications	Compendium

Section 4 — Data Validation Methods

Method Number	Method Description	Document
DV-001	CLP Volatile Organic Data Validation	QAPP
DV-002	Volatile Organic Method 8021 Data Validation	QAPP
DV-003	CLP Semi-Volatile Organic Analysis (SVOA) Data Validation	QAPP
DV-004	CLP Pesticide/PCB Data Validation	QAPP
DV-005	CLP Inorganic (Metals) Data Validation	QAPP
DV-006	Cyanide Data Validation	QAPP
DV-007	General Chemistry Data Validation	QAPP
DV-008	Total Dissolved Solids (TDS) and Total Suspended Solids (TSS) Data Validation	QAPP
DV-009	Total Organic Carbon (TOC) Data Validation	QAPP
DV-010	Explosives Data Validation	QAPP
DV-011	Alkalinity Data Validation	QAPP
DV-012	Uranium, Plutonium, and Thorium Alpha Spectrometry Data Validation	QAPP
DV-013	Americium Data Validation	QAPP
DV-014	Tritium Data Validation	QAPP
DV-015	Gamma Spectrometry Data Validation	QAPP
DV-016	Strontium^{90} Data Validation	QAPP
DV-017	Radium^{226} Data Validation	QAPP
DV-018	Volatile Organic Analysis (VOA)/Method SW8030 Data Validation	QAPP

Method Number	Method Description	Document
DV-019	Hexavalent Chromium Data Validation	QAPP
DV-020	Volatile Organic Analysis (VOA)/Method SW8020 Data Validation	Compendium
DV-021	Volatile Organic Analysis (VOA) by EPA 601 Data Validation	Compendium
DV-022	Gasoline Range Organic (GRO) Analysis by SW8015 Data Validation	Compendium
DV-023	Total Petroleum Hydrocarbons (TPH) by EPA 418.1 Data Validation	Compendium
DV-024	Diesel Range Organic (DRO) Analysis by SW8015 for Data Validation	Compendium

Section 5 — Field Standard Operating Procedures

Method Number	Method Description	Document
S-001	General Instructions for Field Personnel	QAPP
S-002	Soil Sampling with a Spade and Scoop	QAPP
S-003	Soil and Rock Borehole Logging and Sampling	QAPP

INTRODUCTION

INTRODUCTION

In 1995, the DOE Mound Plant Environmental Restoration (ER) department, the Ohio Environmental Protection Agency (OEPA) and the United States Environmental Protection Agency (USEPA) developed a program to identify and evaluate potential release sites and, if required, remediate the contaminated sites. These potential release sites were identified on the basis of data collected during previous sampling and investigative programs. Because there is previous data which characterizes the contamination at these release sites, the sampling and analysis methods selected for further evaluating the release sites could, in many cases, be highly focused. For example, if a potential release site had been identified to have chromium contamination and more information was required, then DOE resources could be used to focus the sampling and analysis methods on collecting additional chromium data and not spent confirming the lack of other contaminants (e.g. volatile organics, semi-volatile organics, other metals, etc.)

Given this change in the focus of the DOE Mound Plant mission, this Compendium was generated. The Compendium was designed to act as a depository for sampling, analysis, and quality control methods implemented on the plant site. The initial Compendium methods were extracted from the Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan (QAPP), April 1995, revision 4. Because the QAPP was approved for evaluating the nature and extent of contamination throughout the plant site and contains extensive target analyte lists, the QAPP methods were included in the Compendium to provide a common basis in the event potential release sites are identified which lack adequate data to develop a focused target analyte list.

The Compendium is divided into five sections: Analytical Methods, Field Methods, Quality Assurance Methods, Data Validation Methods, and Field Standard Operating Procedures. Methods within each section have been identified using a prefix and a sequential number. The prefix codes are shown below.

Type of Method	Prefix
Analytical Methods	A
Field Methods	F
Quality Assurance Methods	Q
Data Validation Methods	DV
Field Standard Operating Procedures	S

At the beginning of the each section, there is a short introduction and a table of contents for the section. The methods extracted from the QAPP can be identified in three ways: by the table of contents at the beginning of each section, within the introduction to each of the sections, or on the title page of each of the methods where the source document is listed. As new methods are

added, the table of contents will be updated and distributed to the copy holders with the new methods. Each of the new methods will include a source document reference and document date. The source document and document date will be used to identify the first potential release site approved for the use of the method.

ANALYTICAL METHODS

Table of Contents

Method Number	Method Description	Document
A-001	Volatile Organic Analysis by CLP SOW OLM01.8	QAPP
A-002	Volatiles Organic Analysis/EPA Method 8021	QAPP
A-003	CLP Semi-Volatile Analysis/CLP SOW OLM01.8	QAPP
A-004	CLP Pesticide Analysis/CLP SOW OLM01.8	QAPP
A-005	CLP Metals/ILM03.0	QAPP
A-006	Cyanide/CLP SOW ILM03.0	QAPP
A-007	General Chemistry	QAPP
A-008	Total Dissolved Solids/Total Suspended Solids	QAPP
A-009	Total Organic Carbon	QAPP
A-010	Explosives Analysis by USEPA Method 8330	QAPP
A-011	Alkalinity	QAPP
A-012	Isotopic Uranium, Isotopic Plutonium, and Isotopic Thorium by Alpha Spectrometry	QAPP
A-013	Isotopic Americium^{241} in Water by Alpha Spectrometry	QAPP
A-014	Tritium	QAPP
A-015	Gamma Spectrometry	QAPP
A-016	Strontium^{90}	QAPP
A-017	Isotopic Radium^{226} in Water	QAPP
A-018	Volatiles Organic Analysis/EPA Method 8030	QAPP
A-019	Hexavalent Chromium/EPA Method SW7196A	PRS99/100
A-020	Volatiles Organics Analysis/EPA Method 8020	Compendium
A-021	Volatiles Organics Analysis/EPA Method 601	Compendium
A-022	Gasoline Range Organic (GRO)/EPA Method 8015	Compendium
A-023	Total Petroleum Hydrocarbon, Method 418.1	Compendium
A-024	Diesel Range Organic (DRO)/EPA Method 8015	Compendium

ANALYTICAL METHODS

Analytical methods describe the quality control requirements for methods of analysis performed at off-site laboratories. Analytical methods 1 to 18 were extracted from the Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan (QAPP). The extracted methods retain as much of the original text from the QAPP as feasible. However, because many of the methods with similar quality control requirements were discussed within the same paragraph of the QAPP, some text was revised for inclusion in the individual methods. Additionally, the method modifications in Appendix B of the QAPP, and included in each of the CLP methods (A-001, A-003, A-004, and A-005) were revised to only address the changes applicable to the attached method. Each of the methods extracted from the QAPP lists the source document as QAPP and the document date as April 1995.

The methods extracted from the QAPP should be usable for characterizing the extent and degree of contamination of potential release sites which have:

- limited analytical data;
- inconclusive analytical data; or
- no previous sample data.

Where release site data are available, the analyte list for the QAPP approved methods should be appropriately reduced or new methods should be introduced to collect focused and usable analytical data. If the analyte list is reduced, the reduced analyte list should be noted in the appropriate sample plan. If a new method is added, then:

- the method should be added to this Compendium;
- section 1.1 of the method should describe the use of the method; and
- the method should be identified in the sample plan.

When a new method is approved for use with a specific release site, then:

- the Source Document and Document Date on the title page of the method must be updated; and

- both the method and a revised table of contents for the section must distributed to all copy holders. The DOE prime contractor will be responsible for the distribution or assigning the distribution to a subcontractor.

MOUND

Environmental
Restoration
Program

Method: A-010

Explosives Analysis by USEPA Method 8330

Revision 3.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Description

Both soils/sediments and water samples will be analyzed for ten SW8330 explosives and PETN using high performance liquid chromatography (HPLC). Analysis will be performed according to laboratory SOPs which are based on USEPA SW846, Method 8330 (EPA 1990). Second column confirmation will be performed if positive results are obtained on the primary column. PETN will be detected at a different wavelength (220 nm) on a separate analytical run. A statement of work for analysis of PETN is provided in Attachment B describing the required variation from method 8330.

1.2 References

EPA. 1986. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. November 1986.

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Explosives Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Explosives	SW8330	Amber glass bottle with Teflon-lined lid	1 liter	Cool 4°C	7 days extraction/ 40 days analysis
Soil	Explosives	SW8330	125-mL wide-mouth amber glass jar with Teflon-lined lid.	100 grams	Cool 4°C	14 days extraction/ 40 days analysis

3. CALIBRATION

3.1 High Performance Liquid Chromatography (HPLC)

High-performance liquid chromatography (HPLC) is used for analysis of explosives. Initial calibration is performed with a minimum of five concentrations of standards whenever there is change in chromatographic conditions or when the check standard is outside acceptance criteria. The resulting calibration curve must have an average response factor with a relative standard deviation less than or equal to 20%.

The initial calibration is checked prior to sample analysis and once every 10 samples analyzed with a midrange standard for each analyte. The response of the check standard must be within 15% of the predicted response in order for the initial calibration to be valid. If the calibration check is outside this criteria, a new calibration curve will be performed. The retention times and peak heights of the check standard for every 10 samples are compared to those of the check standard run at the beginning of the day. If significant deviation or visible chromatographic abnormalities are observed, then all samples analyzed after the last acceptable standard check will be reanalyzed.

4. QC CRITERIA

Table 4.1 - Explosives Analysis - EPA Method SW8330 Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Explosives	Field Duplicate	1 every 10 or fewer field samples (**water**) 1 every 10 or fewer field samples (**soil**)	≤35% RPD Not Applicable	Evaluate data for usability. Evaluate variability.
	Equipment (rinsate) blank	1 every 10 or fewer field samples (**water**)	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.

Table 4.2 - Explosives Analysis - EPA Method SW8330 Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Explosives	Method blank	1 per 20 samples of given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	<PQL	Reanalyze blank.
	Calibration	(5 pt.) when calibration check limit criteria exceeded.	≤20% RSD	Recalibrate
	Surrogate Spike	All lab and field samples	See Table 4.3	Reanalyze
	Matrix spike (MS)	1 per 20 samples of a given matrix.	See Table 4.3	Evaluate data for usability
	Matrix spike duplicate (MSD)	1 per 20 samples of a given matrix.	See Table 4.3	Evaluate data for usability
	Laboratory control sample (LCS)	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	See Table 4.3	Evaluate data for usability
	Retention time window	With every calibration check	Column and Compound Specific	Identify source, correct problem; reanalyze samples since last good calibration check
	Calibration check	Prior to sample analysis and 1 per 10 samples analyzed.	±16% of peak height of initial calibration	Recalibrate
	Secondary column confirmation	Every positive detection ≥ PQL	Not applicable	Evaluate positive identification of analyte.

Table 4.3 - Explosives Analysis- EPA Method SW8330 Laboratory Surrogate and Matrix Spike Limits

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits Percent Recovery		Advisory Limits Relative Percent Difference (%)	
		Water (µg/L)	*Soil (µg/kg)*	*Water*	*Soil*	*Water*	*Soil*
	Matrix Spike/LCS (low concentration)						
Explosives	RDX	11.6	N/A	62-87	N/A	32	N/A
	1,3,5-TNB	28	N/A	85-100	N/A	19	N/A
	2,4,6-TNT	5.8	N/A	78-102	N/A	29	N/A
	2,6-DNT	1.0	N/A	66-102	N/A	45	N/A
	2,4-DNT	0.8	N/A	74-99	N/A	31	N/A
	PETN	*	*	75-100	40-160	20	30
	Matrix Spike/LCS (high concentration)						
	RDX	140	13	49-71	40-160	20	30
	1,3,5-TNB	29	13	85-108	40-160	20	30
	2,4,6-TNT	5.0	13	83-104	40-160	20	30
	2,6-DNT	4.0	3.0	74-96	40-160	20	30
	2,4-DNT	*	*	77-100	40-160	20	30
	PETN	*	*	75-100	40-160	20	30
	Surrogates						
	4-Nitrotoluene	4.0	25	50-150	50-150	N/A	N/A
	4-Nitroglycerin	*	*	50-150	50-150	N/A	N/A

*Per standard laboratory specification (mid-range response)

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Explosives Analysis Target Analyte List

Analyte	Water (µg/L)	Soil (mg/kg)
HMX	20	3.0
RDX	6.0	2.5
NB	15	1.5
1,3-DNB	15	1.5
1,3,5-TNB	15	1.5
2,4-DNT	0.5	0.5
2,6-DNT	0.5	1.5
TNT	3.0	1.5
2A,4,6-DNT	3.0	1.5
Tetryl	3.0	2.5
PETN	1	1

ATTACHMENT A FOR

METHOD A-010

Attachment A for Method A-010

Statement of Work for Soil Preparation for Common Organic, Inorganic, and Selected Radiological Analyses

1.0 Scope and Application

This procedure describes how to aliquot Mound soil and soil-like samples for laboratory preparation and analysis. This procedure applies to soil analysis for metals, semi-volatiles, pesticide/PCBs, cyanide, anions, explosives, and radiological analyses which do not have a prescribed soil preparation procedure. This procedure should not be used for volatile organic analysis. Soils for volatile organic analysis will be prepared and homogenized as described in the method of analysis.

2.0 Summary

A representative aliquot of a sample is taken in the laboratory by either visually examining and taking a representative portion from each layer in a sample or taking a core of the sample.

3.0 Interferences

Soil samples are heterogeneous by nature. Because of this nature, target analytes are often channeled and concentrated in the soil in specific layers or locations. This heterogeneity may affect both how representative the sample is of the field location and how representative the laboratory aliquot is of the sample.

Heterogeneous nature of soils can sometimes be eliminated in laboratory aliquoting by visually inspecting the sample for layering and selecting a representative aliquot or by taking a core of the sample.

4.0 Equipment

4.1 Spatula or Scoop

4.2 Glass tray, plastic tray, or other material for containing spilled soil

4.3 Large container, i.e. 1000 mL Pyrex beaker

5.0 Reagents/Supplies

5.1 Disposable gloves

6.0 Sample Collection/Holding Time/Preservation

6.1 See Section 2.0 of Method A-010.

7.0 Procedure

7.1 Place a glass tray, plastic tray, or disposable paper beneath the sample container. The tray or paper will be used to contain any soil which accidentally falls off the bottle lip when the cap is opened or falls out while the sample is taken.

7.2 Visually examine the contents of the sample container. If obvious layering is present, then representative portions of each layer must be taken for the aliquot.

If the sample is obviously a core sample (cylindrical soil mass), then use the spatula to core from the top of the sample to the bottom of the sample. This procedure should be representative of the entire core.

If the sample cannot be easily cored, it may be necessary to transfer the sample to a large container and thoroughly and carefully mix the sample with a spatula or scoop. Mixing will not be performed on soil samples for volatile and semi-volatile analyses.

If the sample is neither layered nor a core sample, then use a spatula to core through the middle of the sample. The core should be representative of the entire sample.

7.3 Process the sample as specified in the applicable method.

8.0 Quality Control

8.1 Each analytical method has specific types of quality control samples introduced to evaluate laboratory precision and reproducibility of sample results. Typically, these quality control samples are laboratory duplicates or matrix spike duplicates. These quality control samples permit the laboratory to calculate the relative percent difference and evaluate the soil aliquoting procedure and the precision of the method.

9.0 References and Associated Standard Operating Procedures

None

ATTACHMENT B FOR

METHOD A-010

Attachment B for Method A-010

Statement of Work for
PETN Water Extraction and Analysis
Using EPA Method 8330

1.0 Scope and Application

This procedure describes the required modification to extract and analyze pentaerythritol tetranitrate (PETN) from water by EPA Method 8330, (EPA 1990). This procedure also describes the surrogates required for the analysis of explosives.

2.0 Summary of Method

No change.

3.0 Interferences

No change.

4.0 Apparatus and Equipment

No change.

5.0 Reagents/Supplies

5.2.19 PETN - Reagent Grade
5.2.20 4-Nitrobutene - Reagent Grade
5.4.3 Surrogate Spiking Solution
Prepare a 2000 µg/mL solution of 4-nitrotoluene and 4-nitrobutene in acetonitrile. 50 µL of solution will be added to each sample.

6.0 Sample Collection, Preservation, and Handling

No change.

7.0 Sample Analysis Procedure

7.1 Instrument Conditions

<u>Primary HPLC Column</u>

Column:	LC-18
Mobile Phase:	1:1 Methanol/Water
Flow:	1.5 ml/min.

Detector:	UV 220
Injection Size:	100 µl loop

Confirmation HPLC Column

Column:	LC-CN
Mobile Phase:	1:1 Methanol/Water
Flow:	1.5 ml/min.
Detector:	UV 220
Injection Size:	100 µl loop

8.0 Quality Control

8.7 Two surrogates, 4-nitrotoluene and 4-nitrobutene, are added to each sample.

8.8 A control spike (blank spike) is required for every 20 samples extracted or each batch of samples, whichever is more frequent.

9.0 References

No change.

Statement of Work for PETN Soil Extraction and Analysis Using EPA Method 8330

1.0 Scope and Application

This procedure describes the required modification to extract and analyze pentaerythritol tetranitrate (PETN) from soil and other solid matrices by EPA Method 8330, (EPA 1990). This procedure also describes the surrogates required for the analysis of explosives.

2.0 Summary of Method

No change.

3.0 Interferences

No change.

4.0 Apparatus and Equipment

No change.

5.0 Reagents/Supplies

5.2.19 PETN - Reagent Grade
5.2.20 4-Nitrobutene - Reagent Grade
5.4.3 Surrogate Spiking Solution
Prepare a 2000 µg/mL solution of 4-nitrotoluene and 4-nitrobutene in acetonitrile. 12.5 µL of solution will be added to each sample.

6.0 Sample Collection, Preservation, and Handling

No change.

7.0 Sample Analysis Procedure

7.1 Instrument Conditions

<u>Primary HPLC Column</u>

Column:	LC-18
Mobile Phase:	1:1 Methanol/Water
Flow:	1.5 ml/min.
Detector:	UV 220
Injection Size:	100 µl loop

Confirmation HPLC Column

Column:	LC-CN
Mobile Phase:	1:1 Methanol/Water
Flow:	1.5 ml/min.
Detector:	UV 220
Injection Size:	100 µl loop

8.0 Quality Control

8.7 Two surrogates, 4-nitrotoluene and 4-nitrobutene, are added to each sample.

8.8 A control spike (blank spike) is required for every 20 samples extracted or each batch of samples, whichever is more frequent.

9.0 References

No change.

MOUND

Environmental
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Method: A-020

Volatiles Organic Analysis/ EPA Method 8020

(includes specifications for substituting Method 8240 or 8260)

Revision 3.0

Mound Plant
Miamisburg, OH

Source Document: Compendium (January 1997)

1. INTRODUCTION

1.1 Description

Soil and water samples will be analyzed for aromatic VOCs using gas chromatography with a photoionization detector. The methodology to be followed is EPA Method 8020 (EPA 1987). This method was added to satisfy State of Ohio Buried Underground Storage Tank Requirements. The method is appropriate for verifying compliance to the Ohio BUSTR regulations.

1.2 References

EPA. 1986. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. November 1986.

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

2. PRESERVATION

Volatile Organic Analysis - EPA Method 8020
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Volatile Organic Compounds	SW5030/SW8020	Glass vial with Teflon-lined septum (no headspace)	Two 40 mL vials	HCl to pH<2 Cool 4°C	14 days
Soil	Volatile Organic Compounds	SW5030/SW8020	Glass bottle with Teflon-lined lid (no headspace)	120g bottle	Cool 4°C	14 days

3. CALIBRATION

Gas chromatography will be used for analysis of volatile organic compounds in groundwater (Methods SW-8020). Initial calibration is performed when chromatographic conditions are changed (e.g., change in flow rate, detectors, new column. A minimum of five external standards for volatile organic analysis are analyzed to determine the linearity of the gas chromatograph. Response factors for each compound are calculated (as specified in the methods) from the results, and a calibration curve generated. Linearity criteria for volatile organic compounds (VOCs) are

valid if there is less than or equal to 20% relative standard deviation among the calibration factors. A quadratic curve may also be used.

The linearity of the gas chromatograph for volatile organic analysis is checked by analysis of a check standard after every 10 sample analyses. The response for any analyte must be within a 15% difference of the response from the initial calibration. If the percent difference exceeds this criterion, then the instrument is checked and a new calibration curve is performed before samples are analyzed.

Retention time windows for VOCs are established when a column is changed or after other changes are made in instrument conditions that will alter the retention times of the analytes of interest. The windows are established according to procedures defined in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, USEPA (EPA 1987).

4. QC CRITERIA

Table 4.1 - Volatile Organic Analysis - EPA Method 8020 Field QC Sample Frequency

Parameters	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOC, SW8020	Trip Blank	1 per shipping container to lab	≤ 0.10 x level in associated samples, or ≤ PQL	Evaluate potential sources; Evaluate associated data for usability.
	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤ 0.10 x level in associated samples, or ≤ PQL	Evaluate potential sources; Evaluate associated data for usability.
	Field Duplicate	1 every 10 or fewer field samples (water)	≤ 35% RPD	Evaluate data for usability.
		1 every 10 or fewer field samples (soil)	≤ 50% RPD	

Table 4.2 - Volatile Organic Analysis - EPA Method 8020 Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOC, SW8020	Method Blank	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	≤ PQL	Identify and correct source. Reanalyze blank and associated samples.
	Calibration	5 points; when calibration check criteria exceeded.	≤ 20% RSD for calibration factors	Recalibrate
	Calibration check	Once per 10 samples analyzed.	± 15% from initial response factor	Recalibrate
	Matrix spike	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Matrix spike duplicate	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Surrogate spikes	All field and lab samples	See Table 4.3	Check calculations, surrogate and standard solutions, and instrument. If problem not identified then reanalyze sample.
	Retention time window	When new column installed and as needed	±3σ of three retention times for each analyte as per SW 846.	Identify source, correct problem.
	Laboratory control sample (LCS)	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	See Table 4.3	Identify and correct problem prior to further sample analyses, reanalyze.

Table 4.3 - Volatile Organic Analysis - EPA Method 8020 Laboratory Surrogate and Matrix Spike Limits

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits			
				Percent Recovery		Relative Percent Difference (%)	
		Water (µg/L)	*Soil (µg/kg)*	*Water*	*Soil*	*Water*	*Soil*
	Matrix Spike/LCS						
Volatile Organic Compounds, SW8020	Benzene	*	*	80-120	80-120	≤15	≤30
	Toluene	*	*	80-120	80-120	≤15	≤30
	Ethyl Benzene	*	*	80-120	80-120	≤15	≤30
	Xylenes	*	*	80-120	80-120	≤15	≤30
	Surrogates						
	Fluorobenzene	30	30	80-120	80-120	≤15	≤30

* Per standard laboratory specification (mid-range response).

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 - Volatile Organic Analysis - EPA Method 8020 Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
Benzene	2.0	NA
Ethylbenzene	2.0	NA
Toluene	2.0	NA
Xylene	2.0	NA

6. ALTERNATIVE METHOD REQUIREMENTS FOR SW8240/SW8260

There are instances where a contracted laboratory may request permission to substitute Method 8240 or 8260 for Method 8020. These methods (8240 and 8260) are GC/MS methods of analysis and are acceptable alternatives for method 8020. The calibration and QC requirements for these methods are presented in section 6.1 and 6.2. The reporting limit is no different.

6.1 GC/MS Calibration

Gas Chromatograph/Mass Spectrometry (GC/MS) will be used for analysis of volatile organic compounds. Mass spectral abundance criteria must be met prior to sample analysis. Bromofluorobenzene (BFB) is used to verify instrument performance of the GC/MS system and must meet specific ion abundance criteria established in the CLP SOW. Meeting these criteria is demonstrated daily or once during every 12-hour time period, whichever is more frequent. The instrument performance is also verified whenever a corrective action to the GC/MS system is taken that affects the tuning (e.g., ion source cleaning or repair).

Initial calibration of the GC/MS system is accomplished with a minimum of five concentrations of target compounds. Relative Response Factors (RRFs) must be greater than or equal to 0.05. Relative standard deviations for the RRFs must be less than or equal to 30%. The relative retention times of each compound in each standard run must agree within 0.06 units. Initial calibration is not valid if this criteria are not met.

The initial calibration is verified every 12-hour period with a continuing calibration standard containing all target volatile compounds and surrogate compounds. RRFs are compared to the average RRF from the initial calibration. The minimum RRF for the target compounds must be met. The percent difference between the initial RRFs and the continuing RRF must be less than or equal to 25 percent for the initial calibration to be valid. Prior to sample analysis, the GC/MS system is evaluated and corrective action taken if these criteria are not met.

6.2 GC/MS QC Criteria

Table 6.2.1- Volatile Organic Analysis - EPA Method SW8240/SW8260 Field QC Sample Frequency

Parameters	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOC, SW8240/ SW8260	Trip Blank	1 per shipping container to lab	≤ 0.10 x level in associated samples, or ≤ PQL	Evaluate potential sources; Evaluate associated data for usability.
	Equipment (rinsate) blank	1 every 10 or fewer field samples (**water**)	≤ 0.10 x level in associated samples, or ≤ PQL	Evaluate potential sources; Evaluate associated data for usability.
	Field Duplicate	1 every 10 or fewer field samples (**water**)	≤ 35% RPD	Evaluate data for usability.
		1 every 10 or fewer field samples (**soil**)	≤ 50% RPD	

Table 6.2.2 - Volatile Organic Analysis SW8240/SW8260 Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOC, SW8240/ SW8260	Method Blank	Once per 12-hour period	≤ 5 x CRQL of common lab contaminants ≤ CRQL others	Investigate source; reanalyze associated samples.
	Matrix Spike	1 per 20 samples of a given matrix in a case or fewer	See Table 6.2.3	Evaluate data for usability.
	Matrix spike duplicate	1 per 20 samples of a given matrix in a case or fewer	See Table 6.2.3	Evaluate data for usability.
	Laboratory control sample	Once per 12-hour period	See Table 6.2.3	Evaluate associated data for usability.
	System monitoring compounds	All lab and field samples	SW846 use benzene, toluene, ethyl benzene, and xylene	See SW846.
	Continuing Calibration check	Daily or each 12-hour period, whichever is more frequent	SW846, use benzene toluene, ethyl benzene, and xylene	Retune: Reanalyze associated samples
	Calibration	See SW846	See SW846	Recalibrate before sample analysis
	Retention time window	See SW846	SW846	See SW846.
	Qualitative verification	When a detection occurs in a sample	SW846	See SW846.
	Internal standard	Every standard and sample	SW846	See SW846.

Table 6.2.3 - Volatile Organic Analysis - EPA Method SW8240/SW8260 Laboratory Surrogate and Matrix Spike Limits

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits			
				Percent Recovery		Relative Percent Difference (%)	
		Water (µg/L)	*Soil (µg/kg)*	*Water*	*Soil*	*Water*	*Soil*
	Matrix Spike/LCS						
VOC, SW8240/ SW8260	Benzene	*	*	80-120	80-120	≤15	≤30
	Toluene	*	*	80-120	80-120	≤15	≤30
	Ethyl Benzene	*	*	80-120	80-120	≤15	≤30
	Xylenes	*	*	80-120	80-120	≤15	≤30
	Surrogates						
	Toluene - d8	*	*	88-110	84-138	NA	NA
	4 - bromofluorobenzene	*	*	86-115	59-113	NA	NA
	1,2 - dichloroethane	*	*	76-114	70-121	NA	NA

* Per standard laboratory specification (mid-range response).

MOUND

Environmental
Restoration
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Method: A-021

Volatiles Organic Analysis (VOA)/ EPA Method 601

Revision 3.0

Mound Plant
Miamisburg, OH

Source Document: Compendium (January 1997)

1. INTRODUCTION

1.1 Description

Method 601 describes the gas chromatography analysis of water for benzene, ethylbenzene, and toluene. An additional analyte, total xylenes, is required by the Ohio Bureau of Underground Storage Tank Regulations (BUSTR). BUSTR is implemented by the Ohio State Fire Marshall for the evaluation of underground storage tanks. Method 601 is specifically used for evaluating water associated storage tanks which contain or contained gasoline or other middle petroleum distillates (kerosene, diesel fuel, light oil, etc.).

1.2 References

EPA 1983. "Methods for Chemical Analysis of Water and Wastes", U.S. Environmental Protection Agency, EPA-600/4-79-020, March 1983.

2. PRESERVATION

Volatile Organic Analysis - EPA Method 601
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation[1]	Holding Time
Water	Volatile Organic Compounds	EPA Method 601	Glass vial with Teflon-lined septum (no headspace)	Three 40 mL vials	HCl to pH<2 Cool 4°C	14 days

Regulation requires 500 mL bottle, but there is better sample integrity using 40 mL vials.

3. CALIBRATION

Gas chromatography will be used for analysis of volatile organic compounds in water (Method 601). Initial calibration is performed when chromatographic conditions are changed (e.g., change in flow rate, detectors, new column. A minimum of three external standards for volatile organic analysis are analyzed to determine the linearity of the gas chromatograph. Response factors for each compound are calculated (as specified in the methods) from the results, and a calibration curve generated. Linearity criteria for volatile organic compounds (VOCs) are valid if there is less than or equal to 10% relative standard deviation among the calibration factors.

The linearity of the gas chromatograph for volatile organic analysis is checked by analysis of a check standard each day and after every 10 sample analyses. The response for any analyte must be within a 10% difference of the response from the initial calibration. If the percent difference exceeds this criterion, then the instrument is checked and a new calibration curve is performed before samples are analyzed.
Retention time windows for VOCs are established when a column is changed or after other changes are made in instrument conditions that will alter the retention times of the analytes of

interest. The windows are established according to procedures defined in "Methods of Chemical Analysis of Water and Wastes".

4. QC CRITERIA

Table 4.1 - Volatile Organic Analysis - EPA Method 601 Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOC, EPA Method 601	Trip Blank	1 per shipping container to lab	≤ 0.10 x level in associated samples, or <PQL	Evaluate potential sources; Evaluate associated data for usability.
	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤ 0.10 x level in associated samples, or <PQL	Evaluate potential sources; Evaluate associated data for usability.
	Field Duplicate	1 every 10 or fewer field samples (water)	≤ 25% RPD	Evaluate data for usability.

Table 4.2 - Volatile Organic Analysis - EPA Method 601 Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOC, EPA Method 601	Method Blank	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	≤ PQL	Identify and correct source. Reanalyze blank and associated samples.
	Calibration	3 points; when calibration check criteria exceeded.	≤ 10% RSD for calibration factors	Recalibrate
	Calibration check	Once per 10 samples analyzed.	± 10% from initial response factor	Recalibrate
	Matrix spike	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Matrix spike duplicate	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Surrogate spikes	All field and lab samples	See Table 4.3	Check calculations, surrogate and standard solutions, and instrument. If problem not identified then reanalyze sample.
	Retention time window	When new column installed and as needed	±3 x SD of three retention times for each analyte as per EPA Methods.	Identify source, correct problem.
	Laboratory control sample (LCS)	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	See Table 4.3	Identify and correct problem prior to further sample analyses, reanalyze.

Table 4.3 - Volatile Organic Analysis - EPA Method 601 Laboratory Surrogate and Matrix Spike Limits

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits			
				Percent Recovery		Relative Percent Difference (%)	
		Water (µg/L)	*Soil (µg/kg)*	*Water*	*Soil*	*Water*	*Soil*
Volatile Organic Compounds, EPA Method 601	*Matrix Spike/LCS*						
	Benzene	*	*	70-130	NA	≤25	NA
	Ethylbenzene	*	*	70-130	NA	≤25	NA
	Toluene	*	*	70-130	NA	≤25	NA
	Xylenes	*	*	70-130	NA	≤25	NA
	Surrogates						
	Fluorobenzene	*	*	70-130	NA	NA	NA

* Per standard laboratory specification (mid-range response).

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 - Volatile Organic Analysis - EPA Method 601 Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
Benzene	1.0	NA
Ethylbenzene	1.0	NA
Toluene	1.0	NA
Xylene (Total)	1.0	NA

MOUND

Environmental
Restoration
Program

Method: A-022

Gasoline Range Organic (GRO)/ EPA Method 8015

Revision 3.0

Mound Plant
Miamisburg, OH

Source Document: Compendium (January 1997)

1. INTRODUCTION

1.1 Description

Water and soil samples will be analyzed for gasoline range organic (GRO) analysis by a gas chromatograph (GC) with flame ionization detector. This specific procedure detects GRO compounds. Method A-024 describes the analysis of Diesel Range Organic (DRO) compounds. The sample aliquot will be introduced into the GC using a purge and trap procedure (SW5030). Soil samples will be heated during purging.

This method is used for the evaluation of the underground gasoline storage tanks as required by the State of Ohio Fire Marshall in the Bureau of Underground Storage Tank Regulations (BUSTR).

1.2 References

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1987. Data Quality Objectives for Remedial Response Activities. U.S. Environmental Protection Agency. Office of Emergency and remedial Response and Office of Waste Programs Enforcement. Report No. EPA 540/g-87/003. Washington, D.C. March 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

2. PRESERVATION

Gasoline Range Organic Analysis - EPA Method 8015
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	GRO	SW5030/SW8015	Glass vial with Teflon-lined septum (no headspace)	Two 40 mL vials	HCl to pH<2 Cool 4°C	14 days
Soil	GRO	SW5030/SW8015	Glass bottle with vial with Teflon-lined lid	120g	Cool 4°C	14 days

3. CALIBRATION

Gas chromatography will be used for analysis of GRO (Methods SW-8015B). Initial calibration is performed when chromatographic conditions are changed (e.g., change in flow rate, detectors, new column. A minimum of five external standards for GRO analysis are analyzed to determine the linearity of the gas chromatograph. Response factors for each compound are calculated (as

specified in the methods) from the results, and a calibration curve generated. Linearity criteria for GRO are valid if there is less than or equal to 20% relative standard deviation among the calibration factors. A quadratic curve may also be used.

The linearity of the gas chromatograph for GRO is checked by analysis of a check standard after every 10 sample analyses. The response for any analyte must be within a 15% difference of the response from the initial calibration. If the percent difference exceeds this criterion, then the instrument is checked and a new calibration curve is performed before samples are analyzed.

Retention time window for GRO is established when a column is changed or after other changes are made in instrument conditions that will alter the retention times of the analytes of interest. The windows are established according to procedures defined in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, USEPA (EPA 1987).

4. QC CRITERIA

Table 4.1 - Gasoline Range Organic Analysis - EPA Method 8015 Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
GRO, SW8015	Trip Blank	1 per shipping container to lab	≤ 0.10 x level in associated samples, or <PQL	Evaluate potential sources; Evaluate associated data for usability.
	Equipment (rinsate) blank	1 every 10 or fewer field samples (**water**)	≤ 0.10 x level in associated samples, or <PQL	Evaluate potential sources; Evaluate associated data for usability.
	Field Duplicate	1 every 10 or fewer field samples (**water**)	≤ 35% RPD	Evaluate data for usability.
		1 every 10 or fewer field samples (**soil**)	≤ 50% RPD	Evaluate data for usability.

Table 4.2 - Gasoline Range Organic Analysis - EPA Method 8015 Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
GRO, SW8015	Method Blank	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	≤ PQL	Identify and correct source. Reanalyze blank and associated samples.
	Calibration	5 points; when calibration check criteria exceeded.	≤ 20% RSD for calibration factors	Recalibrate
	Calibration check	Once per 10 samples analyzed.	± 15% from initial response factor	Recalibrate
	Matrix spike	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Matrix spike duplicate	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Surrogate spikes	All field and lab samples	See Table 4.3	Check calculations, surrogate and standard solutions, and instrument. If problem not identified then reanalyze sample.
	Retention time window	When new column installed and as needed	±3σ of three retention times for each analyte as per SW 846.	Identify source, correct problem.
	Laboratory control sample (LCS)	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	See Table 4.3	Identify and correct problem prior to further sample analyses, reanalyze.

Table 4.3 - Gasoline Range Organic Analysis - EPA Method 8015 Laboratory Surrogate and Matrix Spike Limits

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits			
				Percent Recovery		Relative Percent Difference (%)	
		Water (µg/L)	*Soil (µg/kg)*	*Water*	*Soil*	*Water*	*Soil*
GRO, SW8015B	*Matrix Spike/LCS*						
	Gasoline	*	*	60-140	50-150	≤20	≤30
	Surrogates						
	Fluorobenzene	30	30	60-140	50-150	≤20	NA

* Per standard laboratory specification (mid-range response).

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 - Gasoline Range Organic Analysis - EPA Method 8015 Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
Gasoline Range Organics	40	40

MOUND

Environmental
Restoration
Program

Method: A-024

Diesel Range Organic (DRO)/ EPA Method 8015

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: Compendium (January 1997)

1. INTRODUCTION

1.1 Description

Water and soil samples will be analyzed for diesel range organic (DRO) analytes by a gas chromatograph (GC) with flame ionization detector. This specific procedure detects DRO compounds. Method A-022 describes the analysis of gasoline range organic (GRO) compounds. The sample aliquot will be introduced into the GC using a purge and trap procedure (SW5030). Soil samples will be heated during purging.

This method is used for the evaluation of the underground gasoline storage tanks as required by the State of Ohio Fire Marshall in the Bureau of Underground Storage Tank Regulations (BUSTR).

1.2 References

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1987. Data Quality Objectives for Remedial Response Activities. U.S. Environmental Protection Agency. Office of Emergency and remedial Response and Office of Waste Programs Enforcement. Report No. EPA 540/g-87/003. Washington, D.C. March 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

2. PRESERVATION

Diesel Range Organic Analysis - EPA Method 8015
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	DRO	SW5030/SW8015	Glass vial with Teflon-lined septum (no headspace)	Two 40 mL vials	HCl to pH<2 Cool 4°C	14 days
Soil	DRO	SW5030/SW8015	Glass bottle with vial with Teflon-lined lid	120g	Cool 4°C	14 days

3. CALIBRATION

Gas chromatography will be used for analysis of DRO compounds (Methods SW-8015B). Initial calibration is performed when chromatographic conditions are changed (e.g., change in flow rate, detectors, new column. A minimum of five external standards for DRO analysis are analyzed to determine the linearity of the gas chromatograph. Response factors for each compound are

calculated (as specified in the methods) from the results, and a calibration curve generated. Linearity criteria for DRO are valid if there is less than or equal to 20% relative standard deviation among the calibration factors. A quadratic curve may also be used.

The linearity of the gas chromatograph for DRO analysis is checked by analysis of a check standard after every 10 sample analyses. The response for any analyte must be within a 15% difference of the response from the initial calibration. If the percent difference exceeds this criterion, then the instrument is checked and a new calibration curve is performed before samples are analyzed.

Retention time window for DRO is established when a column is changed or after other changes are made in instrument conditions that will alter the retention times of the analytes of interest. The windows are established according to procedures defined in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, USEPA (EPA 1987).

4. QC CRITERIA

Table 4.1 - Diesel Range Organic Analysis - EPA Method 8015 Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
DRO, SW8015	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤ 0.10 x level in associated samples, or <PQL	Evaluate potential sources; Evaluate associated data for usability.
	Field Duplicate	1 every 10 or fewer field samples (water)	≤ 35% RPD	Evaluate data for usability.
		1 every 10 or fewer field samples (soil)	≤ 50% RPD	Evaluate data for usability.

Table 4.2 - Diesel Range Organic Analysis - EPA Method 8015 Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
DRO, SW8015	Method Blank	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	≤ PQL	Identify and correct source. Reanalyze blank and associated samples.
	Calibration	5 points; when calibration check criteria exceeded.	≤ 20% RSD for calibration factors	Recalibrate
	Calibration check	Once per 10 samples analyzed.	± 15% from initial response factor	Recalibrate
	Matrix spike	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Matrix spike duplicate	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Surrogate spikes	All field and lab samples	See Table 4.3	Check calculations, surrogate and standard solutions, and instrument. If problem not identified then reanalyze sample.
	Retention time window	When new column installed and as needed	±3σ of three retention times for each analyte as per SW 846.	Identify source, correct problem.
	Laboratory control sample (LCS)	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	See Table 4.3	Identify and correct problem prior to further sample analyses, reanalyze.

Table 4.3 - Diesel Range Organic Analysis - EPA Method 8015 Laboratory Surrogate and Matrix Spike Limits

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits			
				Percent Recovery		Relative Percent Difference (%)	
		Water (μg/L)	*Soil (μg/kg)*	*Water*	*Soil*	*Water*	*Soil*
DRO, SW8015	*Matrix Spike/LCS*						
	Diesel Fuel	*	*	50-150	50-150	≤20	≤30
	Surrogates						
	p-terphenyl	*	*	50-150	50-150	NA	NA

* Per standard laboratory specification (mid-range response).

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 - Diesel Range Organic Analysis - EPA Method 8015 Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
Diesel Range Organics	100	4000

MOUND

Environmental
Restoration
Program

Method: Q-012

Electronic Data Deliverable Format Specification - MEIMS Non-CLP Standard Format

Revision 3.0

Mound Plant
Miamisburg, OH

Source Document: Compendium (November 1996)

1. INTRODUCTION

This procedure contains the specifications for the standard Mound Environmental Information Management System (MEIMS) non-CLP electronic data deliverable (EDD) format. Quality Method Q-013 describes an alternate deliverable format which can be used for non-CLP analyses. The alternative specification should only be used if the laboratory is unable to meet the specifications in this procedure.

2. RESPONSIBILITIES

Laboratory Reporting Staff — The reporting staff is responsible for accurately transferring the laboratory data to 3.5 inch floppy diskettes in the format specified in this procedure.

Database Clerk — The database clerk is responsible for loading the EDD into MEIMS and reporting entry errors to the database administrator.

Database Administrator — The database administrator is responsible for maintaining the format specification in this procedure and resolving laboratory EDD problems. The database administrator may designate an individual to resolve laboratory EDD problems.

3. PROCEDURE

The EDDs for the MEIMS are prepared at the laboratory and stored on 3.5 inch floppy disks. EDDs for non-CLP data shall follow a specified format. The following sections describe the format for each data record including the fields and allowable codes for each field.

Section 3.1 lists each field, the position of the field, and the field length. The section also identifies the source of the field information, shows an example for each field, and identifies whether the field is required.

3.1. Non-CLP Record Format

Field Description	Position	Length	Source	Example	Required**
Header Record*					
Project Number	1-20	20	CONTRACT	E123A1156	Opt
Submission Date	21-28	8	LAB	12/30/93	Opt
Number of records	29-33	6	LAB	1235	Opt
Detail Record					
Client Sample ID	1-20	20	COC	900123	Mand
Date Collected	21-28	8	COC	11/23/93	Mand***
Time Collected	29-33	5	COC	1300	Opt
Lab Batch/SDG Number	34-48	15	LAB	50071	Mand
Matrix	49-56	8	COC	WATER	Mand
Lab Sample ID	57-76	20	LAB	CC113091	Mand
Lab Code	77-81	5	VALID CODE LIST	COMPUC	Mand
Date Extracted/Prepared	82-89	8	LAB	12/10/93	Opt
Date Analyzed	90-97	8	LAB	12/12/93	Mand

Field Description	Position	Length	Source	Example	Required**
Time Analyzed	98-102	5	LAB	1200	Mand
Lab Blank Sample Number	103-122	20	LAB	CC113124	Mand
Analysis Type Code	123-132	10	VALID CODE LIST	ORVOA	Opt
Result Type Code	133-135	3	VALID CODE LIST	REG	Mand
Parameter Code	136-146	11	VALID CODE LIST	100-41-4	Mand
Result	147-156	10	LAB	10.0	Mand
Result Qualifier Code	157-161	5	VALID CODE LIST	U	Mand
Uncertainty	162-171	10	LAB	0.0025	Opt
Unit of Measure Code	172-179	8	VALID CODE LIST	UG/L	Mand
Retention Time (TICs only)	180-186	7	LAB	2330	Opt
Analyte Name	187-216	30	VALID CODE LIST	Endrin	Mand
Detection Limit	217-226	10	LAB	10.0	Mand
Method ⅄	227-236	10	LAB	SW8020	Mand
Percent Solids	237-241	5	LAB	82.3	Opt
Sample Weight/Volume	242-246	5	LAB	5.0	Mand
Weight/Volume Units	247-248	2	LAB	G	Mand
Dilution	249-253	5	LAB	1.0	Mand

* If the header is not used, leave the first line blank

** Opt = Optional, Mand = Mandatory. The optional fields may be lab/matrix/method dependent. The fields become required when the associated lab/matrix/method dictates.

*** The data collected must have a date entered, or if the date is unknown, the date format must be entered i.e. " / / ".

⅄ % solids method should be 'UNKNOWN.'

CONTRACT — indicates the data is found on the laboratory subcontract.

LAB — indicates the information is provided by the laboratory.

COC — indicates that the data are found on the Chain-of-Custody form.

VALID CODE LIST — indicates that only codes listed in the valid codes list are to be used. In case where a code definition does not meet the needs of the data recorded, the data administrator will be contacted so that concurrence can be obtained in establishing required coding conventions. ASCII data files that contain all valid code lists will be provided to all participating labs. See Section 3.3 for the valid code lists.

The following conventions must be used:

- The code LCS must be used as the sample ID for blank spikes — spikes that do not involve true environmental samples from Mound. A sample type of LCS must be used for blank spikes.

- The code BLK must be used as the sample ID for method blanks. A sample type of BLK must also be used.

- The code MS must be appended to the end of the sample ID for matrix spike and surrogate results, and the sample type must be specified as SPK. Further, these results must follow the sample analytical results in the EDD. For example, if the surrogate recovery for BTEX analysis for sample 10 is entered in EDD record 10 and the analytical BTEX results are in EDD records 11, 12, 13 and 14, the data will not load. Table 3.3 lists the valid suffixes for the client sample ids. No characters should be entered between the client sample id and the special results suffix.

3.2. Field Definitions

Field Description	Field Definition
Header Record	
Project Number	Laboratory subcontract number for this project.
Submission Date	Date the data were submitted.
Number of records	Number of detail records in this file.
Detail Record	
Client Sample ID	Sample identifier assigned by the client. Append the suffix for special results - see Table 3.3.1.
Date Collected	Date sample was collected.
Time Collected	Time sample was collected.
Lab Case Number	Laboratory case number for this data submittal.
Lab Batch/SDG Number	Laboratory sample delivery group number or batch number.
Matrix	Sample matrix as specified on the Chain-of-Custody.
Lab Sample ID	Sample identifier assigned by the laboratory.
Lab Code	Code identifying the laboratory. See Table 3.3.2.
Date Extracted/Prepared	Date the sample was extracted.
Date Analyzed	Date the sample was analyzed.
Time Analyzed	Time the sample was analyzed.
Lab Blank Sample Number	Sample identifier for the laboratory blank associated to this sample.
Analysis Type Code	Code identifying the type of analysis performed on the sample. See Table 3.3.3.
Result Type Code	Code identifying the type of laboratory result. See Table 3.3.4.
Parameter Code	Code identifying the parameter. See Table 3.3.5.
Result	Result of the parameter for the sample.
Result Qualifier	Laboratory qualifier for the result.
Uncertainty	2 sigma error for radiological results.
Unit of Measure	Code identifying the unit of measure. See Table 3.3.6.
Retention Time (TICs only)	Retention time for tentatively identified compounds.
Detection Limit	Detection limit associated with the parameter results.
Method	Analytic method of analysis. Lab SOP if not standard method. See Table 3.3.7.
Percent Solids	The solid fraction of a non-aqueous sample. Blank if aqueous.
Sample Weight/Volume	The weight or volume of the sample.
Weight/Volume Units	Unit of measure of the sample weight or volume.
Dilution	Dilution factor associated with the parameter results.

3.3. Valid Code Lists

3.3.1. Special Results

Code	Description
MS	Matrix Spike/Matrix Spike Duplicate
MS	Surrogate Results
DL	Dilutions
RE	Re-analysis

3.3.2. Laboratories

Code	Description
DATAC	Data Chem Laboratory
WESTO	Weston Laboratory
QTESR	Quanterra Environmental Services - Richland
QUANT	Quanterra Environmental Services - Denver

3.3.3. Analysis Types

Code	Description
ANION	Common Anions
EPTOX	EP TOX Leachate
GENERA	General Chemistry
GEOTEC	Geotechnical
INORG	Metals
OILGRS	Oil and Grease
ORBTEX	BTEX Compounds
ORDIOX	Dioxins/Dibenzofurans
ORDRO	Diesel Range Organics
OREXP	Explosives
ORGRO	Gasoline Range Organics
ORHERB	Herbicides
ORMORO	Motor Oil Range Organics
ORMRO	Motor Oil Range Organics
ORPETH	Petroleum Hydrocarbons
ORPHNL	Phenols
ORPPB	Pesticides and/or PCBs
ORSVO	Semi-Volatile Organics
ORVOA	Volatile Organics
OTHER	Other
RAD	Radiological
TCLPHB	TCLP Herbicides
TCLPIN	TCLP Metals
TCLPPP	TCLP PCBs
TCLPR	TCLP Reactivity, Corrosivity
TCLPSV	TCLP Semi-Volatiles
TCLPVO	TCLP Volatiles

3.3.4. Result Types

Code	Description
BLK	Method Blank
DIL	Dilution
DUP	Analytical Duplicate
LCS	Lab Control Sample / Blank Spikes
PB	Prep blank
R1	Replicate Sample
REA	Reanalyzed Sample
REG	Regular Sample or Reported Value if Reanalyzed
SPK	Matrix Spike / Matrix Spike Duplicate
TIC	Tentatively Identified Compound

3.3.5. Parameter Codes

Code	Parameter name
100	% Clay
54100	% Gravel
8000	% Sand
8400	% Silt
630-20-6	1,1,1,2-Tetrachloroethane
71-55-6	1,1,1-Trichloroethane
79-34-5	1,1,2,2-Tetrachloroethane
76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane
79-00-5	1,1,2-Trichloroethane
75-34-3	1,1-Dichloroethane
75-35-4	1,1-Dichloroethene
563-58-6	1,1-Dichloropropene
35822-46-9	1,2,3,4,6,7,8-HpCDD
67562-39-4	1,2,3,4,6,7,8-HpCDF
55673-89-7	1,2,3,4,7,8,9-HpCDF
39227-28-6	1,2,3,4,7,8-HxCDD
70648-26-9	1,2,3,4,7,8-HxCDF
57653-85-7	1,2,3,6,7,8-HxCDD
57117-44-9	1,2,3,6,7,8-HxCDF
19408-74-3	1,2,3,7,8,9-HxCDD
72918-21-9	1,2,3,7,8,9-HxCDF
40321-76-4	1,2,3,7,8-PeCDD
57117-41-6	1,2,3,7,8-PeCDF
87-61-6	1,2,3-Trichlorobenzene
96-18-4	1,2,3-Trichloropropane
120-82-1	1,2,4-Trichlorobenzene
95-63-6	1,2,4-Trimethylbenzene
156-59-2	1,2-cis-Dichloroethene
96-12-8	1,2-Dibromo-3-chloropropane
106-93-4	1,2-Dibromoethane
95-50-1	1,2-Dichlorobenzene
2199-69-1	1,2-Dichlorobenzene-d4
107-06-2	1,2-Dichloroethane
17060-07-0	1,2-Dichloroethane-d4

Code	Parameter name
540-59-0	1,2-Dichloroethene
78-87-5	1,2-Dichloropropane
135-01-3	1,2-Diethylbenzene
156-60-5	1,2-trans-Dichloroethene
108-67-8	1,3,5-Trimethylbenzene
99-35-4	1,3,5-Trinitrobenzene
10061-01-5	1,3-cis-Dichloropropene
541-73-1	1,3-Dichlorobenzene
142-28-9	1,3-Dichloropropane
141-93-5	1,3-Diethylbenzene
99-65-0	1,3-Dinitrobenzene
10061-02-6	1,3-trans-Dichloropropene
106-46-7	1,4-Dichlorobenzene
105-05-5	1,4-Diethylbenzene
544-10-5	1-Chlorohexane
108-60-1	2,2'-oxybis(1-chloropropane)
594-20-7	2,2-Dichloropropane
60851-34-5	2,3,4,6,7,8-HxCDF
57117-31-4	2,3,4,7,8-PeCDF
1746-01-6	2,3,7,8-TCDD
51207-31-9	2,3,7,8-TCDF
95-95-4	2,4,5-Trichlorophenol
118-79-6	2,4,6-Tribromophenol
88-06-2	2,4,6-Trichlorophenol
118-96-7	2,4,6-Trinitrotoluene
120-83-2	2,4-Dichlorophenol
105-67-9	2,4-Dimethylphenol
51-28-5	2,4-Dinitrophenol
121-14-2	2,4-Dinitrotoluene
606-20-2	2,6-Dinitrotoluene
35572-78-2	2-Amino-4,6-dinitrotoluene
120-32-1	2-Benzyl-4-Chlorophenol
78-93-3	2-Butanone
110-75-8	2-Chloroethylvinylether
91-58-7	2-Chloronaphthalene
95-57-8	2-Chlorophenol
93951-73-6	2-Chlorophenol-d4
95-49-8	2-Chlorotoluene
321-60-8	2-Fluorobiphenyl
367-12-4	2-Fluorophenol
591-78-6	2-Hexanone
91-57-6	2-Methylnaphthalene
95-48-7	2-Methylphenol
88-74-4	2-Nitroaniline
88-75-5	2-Nitrophenol
88-72-2	2-Nitrotoluene
91-94-1	3,3'-Dichlorobenzidine
618-87-1	3,5-Dinitroaniline
99-09-2	3-Nitroaniline
99-08-1	3-Nitrotoluene
72-54-8	4,4'-DDD
72-55-9	4,4'-DDE

Code	Parameter name
50-29-3	4,4'-DDT
534-52-1	4,6-Dinitro-o-Cresol
1946-51-0	4-Amino-2,6-Dinitrotoluene
101-55-3	4-Bromophenyl-phenyl Ether
59-50-7	4-chloro-3-methylphenol
106-47-8	4-Chloroaniline
7005-72-3	4-Chlorophenyl-phenylether
106-43-4	4-Chlorotoluene
108-10-1	4-Methyl-2-pentanone
106-44-5	4-Methylphenol
100-01-6	4-Nitroaniline
100-02-7	4-Nitrophenol
99-99-0	4-Nitrotoluene
83-32-9	Acenaphthene
208-96-8	Acenaphthylene
67-64-1	Acetone
75-05-8	Acetonitrile
107-13-1	Acrylonitrile
AC-227	Actinium-227
14331-83-0	Actinium-228
309-00-2	Aldrin
ALK	Alkalinity
ALHCO3	Alkalinity: HCO3
5103-71-9	Alpha Chlordane
ALPHA	Alpha, Total
319-84-6	Alpha-BHC
7429-90-5	Aluminum
14596-10-2	Americium-241
AMM	Ammonia
120-12-7	Anthracene
7440-36-0	Antimony
SB-124	Antimony-124
SB-125	Antimony-125
12674-11-2	Aroclor-1016
11104-28-2	Aroclor-1221
11141-16-5	Aroclor-1232
53469-21-9	Aroclor-1242
12672-29-6	Aroclor-1248
11097-69-1	Aroclor-1254
11096-82-5	Aroclor-1260
7440-38-2	Arsenic
7440-39-3	Barium
BA-133	Barium-133
BA-140	Barium-140
71-43-2	Benzene
92-87-5	Benzidine
56-55-3	Benzo(a)anthracene
50-32-8	Benzo(a)pyrene
205-99-2	Benzo(b)fluoranthene
191-24-2	Benzo(g,h,i)perylene
207-08-9	Benzo(k)fluoranthene
65-85-0	Benzoic Acid

Code	Parameter name
100-51-6	Benzyl Alcohol
7440-41-7	Beryllium
13966-02-4	Beryllium-7
BETA	Beta, Total
319-85-7	Beta-BHC
BOD	Bio Oxygen Demand
111-91-1	Bis(2-chloroethoxy)methane
111-44-4	Bis(2-chloroethyl)ether
117-81-7	Bis(2-ethylhexyl)phthalate
7440-69-9	Bismuth
BI-207	Bismuth-207
BI-210	Bismuth-210
BI-210M	Bismuth-210M
BI-211	Bismuth-211
14913-49-6	Bismuth-212
14733-03-0	Bismuth-214
7440-42-8	Boron
108-86-1	Bromobenzene
74-97-5	Bromochloromethane
75-27-4	Bromodichloromethane
460-00-4	Bromofluorobenzene
75-25-2	Bromoform
74-83-9	Bromomethane
85-68-7	Butyl Benzyl Phthalate
C4C8	C4-C8 Cycloalkanes/Alkenes
7440-43-9	Cadmium
7440-70-2	Calcium
86-74-8	Carbazole
75-15-0	Carbon Disulfide
56-23-5	Carbon Tetrachloride
153861900	Cation Exchange
CEC	Cation Exchange Capacity as Na
CERADA	Ceridaphnia
7440-45-1	Cerium
CE-139	Cerium-139
CE-141	Cerium-141
CE-144	Cerium-144
13967-70-9	Cesium-134
10045-97-3	Cesium-137
COD	Chemical Oxygen Demand
57-74-9	Chlordane
CL	Chloride
7782-50-5	Chlorine
108-90-7	Chlorobenzene
75-00-3	Chloroethane
67-66-3	Chloroform
74-87-3	Chloromethane
25168-05-2	Chlorotoluene
7440-47-3	Chromium
CR6	Chromium, Hexavalent
CR-51	Chromium-51
218-01-9	Chrysene

Code	Parameter name
7440-48-4	Cobalt
CO-57	Cobalt-57
CO-58	Cobalt-58
10198-40-0	Cobalt-60
7440-50-8	Copper
57-12-5	Cyanide
2051-24-3	Decachlorobiphenyl
319-86-8	Delta-BHC
84-74-2	Di-n-butyl Phthalate
117-84-0	Di-n-octyl Phthalate
53-70-3	Dibenzo(a,h)anthracene
132-64-9	Dibenzofuran
124-48-1	Dibromochloromethane
74-95-3	Dibromomethane
75-71-8	Dichlorodifluoromethane
60-57-1	Dieldrin
DRO	Diesel Range Organics
68334-30-5	Diesel Fuel
84-66-2	Diethyl Phthalate
131-11-3	Dimethyl Phthalate
DO	Dissolved Oxygen
TDS	Dissolved Solids
7429-91-6	Dypsprosium
ECOLI	E. Coli
959-98-8	Endosulfan I
33213-65-9	Endosulfan II
1031-07-8	Endosulfan Sulfate
72-20-8	Endrin
7421-93-4	Endrin Aldehyde
53494-70-5	Endrin Ketone
7440-52-0	Erbium
100-41-4	Ethylbenzene
7440-53-1	Europium
14683-23-9	Europium-152
15585-10-1	Europium-154
EU-155	Europium-155
FECAL	Fecal Coliform Bacteria
206-44-0	Fluoranthene
86-73-7	Fluorene
FL	Fluoride
462-06-6	Fluorobenzene
7440-54-2	Gadolinium
GD-153	Gadolinium-153
5103-74-2	Gamma Chlordane
58-89-9	Gamma-BHC (Lindane)
GRO	Gasoline Range Organics
8006-61-9	Gasoline
HARDCA	Hardness as CaCO3
76-44-8	Heptachlor
1024-57-3	Heptachlor Epoxide
118-74-1	Hexachlorobenzene
87-68-3	Hexachlorobutadiene

Code	Parameter name
77-47-4	Hexachlorocyclopentadiene
67-72-1	Hexachloroethane
110-54-3	Hexane
2691-41-0	HMX
7440-60-0	Holmium
37871-00-4	HpCDD
38998-75-3	HpCDF
34465-46-8	HxCDD
55684-94-1	HxCDF
193-39-5	Indeno(1,2,3-cd)pyrene
IOD	Iodide
I-131	Iodine-131
74-88-4	Iodomethane
IR-192	Iridium-192
7439-89-6	Iron
FE-59	Iron-59
78-59-1	Isophorone
98-82-8	Isopropyl Benzene
7439-91-0	Lanthanum
LA-140	Lanthanum-140
7439-92-1	Lead
PB-210	Lead-210
15092-94-1	Lead-212
15067-28-4	Lead-214
7439-93-2	Lithium
7439-94-3	Lutetium
13777-61-2	m&p-Xylene
7439-95-4	Magnesium
7439-96-5	Manganese
MN-54	Manganese-54
5711900	Maximum Density
571582400	Maximum Dry Density
7439-97-6	Mercury
HG-203	Mercury-203
72-43-5	Methoxychlor
75-09-2	Methylene Chloride
717423200	Minimum Dry Density
3711900	Minimum Density
45800	Moisture
7439-98-7	Molybdenum
MORO	Motor Oil Range Organics
104-51-8	n-Butylbenzene
621-64-7	N-Nitroso-di-n-propylamine
86-30-6	N-Nitrosodiphenylamine
103-65-1	n-propylbenzene
91-20-3	Naphthalene
7440-00-8	Neodymium
NP-237	Neptunium-237
7440-02-0	Nickel
NB-95	Niobium-95
NO3	Nitrate
NO2/NO3	Nitrate-Nitrite-N

Code	Parameter name
1497-55-8	Nitrate/Nitrite
NO2	Nitrite
98-95-3	Nitrobenzene
4165-60-0	Nitrobenzene-d5
NITROGEN	Nitrogen
55-63-0	Nitroglycerin
348-51-6	O-Chloro fluorobenzene
95-47-6	o-Xylene
3268-87-9	OCDD
39001-02-0	OCDF
OIL	Oil
OMC	Optimum Moisture Content
TOC	Organic Carbon
7683200	Organic Content
MOIST	Organic Content / Moisture
99-87-6	p-Isopropyltoluene
36088-22-9	PeCDDI
30402-15-4	PeCDF
87-86-5	Pentachlorophenol
%MOISTURE	Percent Moisture
%SOLIDS	Percent Solids
PERM	Permeability
78-11-5	PETN
1006	pH
85-01-8	Phenanthrene
108-95-2	Phenol
4165-62-2	Phenol-d5
PHENOLICS	Phenolics
PHENOLS	Phenols
PO4T	Phosphate
PHOS	Phosphorous
7723-14-0	Phosphorous
PIMEP	Pimephales
PU-238/239	Plutonium 238/239
13981-16-3	Plutonium-238
PU-239/240	Plutonium-239/240
PU-242	Plutonium-242
PO-210	Polonium-210
1336-36-3	Polychlorinated Biphenyl (PCB)
7440-09-7	Potassium
13966-00-2	Potassium-40
7440-10-0	Praseodym
13981-14-1	Protactinium-233
15100-28-4	Protactinium-234 metastable
15117-48-3	Pu-239
129-00-0	Pyrene
15623-45-7	Radium-223
13233-32-4	Radium-224
RA-225	Radium-225
13982-63-3	Radium-226
RA-228	Radium-228
121-82-4	RDX

Code	Parameter name
EH	Redox Potential
RU-103	Ruthenium-103
13967-48-1	Ruthenium-106
7440-19-9	Samarium
7319568600	Saturated Hydraulic Conductivity
SC-46	Scandium-46
135-98-8	sec-Butylbenzene
7782-49-2	Selenium
7440-21-3	Silicon
7440-22-4	Silver
AG-110	Silver-110
7440-23-5	Sodium
13966-32-0	Sodium-22
EC	Specific Conductance
79125400	Specific Gravity
SG	Specific Gravity
SR-85	Strontium-85
14158-27-1	Strontium-89
10098-97-2	Strontium-90
100-42-5	Styrene
SO4	Sulfate
18496-25-8	Sulfide
TSS	Suspended Solids
7440-25-7	Tantalum
41903-57-5	TCDD, Total
30402-14-3	TCDF
TC-99	Techetium-99
7440-27-9	Terbium
98904-43-9	Terphenyl-d14
98-06-6	tert-Butylbenzene
877-09-8	Tetrachloro-m-xylene
127-18-4	Tetrachloroethene
479-45-8	Tetryl
7440-28-0	Thallium
14913-50-9	Thallium-208
TH-227	Thorium-227
14274-82-9	Thorium-228
14269-63-7	Thorium-230
7440-29-1	Thorium-232
15065-10-8	Thorium-234
7440-30-4	Thulium
7440-31-5	Tin
SN-113	Tin-113
SN-126	Tin-126
108-88-3	Toluene
2037-26-5	Toluene-d8
AHYD	Total Aromatic Hydrocarbons
TOGRHY	Total C5 TO C11 Petroleum Hydrocarbons
THAHYC	Total Halogenated Hydrocarbons
TOX	Total Organic Halides
IPHC	Total Petroleum Hydrocarbon
RATOT	Total Radium

Code	Parameter name
TSVHYC	Total Semivolatile Hydrocarbons
8001-35-2	Toxaphene
79-01-6	Trichloroethene
75-69-4	Trichlorofluoromethane
10028-17-8	Tritium
7440-61-1	Uranium
UDAUGH	Uranium Daughters
U-233	Uranium-233
13966-29-5	Uranium-234
15117-96-1	Uranium-235
U-235/236	Uranium-235/236
U-236	Uranium-236
24678-82-8	Uranium-238
7440-62-2	Vanadium
108-05-4	Vinyl Acetate
75-01-4	Vinyl Chloride
1330-20-7	Xylenes, Total
7440-64-4	Ytterbium
Y-88	Yttrium-88
7440-66-6	Zinc
ZN-65	Zinc-65
ZR-95	Zirconium-95

3.3.6. Unit of Measure

Code	Description
BQ/L	Bequerels per Liter
CM/S	Centimeters/Second
COL/10	Coliform/100 milliliters
CFU/G	Colony Forming Units/Gram
CFU/G	Colony Forming Units/Gram Oven Dried
CFU/ML	Colony Forming Units/Milliliter
CFU/ML	Colony Forming Units/ML
CPM	Counts per Minute
C	Degrees Celsius
F	Degrees Fahrenheit
DPM/G	Disintegrations per Minute per Gram
G/CC	Grams per Cubic Centimeter
L/MIN	Liters per Minute
UG/G	Micrograms per Gram
UG/KG	Micrograms per Kilogram
UG/L	Micrograms per Liter
UG/ML	Micrograms per Liter
MEQ/100	Milliequivalents/100 Grams
MG/KG	Milligrams per Kilogram
MG/L	Milligrams per Liter
NCI/L	NanoCuries per Liter
NG/G	Nanograms per Gram
NTU	National Thermal Units
%	Percent
STD UN	pH Standard Units

Code	Description
PCI/G	PicoCuries per Gram
PCI/L	PicoCuries per Liter
PCI/ML	PicoCuries per Milliliter
PG/G	Picograms per Gram
PCF	Pounds per Cubic Foot

3.3.7. Method Codes

Analysis Type	Method	Method Description
ANION	EPA 325.1	Chloride by CAWW Method 325.1
ANION	EPA 325.2	Chloride by CAWW Method 325.2
ANION	EPA 325.3	Chloride by CAWW Method 325.3
ANION	EPA 340.2	Fluoride by Potentiometric SIE, CAWW Method 340.2
ANION	EPA 351.3	Nitrogen
ANION	EPA 353.2	Nitrate-Nitrite by CAWW Method 353.2
ANION	EPA 353.2	Nitrite by Spectrophometric, CAWW 353.2
ANION	EPA 354.1	Nitrite by Spectrophometric, CAWW 354.1
ANION	EPA 375.2	Sulfate by Turbidimetric, CAWW Method 375.2
ANION	EPA 375.4	Sulfate by Turbidimetric, CAWW Method 375.4
ANION	SW846 8460	SW846 8460
ANION	SW846 9030A	Sulfide Analysis SW846 9030A
ANION	SW846 9036	Sulfate by Colorimetric Automated Methyl Thymol Bl
ANION	SW846 9250	Chloride by SW846 Method 9250
EPTOX	UNKNOWN	Unknown Method
GENERA	ASTMD2974-87	Organic Carbon
GENERA	EPA 160.1	Dissolved Solids
GENERA	EPA 160.2	Suspended Solids
GENERA	EPA 310.1	Alkalinity by Titrimetric (pH 4.5), CAWW Method
GENERA	EPA 310.2	Alkalinity
GENERA	EPA 350.1	Ammonia
GENERA	EPA 351.2	Total Nitrogen
GENERA	EPA 365.1	Total Phosphorous Method EPA 365.1
GENERA	EPA 365.2	Phosphate by Method EPA 365.2
GENERA	EPA 365.3	Total Phosphorous Method EPA 365.3
GENERA	EPA 365.4	Total Phosphorous Method EPA 365.4
GENERA	EPA 415.1	Total Organic Carbon CAWW Method 415.1
GENERA	EPA 415.2	Total Organic Carbon CAWW Method 415.2
GENERA	FID	Fidler Data
GENERA	SW846 7196A	SW846 7196A
GENERA	SW846 9020	Total Organic Halides SW846 9020
GENERA	SW846 9022	Total Organic Halides SW846 9022
GENERA	SW846 9060	Total Organic Carbon Analysis SW846 9060
GENERA	TDMS	Thermal Desorption - Mass Spectrometry (TD-MS)
GEOTEC	SW846 9045	SW846 9045
GEOTEC	SW846 9081	SW846 9081
INORG	CLP 200.7	Metals by ICP, CLP Method 200.7
INORG	CLP 204.2	Metals by Graphite Furnace, CLP Method 204.2

Analysis Type	Method	Method Description
INORG	CLP 206.2	Metals by Graphite Furnace, CLP Method 206.2
INORG	CLP 210.2	Metals by Graphite Furnace, CLP Method 210.2
INORG	CLP 213.2	Metals by Graphite Furnace, CLP Method 213.2
INORG	CLP 215.1	Calcium by Graphite Furnace, CLP Method 215.1
INORG	CLP 218.2	Metals by Graphite Furnace, CLP Method 218.2
INORG	CLP 239.2	Metals by Graphite Furnace, CLP Method 239.2
INORG	CLP 242.1	Magnesium by Graphite Furnace, CLP Method 242.1
INORG	CLP 245.1	Mercury in water by manual cold vapor, CLP 245.1
INORG	CLP 245.2	Mercury in water by automated cold vapor, CLP 245.2
INORG	CLP 245.5	Mercury in Soil by Manual Cold Vapor, CLP Method
INORG	CLP 258.1	Potassium by Graphite Furnace, CLP Method 258.1
INORG	CLP 270.2	Metals by Graphite Furnace, CLP Method 270.2
INORG	CLP 272.2	Metals by Graphite Furnace, CLP Method 272.2
INORG	CLP 273.1	Sodium by Graphite Furnace, CLP Method 273.1
INORG	CLP 279.2	Metals by Graphite Furnace, CLP Method 272.2
INORG	CLP 335.2	Cyanide by one of the CLP Methods
INORG	CLPSOWC	Metals by ICP, CLP Method 200.7
INORG	EPA 200.7	Metals by ICP, Method 200.7
INORG	EPA 204.2	Metals by Graphite Furnace, Method 204.2
INORG	EPA 206.2	Metals by Graphite Furnace, Method 206.2
INORG	EPA 210.2	Metals by Graphite Furnace, Method 210.2
INORG	EPA 213.2	Metals by Graphite Furnace, Method 213.2
INORG	EPA 218.2	Metals by Graphite Furnace, Method 218.2
INORG	EPA 239.2	Metals by Graphite Furnace, Method 239.2
INORG	EPA 245.1	Mercury in water by manual cold vapor, EPA 245.1
INORG	EPA 245.2	Mercury in Water by Auto Cold Vapor, Method 245.2
INORG	EPA 245.5	Mercury in Soil by Manual Cold Vapor, Method 245.5
INORG	EPA 270.2	Metals by Graphite Furnace, Method 270.2
INORG	EPA 335.2	Cyanide by one of the EPA Methods
INORG	PXRF	Spectrace9000 Portable X-Ray Fluoroscope Screening
INORG	SW7430	Lithium by Graphite Furnace, Method SW7430
INORG	SW846 6010	Metals by ICP, Method SW846 6010
INORG	SW846 7060	Arsenic by Furnace AA, SW846 Method 7060
INORG	SW846 7470	Mercury by SW846 7470/7471
INORG	SW846 7740	Selenium by Furnace AA, Method SW846 Method 7740
INORG	SW846 9010	Cyanide by Colorimetric, SW846 Method 9010
INORG	SW846 9012	Cyanide by SW846 9012
ORBTEX	EPA 601	BTEX Compounds
ORBTEX	SW846 8020	BTEX Compounds
ORDIOX	SW846 8280	Polychlorinated Dibenzo-p-Dioxins/Dibenzofurans
ORDIOX	SW846 8290	Polychlorinated Dibenzo-p-Dioxins/Dibenzofurans
ORDRO	EPA 8015M	Diesel Range Organics
OREXP	SW846 8330	Explosives
ORGRO	EPA 8015M	Gasoline Range Organics
ORHERB	SW846 8150	Herbicides by Method SW846 8150
ORPETH	EPA 418.1	Total Recoverable Petroleum Hydrocarbons
ORPETH	EPA 418.1	Total Recoverable Petroleum Hydrocarbons
ORPHNL	SW846 8040A	Phenols by Method SW846 8040A

Analysis Type	Method	Method Description
ORPHNL	SW846 9066	Phenolics Spectrophotometic SW846 9066
ORPPB	CLPPCB	Pesticides/PCBs by GC/ECD, CLP Method 608
ORPPB	SW846 8080	Organochlorine Pesticides/PCBs, SW846 Method 8080
ORPPB	SW846 8240	Volatiles by GC/MS, SW846 Method 8240
ORSVO	CLPSVO	Semi-Volatiles by GC/MS, CLP Method 625
ORSVO	SW846 8010	Halogenated Volatile Organics., SW846 Method 8010
ORSVO	SW846 8021	Volatile Organics by Purge & Trap GC/PID, SW846
ORSVO	SW846 8120	Chlorinated Hydrocarbons, SW846 Method 8120
ORSVO	SW846 8270	Semi-Volatiles by GC/MS Capillary Column, SW846
ORVOA	5890A	Volatile Organics by GC/FID
ORVOA	CLPVOA	Volatiles by GC/MS, CLP Method 624
ORVOA	SW846 8030	Acrolein, Acrylonitrile, Acetonitrile
ORVOA	SW846 8260	Volatiles by GC/MS Capillary Column Method 8260
OTHER	9045	pH
OTHER	9081	Cation Exchange
OTHER	ASTM D1429	Specific Gravity
PH	SW846 9045	pH Analysis USEPA SW846 9045
RAD	ALPHA	Alpha Spectroscopy
RAD	ASTMD2460-70	Radium-226
RAD	BICRON	Bicron Detector screening for Plutonium & Thorium
RAD	DOEE-Pu-06	Unknown Radiological Method
RAD	E84006	Unknown Radiological Method
RAD	EERF-00-07	Thorium Isotopes
RAD	EML AM-01	Americium-241
RAD	EPA 900	Gross Alpha and Gross Beta Radioactivity
RAD	EPA 901	Radioactive Method 901
RAD	EPA 901.0	Radioactive Method 901.0
RAD	EPA 903	Alpha-Emitting Radium Isotopes, Method 903
RAD	EPA 903.1	EPA 903.1
RAD	EPA 905	Radioactive Strontium, Method 905
RAD	EPA 905.1	EPA 905.1
RAD	EPA 906	Tritium, Method 906
RAD	EPA 906.0	Tritium, Method 906.0
RAD	EPA 906.1	Tritium, Method 906.1
RAD	EPA 907.0	EPA 907.0
RAD	EPA 908	Uranium by Radiochemical, Method 908
RAD	EPA 908.0	Uranium by Radiochemical, Method 908.0
RAD	ESM430	Uranium by Radiochemical
RAD	GAMMA SCAN	Gamma Spectrometer screening for radioisotopes
RAD	GERANIUM	Geranium Detector screening for radioisotopes
RAD	HASL 300	Unknown HASL 300 Method
RAD	INSITG	In-situ Gamma Spectrometry with Germanium Detector
RAD	NAS 1960	Thorium Isotopes / Strontium-90
RAD	NAS 1962	Uranium Isotopes
RAD	NAS 1965	Plutonium Isotopes
RAD	NDI1986	Gamma Spectrometry
RAD	PD8030-1302	Radiologicals in Large Soil Samples by an Acid-Lea
RAD	PD8030-1343	Plutonium in Small Soil Samples by an Acid-Leach

Analysis Type	Method	Method Description
RAD	PD8030-1343	Uranium, Thorium Lead-210 in Solid Samples
RAD	PD8030-2403	Tritium in Urine
RAD	PD8030-3605	Plutonium in Small Soil Samples by an Acid-Leach

If a laboratory SOP or another method is used, the laboratory must write a notification letter for enclosure with the EDD. The notification letter will list the method code, method description, and applicable analytes.

DATA VALIDATION METHODS

Table of Contents

Method Number	Method Description	Document
DV-001	CLP Volatile Organic Data Validation	QAPP
DV-002	Volatile Organic Method 8021 Data Validation	QAPP
DV-003	CLP Semi-Volatile Organic Analysis (SVOA) Data Validation	QAPP
DV-004	CLP Pesticide/PCB Data Validation	QAPP
DV-005	CLP Inorganic (Metals) Data Validation	QAPP
DV-006	Cyanide Data Validation	QAPP
DV-007	General Chemistry Data Validation	QAPP
DV-008	Total Dissolved Solids (TDS) and Total Suspended Solids (TSS) Data Validation	QAPP
DV-009	Total Organic Carbon (TOC) Data Validation	QAPP
DV-010	Explosives Data Validation	QAPP
DV-011	Alkalinity Data Validation	QAPP
DV-012	Uranium, Plutonium, and Thorium Alpha Spectrometry Data Validation	QAPP
DV-013	Americium Data Validation	QAPP
DV-014	Tritium Data Validation	QAPP
DV-015	Gamma Spectrometry Data Validation	QAPP
DV-016	Strontium^{90} Data Validation	QAPP
DV-017	Radium^{226} Data Validation	QAPP
DV-018	Volatile Organic Analysis (VOA)/Method SW8030 Data Validation	QAPP
DV-019	Hexavalent Chromium Data Validation	QAPP
DV-020	Volatile Organic Analysis (VOA)/Method SW8020 Data Validation	Compendium
DV-021	Volatile Organic Analysis (VOA) by EPA 601 Data Validation	Compendium
DV-022	Gasoline Range Organic (GRO) Analysis by SW8015 Data Validation	Compendium
DV-023	Total Petroleum Hydrocarbons (TPH) by EPA 418.1 Data Validation	Compendium
DV-024	Diesel Range Organic (DRO) Analysis by SW8015 for Data Validation	Compendium

DATA VALIDATION METHODS

Data validation methods were included in the Compendium to provide consistency to subcontractors who perform data validation and data assessment. The numbers assigned to the data validation methods match the numbers assigned to the corresponding analytical method. These methods describe when a qualification should be applied to a data point and what qualification should be applied. The data validation report format is described in Quality Assurance Procedure Q-006.

The first 18 data validation methods were developed from Appendix H of Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan (QAPP). The subsequent data validation methods were developed for the analytical methods that were not in the original QAPP, but were added to the Compendium. As new data validation methods are required, the methods will be introduced into the Compendium. If appropriate, the title page of the quality assurance procedure will reference a Source Document and Document Date. If the method is not introduced as part of a sample plan, then only the Document Date will be included. When a new method is added, the revised table of contents for the section and the method will be distributed to the copyholders of the Compendium.

The forms described within the methods are available on electronic media as Microsoft ™ Office95® binders. The electronic binder files include method text and the forms in a single file. The electronic files will be provided upon request.

MOUND

Environmental
Restoration
Program

Method: DV-002

Volatile Organic Analysis (VOA)/ Method 8021 Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for volatile organic analysis (VOA) performed by USEPA SW846 SW8021. These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

EPA. 1986. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. November 1986.

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory data package. Missing data is requested from the laboratory as soon as it is noted. If missing data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.

- Results of the equipment rinsate, ambient, sample bank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.

- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- Initial calibration - at least one RRF and %RSD.
- Continuing calibration - at least one RRF and %D.
- Surrogate - at least one set of surrogates from one sample.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
N	Presumptive evidence of presence of material.
NJ	Presumptive evidence of the presence of the material at an estimated quantity.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration
H	Holding time exceeded
K	Qualified due to surrogate recovery
S	Qualified due to matrix spike recovery
I	Qualified due to internal standard
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Holding Time: Verify that samples were analyzed within the holding time period of 14 days. If the holding time was exceeded, qualify results as estimated with (J) and (UJ). If the holding time was grossly

exceeded, professional judgment should be used for qualifying the data and determining whether the data are unusable (R).

Calibration:

Verify that the percent relative standard deviation (%RSD) of the response factors (RF) for the initial 5 point calibration was < 20%. Verify that a continuing calibration was run every 10 samples and that the %difference (%D) in RFs was within 15% of average RF from the initial calibration. If any samples were analyzed with a non-compliant calibration standard, then the results associated with the non-compliant standard must be estimated (J). If %D or %RSD was > 50%, then all positive and non-detected values for that analyte must be estimated (J) and (UJ).

Verify retention time windows: $\pm 3\sigma$ of three retention times for each analyte as per SW846.

Blanks:

Method blanks must be analyzed one per 20 samples of a given matrix per day and contamination must be less than PQL.

Verify that trip blanks are performed one per shipping container to laboratory.

Verify that equipment blanks, sample bank blanks and ambient blanks are performed one every 10 samples or less. These field blanks are qualified using qualification guidance levels derived form method blank contamination. Qualification level for samples are determined from the maximum contamination levels in method and equipment blank contamination.

Qualification Guidance Level for acetone, methylene chloride, toluene, 2-butanone is 10*x* contamination in blank.

Qualification Guidance Level for other compounds is 5x contamination in blank

Qualification Guidelines:

- If results < PQL, report PQL as U.
- If results > PQL and < guidance level, then report the value as U.
- If results > PQL and > guidance level, then report the value unqualified.

Surrogates: The recoveries for the surrogates should be within QC limits specified in Method A-002 of the Methods Compendium. If recoveries are not within QC limits, the samples should be re-extracted and re-analyzed. If recoveries are still outside limits then associated positive sample results should be estimated (J). If recoveries are below 10%, then associated sample results should be considered as unusable (R).

Internal Standards: The recoveries of internal standards fluorobenzene and 2-bromo-1-chloropropane should be within QC Limits specified in Method A-002 of the Methods Compendium. If recoveries are not within QC limits, the samples should be reanalyzed. If recoveries are still outside limits then associated sample results should be estimated (J) and (UJ). If extremely low area counts are reported, then associated non-detected sample results should be considered as unusable (R).

Laboratory Control Sample (LCS): Verify that an LCS was performed 1 per 20 samples of a given matrix per day. Verify that LCS recoveries are within QC limits specified in Method A-002 of the Methods Compendium.

If LCS recoveries are not within QC limits, then all associated samples must be reanalyzed with another LCS. Professional judgment should be used to evaluate data associated with failed LCS analysis. If recoveries are below 10%, then associated results could be considered as unusable (R).

Matrix Spike (MS)/ Matrix Spike Duplicate (MSD): Verify that an MS/MSD was performed one per 20 samples of a given matrix. Verify that MS/MSD recoveries are within QC limits specified in Method A-002 of the Methods Compendium. The positive values for those compounds that fail MS/MSD criteria should be estimated in the unspiked sample. If recoveries are below 10%, then associated results could be considered as unusable (R).

Field Duplicate: Verify that a field duplicate sample was analyzed for every 10 field samples or less of each matrix. The RPD must be < 35% for waters and < 50% for soils. If criteria was not met, then estimate (J) the positive results for that analyte(s) in both samples. If one of the sample results is > PQL and the other is non-detected, then both results are estimated (J, UJ).

Compound ID and Quantitation Limits: Verify that the secondary column confirmation was performed for all results > PQL. If the percent difference (%D) between the two results is > 25, estimate the result. If the %D > 100, it is highly unlikely that the analyte is present or its quantitation is correct or it is the same analyte, therefore, the resultant value should be considered unusable (R). However, if both results are < CRQL and %D > 100% the value should be reported as non-detected (UJ).

Verify that quantitation limits stated in Method A-002 of the Methods Compendium were met.

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Surrogate Recovery Forms

Complete header section. Complete remainder of table only for those samples which have surrogate outliers. If no outliers, then write 'None'.

3.5.3 Matrix Spike Recovery Forms

Complete header section. Complete remainder of table only for those samples which have MS/MSD outliers. Enter the percent recovery as appropriate. The RPD should be entered in parentheses. If no outliers, write 'None'.

3.5.4 Blank Data Summary Forms

Complete the appropriate form from Attachment III. Enter blank outliers only and note appropriate action level based on 5*x* or 10*x*. Enter the associated sample IDs.

3.5.5 Calibration Forms

Complete header section, instrument, and date and time for each calibration run. Enter the outlying RF, RRF, RSD, or %D as necessary. List all associated sample IDs at bottom of form.

3.5.6 Field Duplicate Forms

Complete header section and sample IDs. Enter outlying RPD values for analytes. If no values are outside the required range, so state.

3.5.7 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for surrogate recovery, matrix spike recovery, calibration, field duplicate, and replicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- Initial calibration - at least one RRF and %RSD.
- Continuing calibration - at least one RRF and %D.
- Surrogate - at least one set of surrogates from one sample.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.8 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC ANALYSIS

- ☐ Table of Contents (Check List)
  - I. Case Narrative
    - ☐ Cover Page
    - ☐ Case Narrative
    - ☐ Cross Reference of Sample ID to Laboratory ID
    - ☐ Description of all Qualifiers used by the Laboratory
    - ☐ Applicable Laboratory SOP and Revision Date
  - II. QC Summary
    - ☐ Surrogate Recoveries Summary
    - ☐ Matrix Spike/Matrix Spike Duplicate Summary
    - ☐ Method Blank Summary
    - ☐ Laboratory Control Sample
  - III. Raw Data
    - IIIA. Sample Data
      - ☐ Example Calculation
      - ☐ Target Compound Results
      - ☐ Chromatograms
      - ☐ Quantitation / Calculation of Concentrations
    - IIIB. Standard(s) Data
      - ☐ Initial Calibration Data
      - ☐ Standard(s) Chromatograms and Quantitation Reports
      - ☐ Continuing Calibration
      - ☐ Standard(s) Chromatograms and Quantitation Reports

DATA PACKAGE COMPLETENESS CHECKLIST (Con't)
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC ANALYSIS

IIIC. Raw QC Data

- ☐ Blank Data
  - ☐ Chromatogram
  - ☐ Quantitation / Calculation of Concentrations
- ☐ Matrix Spike Data
  - ☐ Tabulated Results
  - ☐ Chromatogram(s)
- ☐ Matrix Spike Duplicate Data
  - ☐ Tabulated Results
  - ☐ Chromatogram(s)
- ☐ Instrument Settings
- ☐ Instrument Run Logs
- ☐ Percent Solids Determination Logs
- ☐ Sample Preparation/Extraction Logs

IV. Sample Receipt

- ☐ Traffic Reports
- ☐ Sample Log In Record

☐ V. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I	-	Data Outlier Summary Forms
ATTACHMENT II	-	Qualified Data Summary Reports *
ATTACHMENT III	-	Data Completeness Checklist
ATTACHMENT IV	-	Laboratory Case Narrative
ATTACHMENT V	-	Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
Volatile Organics - Method 8021

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Laboratory Sample Identification	Mound Sample Identification	Date Collected	Date Extracted	Date Analyzed	Number of Days Past Holding Time

Holding Time: Analysis within 14 days from collection

Reviewer: _____

Surrogate Recovery Outliers
Volatile Organics - Method 8021

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Soil Quality Control Limits

- Bromochloromethane (70 - 130%)
- 1,4 — Dichlorobutane (60 - 140%)
- 2-bromo-1-chloropropane (60 - 140%)
- Fluorobenzene (70 - 130%)

Water Quality Control Limits

- Bromochloromethane (59 - 117%)
- 1,4 — Dichlorobutane (60 - 140%)
- 2-bromo-1-chloropropane (60 - 140%)
- Fluorobenzene (48 - 120%)

Laboratory Sample Id	Mound Sample Id	Sample Matrix	Bromochloromethane	1,4 — Dichlorobutane	2-bromo-1-chloropropane	Fluorobenzene

Reviewer: _____

Matrix Spike Recovery Outliers
Volatile Organics - Method 8021

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Soil Quality Control Limits

-	Bromodichloromethane	(60 - 140%)	(≤ 30%)
-	Bromoform	(60 - 140%)	(≤ 30%)
-	Carbon Tetrachloride	(60 - 140%)	(≤ 30%)
-	Chloroform	(60 - 140%)	(≤ 30%)
-	Dibromochloromethane	(60 - 140%)	(≤ 30%)
-	1,4 — Dichlorobenzene	(60 - 140%)	(≤ 30%)
-	1,2 — Dichloroethane	(60 - 140%)	(≤ 30%)
-	1,1 — Dichloroethene	(60 - 140%)	(≤ 30%)
-	1,1,1 — Trichloroethane	(60 - 140%)	(≤ 30%)
-	Trichloroethene	(60 - 140%)	(≤ 30%)
-	Vinyl Chloride	(60 - 140%)	(≤ 30%)
-	Benzene	(60 - 140%)	(≤ 30%)

Water Quality Control Limits

-	Bromodichloromethane	(42 - 172%)	(≤ 15%)
-	Bromoform	(13 - 159%)	(≤ 15%)
-	Carbon Tetrachloride	(43 - 143%)	(≤ 15%)
-	Chloroform	(49 - 133%)	(≤ 15%)
-	Dibromochloromethane	(24 - 191%)	(≤ 15%)
-	1,4 — Dichlorobenzene	(42 - 143%)	(≤ 15%)
-	1,2 — Dichloroethane	(51 - 147%)	(≤ 15%)
-	1,1 — Dichloroethene	(28 - 167%)	(≤ 15%)
-	1,1,1 — Trichloroethane	(41 - 138%)	(≤ 15%)
-	Trichloroethene	(35 - 146%)	(≤ 15%)
-	Vinyl Chloride	(28 - 163%)	(≤ 15%)
-	Benzene	(39 - 150%)	(≤ 15%)

Laboratory Sample Id	Mound Sample Id	Sample Matrix	Bromodichloromethane	Bromoform	Carbon Tetrachloride	Chloroform

Reviewer: _____

Matrix Spike Recovery Outliers
Volatile Organics - Method 8021

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Soil Quality Control Limits

-	Bromodichloromethane	(60 - 140%)	(≤ 30%)
-	Bromoform	(60 - 140%)	(≤ 30%)
-	Carbon Tetrachloride	(60 - 140%)	(≤ 30%)
-	Chloroform	(60 - 140%)	(≤ 30%)
-	Dibromochloromethane	(60 - 140%)	(≤ 30%)
-	1,4 — Dichlorobenzene	(60 - 140%)	(≤ 30%)
-	1,2 — Dichloroethane	(60 - 140%)	(≤ 30%)
-	1,1 — Dichloroethene	(60 - 140%)	(≤ 30%)
-	1,1,1 — Trichloroethane	(60 - 140%)	(≤ 30%)
-	Trichloroethene	(60 - 140%)	(≤ 30%)
-	Vinyl Chloride	(60 - 140%)	(≤ 30%)
-	Benzene	(60 - 140%)	(≤ 30%)

Water Quality Control Limits

-	Bromodichloromethane	(42 - 172%)	(≤ 15%)
-	Bromoform	(13 - 159%)	(≤ 15%)
-	Carbon Tetrachloride	(43 - 143%)	(≤ 15%)
-	Chloroform	(49 - 133%)	(≤ 15%)
-	Dibromochloromethane	(24 - 191%)	(≤ 15%)
-	1,4 — Dichlorobenzene	(42 - 143%)	(≤ 15%)
-	1,2 — Dichloroethane	(51 - 147%)	(≤ 15%)
-	1,1 — Dichloroethene	(28 - 167%)	(≤ 15%)
-	1,1,1 — Trichloroethane	(41 - 138%)	(≤ 15%)
-	Trichloroethene	(35 - 146%)	(≤ 15%)
-	Vinyl Chloride	(28 - 163%)	(≤ 15%)
-	Benzene	(39 - 150%)	(≤ 15%)

Laboratory Sample Id	**Mound Sample Id**	**Sample Matrix**	Dibromochloromethane	1,4 — Dichlorobenzene	1,2 — Dichloroethane	1,1 — Dichloroethene

Reviewer: _____

Matrix Spike Recovery Outliers
Volatile Organics - Method 8021

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Soil Quality Control Limits

-	Bromodichloromethane	(60 - 140%)	(≤ 30%)
-	Bromoform	(60 - 140%)	(≤ 30%)
-	Carbon Tetrachloride	(60 - 140%)	(≤ 30%)
-	Chloroform	(60 - 140%)	(≤ 30%)
-	Dibromochloromethane	(60 - 140%)	(≤ 30%)
-	1,4 — Dichlorobenzene	(60 - 140%)	(≤ 30%)
-	1,2 — Dichloroethane	(60 - 140%)	(≤ 30%)
-	1,1 — Dichloroethene	(60 - 140%)	(≤ 30%)
-	1,1,1 — Trichloroethane	(60 - 140%)	(≤ 30%)
-	Trichloroethene	(60 - 140%)	(≤ 30%)
-	Vinyl Chloride	(60 - 140%)	(≤ 30%)
-	Benzene	(60 - 140%)	(≤ 30%)

Water Quality Control Limits

-	Bromodichloromethane	(42 - 172%)	(≤ 15%)
-	Bromoform	(13 - 159%)	(≤ 15%)
-	Carbon Tetrachloride	(43 - 143%)	(≤ 15%)
-	Chloroform	(49 - 133%)	(≤ 15%)
-	Dibromochloromethane	(24 - 191%)	(≤ 15%)
-	1,4 — Dichlorobenzene	(42 - 143%)	(≤ 15%)
-	1,2 — Dichloroethane	(51 - 147%)	(≤ 15%)
-	1,1 — Dichloroethene	(28 - 167%)	(≤ 15%)
-	1,1,1 — Trichloroethane	(41 - 138%)	(≤ 15%)
-	Trichloroethene	(35 - 146%)	(≤ 15%)
-	Vinyl Chloride	(28 - 163%)	(≤ 15%)
-	Benzene	(39 - 150%)	(≤ 15%)

Laboratory Sample Id	Mound Sample Id	Sample Matrix	1,1,1 — Trichloroethane	Trichloroethene	Vinyl Chloride	Benzene

Reviewer: _____

Blank Data Summary Table
Volatile Organics - Method 8021

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

ANALYTE	Method Blank ID	Method Blank ID	Trip Blank ID	Ambient Blank ID	Equip. Blank ID	Action Level
Vinyl Chloride						
Trichlorofluoromethane						
Freon - 113						
1,1 — Dichloroethene						
Methylene Chloride						
trans - 1,2 — Dichloroethene						
1,1 — Dichloroethane						
cis - 1,2 — Dichloroethene						
Chloroform						
1,1,1 — Trichloroethane						
Carbon Tetrachloride						
1,2 — Dichloroethane						
Trichloroethene						
1,2 — Dichloropropane						
Bromodichloromethane						
Dibromomethane						
2 — Chloroethyl Vinyl Ether						
cis - 1,3 — Dichloropropene						
trans - 1,3 — Dichloropropene						
1,1,2 — Trichloroethane						
Tetrachloroethene						
Dibromochloromethane						
1 — Chlorohexane						
Chlorobenzene						
1,1,1,2 — Tetrachloroethane						
Bromoform						
Applies to Samples:						

Reviewer: _____

Blank Data Summary Table
Volatile Organics - Method 8021

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

ANALYTE	Method Blank ID	Method Blank ID	Trip Blank ID	Ambient Blank ID	Equip. Blank ID	Action Level
1,1,2,2 — Tetrachloroethane						
1,2,3 — Trichloropropane						
Bromobenzene						
2 — Chlorotoluene						
4 — Chlorotoluene						
1,3 — Dichlorobenzene						
1,4 — Dichlorobenzene						
1,2 — Dichlorobenzene						
2,2 — Oxybis (1 — Chloropropane)						
Acetone						
Carbon Disulfide						
Vinyl Acetate						
Methylethyl Ketone						
Benzene						
Methylisobutyl Ketone						
Toluene						
Ethylbenzene						
Xylene (Total)						
1,3 — Diethylbenzene						
1,4 — Diethylbenzene						
1,2 — Diethylbenzene						
Diethylbenzene (total)						
Applies to Samples:						

Reviewer: _____

Calibration Outliers
Volatile Organics - Method 8021

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Instrument ID:	Matrix (Soil/Water):

	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
ANALYTE		RSD ≤ 20%	*		D ± 15%	*		D ± 15%	*
Vinyl Chloride									
Trichlorofluoromethane									
Freon - 113									
1,1 — Dichloroethene									
Methylene Chloride									
trans - 1,2 — Dichloroethene									
1,1 — Dichloroethane									
cis - 1,2 — Dichloroethene									
Chloroform									
1,1,1 — Trichloroethane									
Carbon Tetrachloride									
1,2 — Dichloroethane									
Trichloroethene									
1,2 — Dichloropropane									
Bromodichloromethane									
Dibromomethane									
2 — Chloroethyl Vinyl Ether									
Affected Samples: (Client ID)									

* These flags should be applied to the analytes on the sample data sheets.

Reviewer: _____

Calibration Outliers
Volatile Organics - Method 8021

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Instrument ID:	Matrix (Soil/Water):

ANALYTE	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
		RSD ≤ 20%	*		D ± 15%	*		D ± 15%	*
cis - 1,3 — Dichloropropene									
trans - 1,3 — Dichloropropene									
1,1,2 — Trichloroethane									
Tetrachloroethene									
Dibromochloromethane									
1 — Chlorohexane									
Chlorobenzene									
1,1,1,2 — Tetrachloroethane									
Bromoform									
1,1,2,2 — Tetrachloroethane									
1,2,3 — Trichloropropane									
Bromobenzene									
2 — Chlorotoluene									
4 — Chlorotoluene									
1,3 — Dichlorobenzene									
1,4 — Dichlorobenzene									
1,2 — Dichlorobenzene									

Affected Samples: (Client ID)			

* These flags should be applied to the analytes on the sample data sheets.

Reviewer: _____

Calibration Outliers
Volatile Organics - Method 8021

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Instrument ID:	Matrix (Soil/Water):

	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
ANALYTE		RSD ≤ 20%	*		D ± 15%	*		D ± 15%	*
2,2 — Oxybis (1 — Chloropropane)									
Acetone									
Carbon Disulfide									
Vinyl Acetate									
Methylethyl Ketone									
Benzene									
Methylisobutyl Ketone									
Toluene									
Ethylbenzene									
Xylene (Total)									
1,3 — Diethylbenzene									
1,4 — Diethylbenzene									
1,2 — Diethylbenzene									
Diethylbenzene (total)									
Affected Samples: (Client ID)									

Was a continuing calibration run every 10 samples? ☐ **Yes** ☐ **No**
Were retention times acceptable? ☐ **Yes** ☐ **No**

* These flags should be applied to the analytes on the sample data sheets.

Reviewer: _____

Field Duplicate Data Summary Table
Volatile Organics - Method 8021

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC LIMITS
Water = ≤ 35% RPD Soil = NA

ANALYTE	Mound Sample ID	Mound Sample ID	Mound Sample ID	Mound Sample ID
Vinyl Chloride				
Trichlorofluoromethane				
Freon - 113				
1,1 — Dichloroethene				
Methylene Chloride				
trans - 1,2 — Dichloroethene				
1,1 — Dichloroethane				
cis - 1,2 — Dichloroethene				
Chloroform				
1,1,1 — Trichloroethane				
Carbon Tetrachloride				
1,2 — Dichloroethane				
Trichloroethene				
1,2 — Dichloropropane				
Bromodichloromethane				
Dibromomethane				
2 — Chloroethyl Vinyl Ether				
cis - 1,3 — Dichloropropene				
trans - 1,3 — Dichloropropene				
1,1,2 — Trichloroethane				
Tetrachloroethene				
Dibromochloromethane				
1 — Chlorohexane				
Chlorobenzene				

Reviewer: _____

Field Duplicate Data Summary Table
Volatile Organics - Method 8021

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC LIMITS
Water = ≤ 35% RPD Soil = NA

ANALYTE	Mound Sample ID	Mound Sample ID	Mound Sample ID	Mound Sample ID
1,1,1,2 — Tetrachloroethane				
Bromoform				
1,1,2,2 — Tetrachloroethane				
1,2,3 — Trichloropropane				
Bromobenzene				
2 — Chlorotoluene				
4 — Chlorotoluene				
1,3 — Dichlorobenzene				
1,4 — Dichlorobenzene				
1,2 — Dichlorobenzene				
2,2 — Oxybis (1 — Chloropropane)				
Acetone				
Carbon Disulfide				
Vinyl Acetate				
Methylethyl Ketone				
Benzene				
Methylisobutyl Ketone				
Toluene				
Ethylbenzene				
Xylene (Total)				
1,3 — Diethylbenzene				
1,4 — Diethylbenzene				
1,2 — Diethylbenzene				
Diethylbenzene (total)				

Was a duplicate sample analyzed for every 10 samples? ☐ **Yes** ☐ **No**

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-003

CLP Semi-Volatile Organic Analysis (SVOA)/Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for semi-volatile organic analysis (SVOA) performed by CLP SOW 1.8. These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

U.S. EPA 1991. "Functional Guidelines for Organic Data Review," U.S. Environmental Protection Agency, Contract Laboratory Program, June 1991.

U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multimedia, Multi-Concentration. Document No. OLM01.8.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory data package. Missing data is requested from the laboratory as soon as it is noted. If missing data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.
- Results of the equipment rinsate, ambient, sample bank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.
- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- Initial calibration - at least two RRF and %RSD for semi-volatile organics.
- Continuing calibration - at least two RRF and %D for semi-volatile organics.
- Surrogate - at least two surrogates from one sample within a batch.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
N	Presumptive evidence of presence of material.
NJ	Presumptive evidence of the presence of the material at an estimated quantity.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration
H	Holding time exceeded
K	Qualified due to surrogate recovery
S	Qualified due to matrix spike recovery
I	Qualified due to internal standard
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Introduction: Follow the Functional Guidelines For Organic Data Review (FGFODR) and apply the FGFODR to these additional criteria:

The additional compounds have been added to the TCL in OLM0.1.8 and their CRQL are as follows:

Analyte	CRQL Water (ug/L)	CRQL Soil (ug/kg)
Benzoic Acid	50	1600
Benzyl Alcohol	10	330
2-Benzyl-4-chlorophenol	10	330

Holding Time: Apply the FGFODR criteria.

Calibration: Verify the additional compounds 2-benzyl-4-chlorophenol, benzoic acid and benzyl alcohol are included in the Target Compound List (TCL) at the required concentration and that they meet the minimum response factor (RF) requirement of 0.01 and the SOW calibration criteria and follow the FGFODR.

Blanks:	Apply the FGFODR criteria.
Surrogates:	Apply the FGFODR criteria.
Matrix Spike:	Apply the FGFODR criteria.
I.S. Retention Time:	Apply the FGFODR criteria.
I.S. Area:	Apply the FGFODR criteria.
Field Duplicate:	The field duplicate RPD must be ≤ 55% for water samples and no limits have been set for soils. The same validation criteria apply to these additional compounds.

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Surrogate Recovery Forms

Complete header section. Complete remainder of table only for those samples which have surrogate outliers. If no outliers, then write 'None'.

3.5.3 Matrix Spike Recovery Forms

Complete header section. Complete remainder of table only for those samples which have MS/MSD outliers. Enter the percent recovery as appropriate. If no outliers, write 'None'.

3.5.4 Blank Data Summary Forms

Complete the appropriate form from Attachment III. Enter blank outliers only and note appropriate action level based on 5*x* or 10*x*. Enter the associated sample IDs.

3.5.5 Calibration Forms

Complete header section, instrument, and date and time for each calibration run. Enter the outlying RF, RRF, RSD, or %D as necessary. List all associated sample IDs at bottom of form.

3.5.6 Internal Standard Forms

Complete header section. In area limits box, write actual area values from Form VIII. For IS outliers, enter the sample ID and enter the actual outlying area value.

3.5.7 Field Duplicate Forms

Complete header section and sample IDs. Enter outlying RPD values for analytes. If no values are outside the required range, so state.

3.5.8 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for surrogate recovery, matrix spike recovery, calibration, and field duplicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- Initial calibration - at least two RRF and %RSD for semi-volatiles.
- Continuing calibration - at least two RRF and %D for semi-volatiles for each continuing calibration.
- Surrogate - at least two surrogates from one sample within a batch for semi-volatiles.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.9 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

SEMIVOLATILES ORGANIC COMPOUNDS (SVOCs)

- ☐ Table of Contents (Check List)
  - I. Case Narrative
    - ☐ Cover Page
    - ☐ Case Narrative
    - ☐ Cross Reference of Sample ID to Laboratory ID
    - ☐ Description of all Qualifiers used by the Laboratory
    - ☐ Applicable Laboratory SOP and Revision Date
  - II. QC Summary
    - ☐ Surrogate Percent Recovery Summary (Form II SV)
    - ☐ Matrix Spike / Matrix Spike Duplicate Summary (Form III SV)
    - ☐ Method Blank Summary (Form IV SV)
    - ☐ GC / MS Instrument Performance Check (Form V SV)
    - ☐ Internal Standard Area and RT Summary (Form VIII SV)
    - ☐ Laboratory Control Sample
  - III. Raw Data
    - IIIA. Sample Data
      - ☐ Example Calculation
      - ☐ Target Compound Results - Organic Analysis Data Sheet (Form I SV)
      - ☐ Tentatively Identified Compounds (Form I SV - TIC)
      - ☐ Reconstructed Total Ion Chromatograms (RIC)
      - ☐ Quantitation Report
      - ☐ Raw Spectra and Background - Subtracted Mass Spectra of Target Compounds Identified
      - ☐ GC / MS Library Search Spectra for TIC (3 Best Library Matches)
      - ☐ Quantitation / Calculation of TIC Concentrations
    - IIIB. Standard(s) Data
      - ☐ Initial Calibration Data (Form VI SV-1, SV-2)
      - ☐ SV Standard(s) Reconstructed Ion Chromatograms and Quantitation Reports
      - ☐ Continuing Calibration (Form VII SV-1, SV-2)
      - ☐ SV Standard(s) Reconstructed Ion Chromatograms and Quantitation Reports
      - ☐ Semivolatile GPC Calibration Data

DATA PACKAGE COMPLETENESS CHECKLIST (Con't)
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

SEMIVOLATILES ORGANIC COMPOUNDS (SVOCs)

IIIC. Raw QC Data

- ☐ DFTPP
  - ☐ Reconstructed Total Ion Chromatogram (RIC)
  - ☐ Bar Graph Spectrum
  - ☐ Mass Listing
- ☐ Blank Data
  - ☐ Tabulated Results (Form I SV-1, SV-2)
  - ☐ TIC's (Form I SV-TIC)
  - ☐ Reconstructed Ion Chromatogram
  - ☐ Target Compound Spectra
  - ☐ GC / MS Library Search Spectra For TIC
  - ☐ Quantitation / Calculation of TIC Concentrations
- ☐ Matrix Spike Data
  - ☐ Tabulated Results (Form I)
  - ☐ Reconstructed Ion Chromatogram(s)
- ☐ Matrix Spike Duplicate Data
  - ☐ Tabulated Results (Form I SV-1, SV-2)
  - ☐ Reconstructed Ion Chromatogram(s)
- ☐ Instrument Settings
- ☐ Instrument Run Logs
- ☐ Sample Extraction / Preparation Logs
- ☐ Percent Solids Determination Logs

IV. Sample Receipt

- ☐ Traffic Reports
- ☐ Sample Log In Record

☐ V. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I - Data Outlier Summary Forms

ATTACHMENT II - Qualified Data Summary Reports *

ATTACHMENT III - Data Completeness Checklist

ATTACHMENT IV - Laboratory Case Narrative

ATTACHMENT V - Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Laboratory Sample Identification	Mound Sample Identification	Date Collected	Date Extracted	Date Analyzed	Number of Days Past Holding Time

Reviewer: _____

Surrogate Compound Recovery Outliers
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Soil Quality Control Limits

- Nitrobenzene-d5 (NBZ) (23-120%)
- 2-Fluorobiphenyl (FBP) (30-115%)
- p-Terphenyl-d14 (TPH) (18-137%)
- Phenol-d5 (PHL) (24-113%)
- 2-Fluorophenol (2FP) (25-121%)
- 2,4,6-Tribromophenol (TBP) (19-122%)
- 2-Chlorophenol-d4 (2CP) (20-130%)
- 1,2-Dichlorobenzene-d4 (DCB) (20-130%)

Water Quality Control Limits

- Nitrobenzene-d5 (NBZ) (35 - 114%)
- 2-Fluorobiphenyl (FBP) (43 - 116%)
- p-Terphenyl-d14 (TPH) (33 - 141%)
- Phenol-d5 (PHL) (10 - 110%)
- 2-Fluorophenol (2FP) (10 - 110%)
- 2,4,6-Tribromophenol (TBP) (10 - 123%)
- 2-Chlorophenol-d4 (2CP) (33 - 110%)
- 1,2-Dichlorobenzene-d4 (DCB) (16 - 110%)

Laboratory Sample Id	Mound Sample Id	Sample Matrix	NBZ	FBP	TPH	PHL	2FP	TBP	2CP	DCB

Reviewer: _____

Matrix Spike Recovery Outliers
Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Soil Quality Control Limits

-	Phenol	(26 - 90%)	/ RPD<35%)
-	2-Chlorophenol	(25 - 102%)	/ RPD<50%)
-	1,4-Dichlorobenzene	(28 - 104%)	/ RPD<27%)
-	N-nitroso-di-n-propylamine	(41 - 126%)	/ RPD<38%)
-	1,2,4-Trichlorobenzene	(38 - 107%)	/ RPD<23%)
-	4-Chloro-3-methylphenol	(26 - 103%)	/ RPD<33%)
-	Acenaphthene	(31 - 137%)	/ RPD<19%)
-	4-Nitrophenol	(11 - 114%)	/ RPD<50%)
-	2,4-Dinitrotoluene	(28 - 89%)	/ RPD<47%)
-	Pentachlorophenol	(17 - 109%)	/ RPD<47%)
-	Pyrene	(35 - 142%)	/ RPD<36%)

Water Quality Control Limits

-	Phenol	(12 - 110%)	/ RPD<42%)
-	2-Chlorophenol	(27 - 123%)	/ RPD<40%)
-	1,4-Dichlorobenzene	(36 - 97%)	/ RPD<28%)
-	N-nitroso-di-n-propylamine	(41 - 116%)	/ RPD<38%)
-	1,2,4-Trichlorobenzene	(39 - 98%)	/ RPD<28%)
-	4-Chloro-3-methylphenol	(23 - 97%)	/ RPD<42%)
-	Acenaphthene	(46 - 118%)	/ RPD<31%)
-	4-Nitrophenol	(10 - 80%)	/ RPD<50%)
-	2,4-Dinitrotoluene	(24 - 96%)	/ RPD<38%)
-	Pentachlorophenol	(9 -103%)	/ RPD<50%)
-	Pyrene	(26 - 127%)	/ RPD<31%)

Laboratory Sample ID	Mound Sample ID	Sample Matrix	Phenol			2-Chlorophenol			1,4-Dichlorobenzene			N-nitroso-di-n-propylamine			1,2,4-Trichlorobenzene		
			%R	%R	RPD	%R	%R	RPD	%R	%R	RPD	%R	%R	RPD	%R	%R	RPD

Reviewer: _____

Matrix Spike Recovery Outliers
Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Soil Quality Control Limits

-	Phenol	(26 - 90%)	/ RPD<35%)
-	2-Chlorophenol	(25 - 102%)	/ RPD<50%)
-	1,4-Dichlorobenzene	(28 - 104%)	/ RPD<27%)
-	N-nitroso-di-n-propylamine	(41 - 126%)	/ RPD<38%)
-	1,2,4-Trichlorobenzene	(38 - 107%)	/ RPD<23%)
-	4-Chloro-3-methylphenol	(26 - 103%)	/ RPD<33%)
-	Acenaphthene	(31 - 137%)	/ RPD<19%)
-	4-Nitrophenol	(11 - 114%)	/ RPD<50%)
-	2,4-Dinitrotoluene	(28 - 89%)	/ RPD<47%)
-	Pentachlorophenol	(17 - 109%)	/ RPD<47%)
-	Pyrene	(35 - 142%)	/ RPD<36%)

Water Quality Control Limits

-	Phenol	(12 - 110%)	/ RPD<42%)
-	2-Chlorophenol	(27 - 123%)	/ RPD<40%)
-	1,4-Dichlorobenzene	(36 - 97%)	/ RPD<28%)
-	N-nitroso-di-n-propylamine	(41 - 116%)	/ RPD<38%)
-	1,2,4-Trichlorobenzene	(39 - 98%)	/ RPD<28%)
-	4-Chloro-3-methylphenol	(23 - 97%)	/ RPD<42%)
-	Acenaphthene	(46 - 118%)	/ RPD<31%)
-	4-Nitrophenol	(10 - 80%)	/ RPD<50%)
-	2,4-Dinitrotoluene	(24 - 96%)	/ RPD<38%)
-	Pentachlorophenol	(9 -103%)	/ RPD<50%)
-	Pyrene	(26 - 127%)	/ RPD<31%)

Laboratory Sample ID	Mound Sample ID	4-Chloro-3-methylphenol			Acenaphthene			4-Nitrophenol			2,4-Dinitrotoluene			Pentachloro-phenol			Pyrene		
		%R	%R	RPD	%R	%R	RPD	%R	%R	RPD	%R	%R	RPD	%R	%R	RPD	%R	%R	RPD

Reviewer: _____

Blank Data Outlier Summary Table
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

ANALYTE	Method Blank ID	Method Blank ID	Equip. Blank ID			Action Level
Phenol						
Bis(2-chloroethyl)ether						
2-Chlorophenol						
1,3-Dichlorobenzene						
1,4-Dichlorobenzene						
Benzyl Alcohol						
1,2-Dichlorobenzene						
2-Methylphenol						
2,2'-oxybis (1-chloropropane)						
4-Methylphenol						
N-Nitroso-di-n-propylamine						
Hexachloroethane						
Nitrobenzene						
Isophorone						
2-Nitrophenol						
2,4-Dimethylbenzene						
Benzoic Acid						
Bis(2-chloroethoxy)methane						
Affected Samples: (Client ID)						

Reviewer: _____

Blank Data Outlier Summary Table
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

ANALYTE	Method Blank ID	Method Blank ID	Equip. Blank ID			Action Level
2,4-Dichlorophenol						
1,2,4-Trichlorobenzene						
Naphthalene						
4-Chloroaniline						
Hexachlorobutadiene						
4-Chloro-3-methylphenol						
2-Methylnaphthalene						
Hexachlorocyclopentadiene						
2,4,6-Trichlorophenol						
2,4,5-Trichlorophenol						
2-Chloronaphthalene						
2-Nitroaniline						
Dimethylphthalate						
Acenaphthylene						
2,6-Dinitrotoluene						
3-Nitroaniline						
Acenaphthene						
2,4-Dinitrophenol						
Affected Samples: (Client ID)						

Reviewer: _____

Blank Data Outlier Summary Table
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

ANALYTE	Method Blank ID	Method Blank ID	Equip. Blank ID			Action Level
4-Nitrophenol						
Dibenzofuran						
2,4-Dinitrotoluene						
Diethylphthalate						
4-Chlorophenyl-phenylether						
Fluorene						
4-Nitroaniline						
4,6-Dinitro-2-methylphenol						
N-nitrosodiphenylamine						
4-Bromophenyl-phenylether						
Hexachlorobenzene						
Pentachlorophenol						
Phenanthrene						
Anthracene						
Carbazole						
Di-n-butylphthalate						
Fluoranthene						
Affected Samples: (Client ID)						

Reviewer: _____

Blank Data Outlier Summary Table
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

ANALYTE	Method Blank ID	Method Blank ID	Equip. Blank ID			Action Level
Pyrene						
Butylbenzylphthalate						
3,3'-Dichlorobenzidine						
Benzo(a)anthracene						
Crysene						
Bis(2-ethylhexyl)phthalate						
Di-n-octyl phthalate						
Benzo(b) Fluoranthene						
Benzo(a) pyrene						
Indeno(1,2,3-cd)pyrene						
Dibenz(a,h)anthracene						
Benzo(g,h,i) perylene						
2-Benzyl-4-chlorophenol						
Benzo(k) Fluoranthene						
Affected Samples: (Client ID)						

Reviewer: _____

Calibration Outlier Summary Table
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Instrument ID:

	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
ANALYTE	RRF < 0.05	RSD>30		RRF<0.05	%D<25		RRF<0.05	%D<25	
Phenol									
Bis(2-chloroethyl)ether									
2-Chlorophenol									
1,3-Dichlorobenzene									
1,4-Dichlorobenzene									
Benzyl Alcohol									
1,2-Dichlorobenzene									
2-Methylphenol									
2,2'-oxybis(1-chloropropane)									
4-Methylphenol									
N-Nitroso-di-n-propylamine									
Hexachloroethane									
Nitrobenzene									
Isophorone									
2-Nitrophenol									
2,4-Dimethylbenzene									
Benzoic Acid									
Bis(2-chloroethoxy)methane									
Affected Samples: (Client ID)									

Was a Continuing Calibration check run every 12 hours? ☐ Yes ☐ No

Reviewer: _____

Calibration Outlier Summary Table
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Instrument ID:

ANALYTE	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
	RRF < 0.05	RSD>30		RRF<0.05	%D<25		RRF<0.05	%D<25	
2,4-Dichlorophenol									
1,2,4-Trichlorobenzene									
Naphthalene									
4-Chloroaniline									
Hexachlorobutadiene									
4-Chloro-3-methylphenol									
2-Methylnaphthalene									
Hexachlorocyclopentadiene									
2,4,6-Trichlorophenol									
2,4,5-Trichlorophenol									
2-Chloronaphthalene									
2-Nitroaniline									
Dimethylphthalate									
Acenaphthylene									
2,6-Dinitrotoluene									
3-Nitroaniline									
Acenaphthene									
2,4-Dinitrophenol									
Affected Samples: (Client ID)									

Was a Continuing Calibration check run every 12 hours? ☐ Yes ☐ No

Reviewer: _____

Calibration Outlier Summary Table
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Instrument ID:

ANALYTE	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
	RRF < 0.05	RSD>30		RRF<0.05	%D<25		RRF<0.05	%D<25	
4-Nitrophenol									
Dibenzofuran									
2,4-Dinitrotoluene									
Diethylphthalate									
4-Chlorophenyl-phenylether									
Fluorene									
4-Nitroaniline									
4,6-Dinitro-2-methylphenol									
N-nitrosodiphenylamine									
4-Bromophenyl-phenylether									
Hexachlorobenzene									
Pentachlorophenol									
Phenanthrene									
Anthracene									
Carbazole									
Di-n-butylphthalate									
Fluoranthene									
Affected Samples: (Client ID)									

Was a Continuing Calibration check run every 12 hours? ☐ Yes ☐ No

Reviewer: _____

Calibration Outlier Summary Table
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Instrument ID:

ANALYTE	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
	RRF < 0.05	RSD>30		RRF<0.05	%D<25		RRF<0.05	%D<25	
Pyrene									
Butylbenzylphthalate									
3,3'-Dichlorobenzidine									
Benzo(a)anthracene									
Crysene									
Bis(2-ethylhexyl)phthalate									
Di-n-octyl phthalate									
Benzo(b) Fluoranthene									
Benzo(a) pyrene									
Indeno(1,2,3-cd)pyrene									
Dibenz(a,h)anthracene									
Benzo(g,h,i) perylene									
2-Benzyl-4-chlorophenol									
Benzo(k) Fluoranthene									

Affected Samples: (Client ID)			

Was a Continuing Calibration check run every 12 hours? ☐ Yes ☐ No

Reviewer: _____

Internal Standard Recovery Outliers
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Quality Control Limits: Area: - 50% to + 100% RT: ± 30 seconds	**Internal Standard**	**Daily Standard Area**	**Lower Area Limit (- 50%)**	**Upper Area Limit (+ 100%)**
	1,4-Dichlorobenzene-d4 (DCB)			
	Naphthalene-d8 (NPT)			
	Acenaphthene-d10 (ANT)			
	Phenanthrene-d10 (PHN)			
	Chrysene-d12 (CRY)			
	Perylene-d12 (PRY)			

Laboratory Sample ID	**Mound Sample ID**	**Sample Matrix**	**(IS1) DCB**	**(IS2) NPT**	**(IS3) ANT**	**(IS4) PHN**	**(IS5) CRY**	**(IS6) PRY**

Reviewer: _____

Field Duplicate Data Summary Table
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Field Sample Identification:	
Field Duplicate Identification:	

ANALYTE	Field Sample Result	Duplicate Result	RPD
Phenol			
Bis(2-chloroethyl)ether			
2-Chlorophenol			
1,3-Dichlorobenzene			
1,4-Dichlorobenzene			
Benzyl Alcohol			
1,2-Dichlorobenzene			
2-Methylphenol			
2,2'-oxybis(1-chloropropane)			
4-Methylphenol			
N-Nitroso-di-n-propylamine			
Hexachloroethane			
Nitrobenzene			
Isophorone			
2-Nitrophenol			
2,4-Dimethylbenzene			
Benzoic Acid			
Bis(2-chloroethoxy)methane			
2,4-Dichlorophenol			
1,2,4-Trichlorobenzene			
Naphthalene			
4-Chloroaniline			

Was a field duplicate sample submitted for every 10 field samples of each matrix? ☐ Yes ☐ No

Reviewer: _____

Field Duplicate Data Summary Table
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Field Sample Identification:	
Field Duplicate Identification:	

ANALYTE	Field Sample Result	Duplicate Result	RPD
Hexachlorobutadiene			
4-Chloro-3-methylphenol			
2-Methylnaphthalene			
Hexachlorocyclopentadiene			
2,4,6-Trichlorophenol			
2,4,5-Trichlorophenol			
2-Chloronaphthalene			
2-Nitroaniline			
Dimethylphthalate			
Acenaphthylene			
2,6-Dinitrotoluene			
3-Nitroaniline			
Acenaphthene			
2,4-Dinitrophenol			
4-Nitrophenol			
Dibenzofuran			
2,4-Dinitrotoluene			
Diethylphthalate			
4-Chlorophenyl-phenylether			
Fluorene			
4-Nitroaniline			
4,6-Dinitro-2-methylphenol			

Was a field duplicate sample submitted for every 10 field samples of each matrix? ☐ Yes ☐ No

Reviewer: _____

Field Duplicate Data Summary Table
Semi-Volatile Organics - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Field Sample Identification:	
Field Duplicate Identification:	

ANALYTE	Field Sample Result	Duplicate Result	RPD
N-nitrosodiphenylamine			
4-Bromophenyl-phenylether			
Hexachlorobenzene			
Pentachlorophenol			
Phenanthrene			
Anthracene			
Carbazole			
Di-n-butylphthalate			
Fluoranthene			
Pyrene			
Butylbenzylphthalate			
3,3'-Dichlorobenzidine			
Benzo(a)anthracene			
Crysene			
Bis(2-ethylhexyl)phthalate			
Di-n-octyl phthalate			
Benzo(b) Fluoranthene			
Benzo(a) pyrene			
Indeno(1,2,3-cd)pyrene			
Dibenz(a,h)anthracene			
Benzo(g,h,i) perylene			
2-Benzyl-4-chlorophenol			
Benzo(k) Fluoranthene			

Was a field duplicate sample submitted for every 10 field samples of each matrix? ☐ Yes ☐ No

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-005

CLP Inorganic (Metals) Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for inorganic (metals) analysis performed by CLP SOW 3.0. These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

USEPA 1989. "Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analysis," U.S. Environmental Protection Agency, Hazardous Site Evaluation Division, October 1989.

EPA 1990b. "U.S. EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, Multimedia, Multi-Concentration." Document No. ILM1.0 including Revisions 1.1 through 1.8. Environmental Protection Agency, March, 1990.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory data package. Missing data is requested from the laboratory as soon as it is noted. If missing

data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.
- Results of the equipment rinsate, ambient, sample bank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.
- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
N	Presumptive evidence of presence of material.
NJ	Presumptive evidence of the presence of the material at an estimated quantity.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration
H	Holding time exceeded
S	Qualified due to matrix spike recovery
I	Interference (ICP Serial Dilution, ICS, or GFAA spike recovery)
L	Qualified due to LCS
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)
D	Duplicate (replicate) limits exceeded

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Follow the Functional Guidelines for Evaluating Inorganic Analyses (FGEIA) (EPA, 1988) and apply the following additional criteria:

The following changes have been incorporated into the CLP SOW Document No. ILM03.0 as Modification A:

Introduction: TAL metals analysis has been extended to include lithium, molybdenum, bismuth and tin. These additional elements have the following CRDLs:

Analyte	CRDL Water (ug/L)	CRDL Soil (mg/kg)
Lithium	100	10
Molybdenum	20	2
Bismuth	150	30
Tin	50	10

All data deliverables are required to include these added elements.

In addition, lower detection limits have been specified for four elements. These are as follows:

Analyte	CRDL Water (ug/L)	CRDL Soil (mg/kg)
Aluminum	20	4
Antimony	10	2
Beryllium	1	0.2
Vanadium	10	2

See the CLP SOW Modification A for details on analytical methodology for these additional metals.

Holding Time: Apply the same holding times as other TAL metals (180 days/28 days for mercury).

Initial and Continuing Calibration Verification: Additional elements as listed above must be incorporated in the calibration. The same criteria as established in the FGEIA are to be utilized in the evaluation of these analytes.

CRDL Standard for AA and ICP: Additional elements as listed above must be incorporated in the TAL. The same criteria as established in the FGEIA are to be utilized in the evaluation of these elements.

Blanks: Additional elements and lower CRDLs as presented above must be included in the blank analyses. The same criteria as established in the FGEIA are to be utilized in the evaluation of these analytes in the laboratory blanks. If there is no contamination in the laboratory blanks, then sample data will be qualified based on the field blank result under the FGEIA blank rules.

ICP Interference Check Sample: Solution AB shall include the following analytes:

Analyte	mg/L
Lithium	1.0
Molybdenum	1.0
Bismuth	1.0
Tin	1.0

A level ± 20% of the true value is to be used as the acceptance criteria. Follow FGEIA for qualification if %D > 20%.

Matrix Spike Recovery: Spikes shall include the following elements at the concentrations specified below:

Analyte	For ICP/Flame AA		For Furnace AA	
	Water (ug/L)	Soil (mg/kg)	Water (ug/L)	Soil (mg/kg)
Lithium	100	10	NA	NA
Molybdenum	20	2	NA	NA
Bismuth	150	30	NA	NA
Tin	50	10	100	50

Spike recoveries for these elements must meet the same criteria as established in the FGEIA.

Post Digest Spike Sample Recovery: Apply FGEIA guidelines.

Duplicates: Additional elements as listed above must be incorporated in the TAL. The aqueous sample criteria for these analytes shall be ≤ 25% RPD for field duplicates. For laboratory duplicates the criteria is ± 20% RPD if the result is ≥ 5*x* the CRDL, and if the result is ≤ 5*x* CRDL, the criteria is ± the CRDL.

For soil samples, the criteria shall be ≤ 50% RPD for field duplicates. For laboratory duplicates the criteria is ± 35% RPD if

the result is ≥ 5*x* the CRDL, and if the result is ≤ 5*x* CRDL, the criteria is ± 2*x* CRDL. Qualification of data is to be performed using the guidelines in FGEIA.

Laboratory Control Sample: All of the four additional elements (Li, Mo, Sb and Sn) will be included in the LCS analysis. The acceptance criteria for these analytes must be in accordance with the FGEIA.

ICP Serial Dilutions: If sample results are > 50*x* the IDL, a serial dilution is required. The results of this dilution for these elements must be ± 10%. Positive results exceeding this criteria will be estimated (J).

IDL (Quarterly): Additional elements and lower CRDLs as listed above must be incorporated. Follow FGEIA for these additional elements.

ICP Interelement Correction Factors (Annually): Additional elements as listed above must be incorporated. Follow FGEIA for these additional elements.

ICP Linear Ranges (Quarterly): Additional elements as listed above must be incorporated. Follow FGEIA for these additional elements.

Analysis Run Log: Additional elements as listed above must be incorporated.

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Laboratory QC Data Summary Forms

Complete header section. Complete remainder of table only for those samples which have outliers. If no outliers, then write 'None'.

3.5.3 Matrix Spike Recovery Forms

Complete header section. Complete remainder of table only for those samples which have MS/MSD outliers. Enter the percent recovery as appropriate. If no outliers, write 'None'.

3.5.4 Blank Data Summary Forms

Complete the appropriate form from Attachment III. Enter blank outliers only and note appropriate action level. Enter the associated sample IDs.

3.5.5 Calibration Forms

Complete header section, instrument, and date and time for each calibration run. Enter the outlying criteria as necessary. List all associated sample IDs at bottom of form.

3.5.6 Replicate Summary Forms

Complete header section. If outliers, enter sample IDs, outlier value, and qualification designation.

3.5.7 Field Duplicate Forms

Complete header section and sample IDs. Enter outlying RPD values for analytes. If no values are outside the required range, so state.

3.5.8 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for surrogate recovery, matrix spike recovery, calibration, field duplicate, and replicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.9 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

METALS / CYANIDE

☐ Table of Contents (Check List)

I. Case Narrative

- ☐ Cover Page
- ☐ Case Narrative
- ☐ Cross Reference of Sample ID to Laboratory ID
- ☐ Description of all Qualifiers used by the Laboratory
- ☐ Applicable Laboratory SOP and Revision Date

II. Raw Data

IIA. Sample Data

- ☐ Results Form I - IN

IIB. Quality Control Data

- ☐ Initial and Continuing Calibration Verification [Form II (Part I) - IN]
- ☐ CRDL Standard For AA and ICP [Form II (Part II) - IN]
- ☐ Blanks [Form III - IN]
- ☐ ICP Interference Check Sample [Form IV - IN]
- ☐ Spike Sample Recovery [Form VA (Part I) - IN]
- ☐ Post Digest Spike Sample Recovery [Form VB (Part II) - IN]
- ☐ Duplicates [Form VI - IN]
- ☐ Laboratory Control Sample [Form VII - IN]
- ☐ Standard Addition Results [Form VIII - IN]
- ☐ ICP Serial Dilutions [Form IX - IN]
- ☐ Instrument Detection Limits (Quarterly) [Form X - IN]
- ☐ ICP Interelement Correction Factors (Annually) [Form XI (Part I) - IN]
- ☐ ICP Interelement Correction Factors (Annually) [Form XI (Part II) - IN]
- ☐ ICP Linear Ranges (Quarterly) [Form XII - IN]
- ☐ Preparation Log [Form XIII - IN]
- ☐ Analysis Run Log [Form XIV - IN]

DATA PACKAGE COMPLETENESS CHECKLIST (Con't)
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

METALS / CYANIDE

IIC. Raw Data

- ☐ Example Calculation
- ☐ Instrument Settings
- ☐ ICP Raw Data
- ☐ Furnace AA Raw Data
- ☐ Mercury Raw Data
- ☐ Cyanide Raw Data
- ☐ Preparation and Distillation Logs Raw Data
- ☐ Percent Solids Determination Logs

IID. Standards

- ☐ Preparation Records
- ☐ Analysis Records AA and ICP Logs

III. Sample Receipt

- ☐ Traffic Reports
- ☐ Sample Log In Record

☐ IV. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I - Data Outlier Summary Forms

ATTACHMENT II - Qualified Data Summary Reports *

ATTACHMENT III - Data Completeness Checklist

ATTACHMENT IV - Laboratory Case Narrative

ATTACHMENT V - Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Laboratory Sample Identification	Mound Sample Identification	Date Collected	Date Analyzed	Number of Days Past Holding Time

Reviewer: _____

Laboratory QC Data Summary Table
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

LCS Matrix:

ANALYTE	LCS > ± 20%	LCS > ± 20%	ICP Serial Dilution > 10% D	Qualification
Aluminum				
Antimony				
Arsenic				
Barium				
Beryllium				
Cadmium				
Calcium				
Chromium				
Cobalt				
Copper				
Iron				
Lead				
Magnesium				
Manganese				
Mercury				
Nickel				
Potassium				
Selenium				
Silver				
Sodium				
Thallium				
Vanadium				
Zinc				
Lithium				
Bismuth				
Molybdenum				
Tin				
Cyanide			NA	
Applies to Samples (Client ID):				

Reviewer: _____

Matrix Spike Recovery Data Summary Table
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC LIMITS
75 - 125%

ANALYTE	Mound Sample ID	Mound Sample ID	Mound Sample ID	Mound Sample ID	Mound Sample ID
Aluminum					
Antimony					
Arsenic					
Barium					
Beryllium					
Cadmium					
Calcium					
Chromium					
Cobalt					
Copper					
Iron					
Lead					
Magnesium					
Manganese					
Mercury					
Nickel					
Potassium					
Selenium					
Silver					
Sodium					
Thallium					
Vanadium					
Zinc					
Lithium					
Bismuth					
Molybdenum					
Tin					
Cyanide					

Reviewer: _____

Blank Outliers
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Preparation Blank Matrix (Soil/Water):	Preparation Blank Concentration Units (UG/L or MG/KG):

ANALYTE	Equip. Blank	Prep Blank	Init Cal Blank	CCB1	CCB2	CCB3	Max Cone	Action Level
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								
Lithium								
Bismuth								
Molybdenum								
Tin								
Cyanide								
Applies to Samples (Client ID):								

Reviewer: _____

Calibration Outliers
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Initial Calibration Source:	Continuing Calibration Source:

	Calibration Verification			Continuing Calibration Verification					
ANALYTE	True	Found	% R (1)	True	Found	% R (1)	Found	% R (1)	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Lithium									
Bismuth									
Molybdenum									
Tin									
Cyanide									
Applies to Samples (Client ID):									

(1) Control Limits: Mercury 80-120%;
Other Metals 90-110%; Cyanide 85-115%

Reviewer: _____

Duplicate Sample (Replicate) Data Summary Table
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC Limits
If Result > 5*X* CRDL, Then ± 20% RPD
If Result < 5*X* CRDL, Then ± CRDL

ANALYTE	CRDL Water	CRDL Soil	Mound Sample ID Primary	Mound Sample ID Replicate	RPD	Qualification
Aluminum	20	4				
Antimony	10	2				
Arsenic	10	2				
Barium	200	40				
Beryllium	1	0.2				
Cadmium	5	1				
Calcium	5000	1000				
Chromium	10	2				
Cobalt	50	10				
Copper	25	5				
Iron	100	20				
Lead	3	0.6				
Magnesium	5000	1000				
Manganese	15	3				
Mercury	0.2	0.1				
Nickel	40	8				
Potassium	5000	1000				
Selenium	5	1				
Silver	10	2				
Sodium	5000	1000				
Thallium	10	2				
Vanadium	10	2				
Zinc	20	4				
Lithium	100	10				
Bismuth	150	30				
Molybdenum	20	2				
Tin	50	10				
Cyanide	10	2				

Reviewer: _____

Field Duplicate Data Summary Table
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC Limits
Water: ± 25% RPD

ANALYTE	Mound Sample ID Primary	Mound Sample ID Replicate	RPD	Qualification
Aluminum				
Antimony				
Arsenic				
Barium				
Beryllium				
Cadmium				
Calcium				
Chromium				
Cobalt				
Copper				
Iron				
Lead				
Magnesium				
Manganese				
Mercury				
Nickel				
Potassium				
Selenium				
Silver				
Sodium				
Thallium				
Vanadium				
Zinc				
Lithium				
Bismuth				
Molybdenum				
Tin				
Cyanide				

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-006

Cyanide Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for cyanide analysis performed by CLP SOW ILM03.0. These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

USEPA 1989. "Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analysis," U.S. Environmental Protection Agency, Hazardous Site Evaluation Division, October 1989.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

EPA. 1996. "U.S. EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, Multi-media, Multi-Concentration." Document No. ILM03.0. U.S. Environmental Protection Agency, March 1990.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory data package. Missing data is requested from the laboratory as soon as it is noted. If missing

data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.
- Results of the equipment rinsate, ambient, sample bank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.
- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- MS/MSD
- LCS

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
N	Presumptive evidence of presence of material.
NJ	Presumptive evidence of the presence of the material at an estimated quantity.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration
H	Holding time exceeded
S	Qualified due to matrix spike recovery
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Holding Time: If a sample is analyzed outside the 14 day holding time, qualify the result estimated (J, UJ). If the holding time is grossly exceeded, apply professional judgment and determine whether to reject (R) the data.

Initial and Continuing Calibration Verification: The same criteria as established in the FGEIA are to be utilized in the evaluation of cyanide.

Blanks: The same criteria as established in the FGEIA are to be utilized in the evaluation of cyanide in the laboratory blanks. If there is no contamination in the laboratory blanks, then sample data will be qualified based on the field blank result under the FGEIA blank rules.

Matrix Spike Recovery: Spike recoveries for cyanide must meet the same criteria as established in the FGEIA.

Field Duplicates: If the RPD for aqueous field duplicates is greater than 25%, then the results will be qualified estimated (J, UJ).

If the RPD for soil field duplicates is greater than 50%, then the results will be qualified estimated (J, UJ).

Laboratory Replicates: If the sample results are less than 5*x* the CRDL, then results which vary by more than the value of the CRDL are qualified estimated (J, UJ).

If an aqueous sample result is greater than 5*x* the CRDL and the RPD for the duplicates is greater than 20%, then the results will be qualified estimated (J, UJ).

If a soil sample result is greater than 5*x* the CRDL, and the RPD for the duplicates is greater than 35%, then the results will be qualified estimated (J, UJ).

Laboratory Control Sample: The acceptance criterion for Cyanide must be in accordance with the FGEIA.

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Laboratory QC Summary Forms

Complete header section. Complete remainder of table only for those samples which have outliers. If no outliers, then write 'None'.

3.5.3 Matrix Spike Recovery Forms

Complete header section. Complete remainder of table only for those samples which have MS/MSD outliers. Enter the percent recovery as appropriate. If no outliers, write 'None'.

3.5.4 Blank Data Summary Forms

Complete the appropriate form from Attachment III. Enter blank outliers only and note appropriate action level based on 5*x* or 10*x*. Enter the associated sample IDs.

3.5.5 Calibration Forms

Complete header section. Enter any calibration outliers into the table and indicate the associated samples. If no outliers, then write 'None'.

3.5.6 Replicate Summary Forms

Complete header section. If outliers, enter sample IDs, outlier value, and qualification designation.

3.5.7 Field Duplicate Forms

Complete header section and sample IDs. Enter outlying RPD values for analytes. If no values are outside the required range, so state.

3.5.8 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for surrogate recovery, matrix spike recovery, calibration, field duplicate, and replicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- MS/MSD
- LCS

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.9 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

METALS / CYANIDE

☐ Table of Contents (Check List)

I. Case Narrative

- ☐ Cover Page
- ☐ Case Narrative
- ☐ Cross Reference of Sample ID to Laboratory ID
- ☐ Description of all Qualifiers used by the Laboratory
- ☐ Applicable Laboratory SOP and Revision Date

II. Raw Data

IIA. Sample Data

- ☐ Results Form I - IN

IIB. Quality Control Data

- ☐ Initial and Continuing Calibration Verification [Form II (Part I) - IN]
- ☐ CRDL Standard For AA and ICP [Form II (Part II) - IN]
- ☐ Blanks [Form III - IN]
- ☐ ICP Interference Check Sample [Form IV - IN]
- ☐ Spike Sample Recovery [Form VA (Part I) - IN]
- ☐ Post Digest Spike Sample Recovery [Form VB (Part II) - IN]
- ☐ Duplicates [Form VI - IN]
- ☐ Laboratory Control Sample [Form VII - IN]
- ☐ Standard Addition Results [Form VIII - IN]
- ☐ ICP Serial Dilutions [Form IX - IN]
- ☐ Instrument Detection Limits (Quarterly) [Form X - IN]
- ☐ ICP Interelement Correction Factors (Annually) [Form XI (Part I) - IN]
- ☐ ICP Interelement Correction Factors (Annually) [Form XI (Part II) - IN]
- ☐ ICP Linear Ranges (Quarterly) [Form XII - IN]
- ☐ Preparation Log [Form XIII - IN]
- ☐ Analysis Run Log [Form XIV - IN]

DATA PACKAGE COMPLETENESS CHECKLIST (Con't)
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

METALS / CYANIDE

IIC. Raw Data

- ☐ Example Calculation
- ☐ Instrument Settings
- ☐ ICP Raw Data
- ☐ Furnace AA Raw Data
- ☐ Mercury Raw Data
- ☐ Cyanide Raw Data
- ☐ Preparation and Distillation Logs Raw Data
- ☐ Percent Solids Determination Logs

IID. Standards

- ☐ Preparation Records
- ☐ Analysis Records AA and ICP Logs

III. Sample Receipt

- ☐ Traffic Reports
- ☐ Sample Log In Record

☐ IV. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I	-	Data Outlier Summary Forms
ATTACHMENT II	-	Qualified Data Summary Reports *
ATTACHMENT III	-	Data Completeness Checklist
ATTACHMENT IV	-	Laboratory Case Narrative
ATTACHMENT V	-	Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Laboratory Sample Identification	Mound Sample Identification	Date Collected	Date Analyzed	Number of Days Past Holding Time

Reviewer: _____

Laboratory QC Data Summary Table
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

LCS Matrix:

ANALYTE	LCS > ± 20%	LCS > ± 20%	ICP Serial Dilution > 10% D	Qualification
Aluminum				
Antimony				
Arsenic				
Barium				
Beryllium				
Cadmium				
Calcium				
Chromium				
Cobalt				
Copper				
Iron				
Lead				
Magnesium				
Manganese				
Mercury				
Nickel				
Potassium				
Selenium				
Silver				
Sodium				
Thallium				
Vanadium				
Zinc				
Lithium				
Bismuth				
Molybdenum				
Tin				
Cyanide			NA	
Applies to Samples (Client ID):				

Reviewer: _____

Matrix Spike Recovery Data Summary Table
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC LIMITS
75 - 125%

ANALYTE	Mound Sample ID	Mound Sample ID	Mound Sample ID	Mound Sample ID	Mound Sample ID
Aluminum					
Antimony					
Arsenic					
Barium					
Beryllium					
Cadmium					
Calcium					
Chromium					
Cobalt					
Copper					
Iron					
Lead					
Magnesium					
Manganese					
Mercury					
Nickel					
Potassium					
Selenium					
Silver					
Sodium					
Thallium					
Vanadium					
Zinc					
Lithium					
Bismuth					
Molybdenum					
Tin					
Cyanide					

Reviewer: _____

Blank Outliers
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Preparation Blank Matrix (Soil/Water):	Preparation Blank Concentration Units (UG/L or MG/KG):

ANALYTE	Equip. Blank	Prep Blank	Init Cal Blank	CCB1	CCB2	CCB3	Max Cone	Action Level
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								
Lithium								
Bismuth								
Molybdenum								
Tin								
Cyanide								
Applies to Samples (Client ID):								

Reviewer: _____

Calibration Outliers
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Initial Calibration Source:	Continuing Calibration Source:

	Calibration Verification			Continuing Calibration Verification					
ANALYTE	True	Found	% R (1)	True	Found	% R (1)	Found	% R (1)	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Lithium									
Bismuth									
Molybdenum									
Tin									
Cyanide									
Applies to Samples (Client ID):									

(1) Control Limits: Mercury 80-120%;
Other Metals 90-110%; Cyanide 85-115%

Reviewer: _____

Duplicate Sample (Replicate) Data Summary Table
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC Limits
If Result > 5*X* CRDL, Then ± 20% RPD
If Result < 5*X* CRDL, Then ± CRDL

ANALYTE	CRDL Water	CRDL Soil	Mound Sample ID Primary	Mound Sample ID Replicate	RPD	Qualification
Aluminum	20	4				
Antimony	10	2				
Arsenic	10	2				
Barium	200	40				
Beryllium	1	0.2				
Cadmium	5	1				
Calcium	5000	1000				
Chromium	10	2				
Cobalt	50	10				
Copper	25	5				
Iron	100	20				
Lead	3	0.6				
Magnesium	5000	1000				
Manganese	15	3				
Mercury	0.2	0.1				
Nickel	40	8				
Potassium	5000	1000				
Selenium	5	1				
Silver	10	2				
Sodium	5000	1000				
Thallium	10	2				
Vanadium	10	2				
Zinc	20	4				
Lithium	100	10				
Bismuth	150	30				
Molybdenum	20	2				
Tin	50	10				
Cyanide	10	2				

Reviewer: _____

Field Duplicate Data Summary Table
Inorganic Compounds - CLP SOW

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC Limits
Water: ± 25% RPD

ANALYTE	Mound Sample ID Primary	Mound Sample ID Replicate	RPD	Qualification
Aluminum				
Antimony				
Arsenic				
Barium				
Beryllium				
Cadmium				
Calcium				
Chromium				
Cobalt				
Copper				
Iron				
Lead				
Magnesium				
Manganese				
Mercury				
Nickel				
Potassium				
Selenium				
Silver				
Sodium				
Thallium				
Vanadium				
Zinc				
Lithium				
Bismuth				
Molybdenum				
Tin				
Cyanide				

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-008

Total Dissolved Solids (TDS) and Total Suspended Solids (TSS) Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for total dissolved and total suspended solids analysis performed by USEPA methods. These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

EPA. 1993. "Methods for Chemical Analysis of Water and Waste," U.S. Environmental Protection Agency, EPA - 600/4-79-020 revised March 1983.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory data package. Missing data is requested from the laboratory as soon as it is noted. If missing data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.

- Results of the equipment rinsate, ambient, sample bank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.

- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- LCS - at least one compound.

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration

H	Holding time exceeded
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Holding Time: Verify the samples were analyzed within the holding time in Method A-008 in the Methods Compendium. If the holding time was exceeded, qualify results as estimated with (J) or (UJ). If the holding time was grossly exceeded, professional judgment may be used for qualifying the data as unusable (R).

Balance Check: Verify the balance was checked with S class weights daily before use. If balance was not calibrated, qualify all results obtained on that balance as estimated since the last successful calibration.

Blanks: Method blank must be analyzed every 20 samples and contamination must be less than PQL.

Equipment blank sample are qualified using qualification guidance levels derived from method blank contamination. Qualification guidance level for samples is determined from the maximum contamination levels in method and equipment blank contamination. If the contamination is greater than the PQL, then:

Qualification Guidance Level = 5x contamination in blank.

Qualification Guidelines:

- If results < PQL, then no action is taken.
- If results < 5*x* contamination, then report the value with a (U).
- If results > 5*x* contamination, then no action is taken.

Laboratory Control Sample: Verify a Laboratory Control Sample (LCS) was analyzed with each initial calibration to demonstrate its validity. If the criteria was not met, then:

- If the recovery is below criteria and greater than 10 percent, qualify positive results estimated, (J) and non-detects unusable, (R).
- If the recovery is less than or equal to 10 percent, then qualify all results unusable, (R).
- For recoveries greater than the specified criteria, no action is taken for non-detects and positive results will be qualified estimated, (J). For recoveries that grossly exceed the specified criteria, positive results may be qualified unusable, (R), based on the validator's professional judgment.

Field Duplicate: Verify a field duplicate sample was analyzed for every 10 field samples or less of each matrix. The RPD must be ≤ 25% for water. If RPD > 25% qualify all sample results associated with that duplicate sample as estimated (J).

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 TSS/TDS Data Summary Forms

Complete header section. Fill in remainder if criteria was exceeded.

3.5.3 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for the field duplicate along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- LCS - at least one compound.

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.4 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

ANION (circle): NO_2-NO_3 SO_4 Cl F TOC TKN TP NH_4 TSS TDS

GENERAL CHEMISTRY AND TOC

- ☐ Table of Contents (Check List)
  - I. Case Narrative
    - ☐ Cover Page
    - ☐ Case Narrative
    - ☐ Cross Reference of Sample ID to Laboratory ID
    - ☐ Description of all Qualifiers used by the Laboratory
    - ☐ Applicable Laboratory SOP and Revision Date
    - ☐ Summary of Sample Results
    - ☐ Summary of Quality Control Results
    - ☐ Summary of Analysis and Preparation Dates
  - II. Raw Data
    - IIA. Sample Data
      - ☐ Example Calculation Sheet
      - ☐ Instrument Settings
      - ☐ Analytical Raw Data (Behind Each Result)
    - IIB. Standards Data
      - ☐ Standards Preparation Data
      - ☐ Initial and Continuing Calibration
    - IIC. Raw QC Data
      - ☐ Quality Control Results [Blanks, LCS, MS/MSD, Replicate (TOC Only)]
      - ☐ Quality Control Raw Data (Behind Each Result)
      - ☐ Instrument Run Logs / Instrument Calibration / Tuning
      - ☐ Sample Preparation / Extraction / Digestion Logs
      - ☐ Percent Solids Determination Logs
  - III. Sample Receipt
    - ☐ Traffic Reports
    - ☐ Sample Log In Record
- ☐ IV. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I - Data Outlier Summary Forms

ATTACHMENT II - Qualified Data Summary Reports *

ATTACHMENT III - Data Completeness Checklist

ATTACHMENT IV - Laboratory Case Narrative

ATTACHMENT V - Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
TSS and TDS

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Laboratory Sample Identification	Mound Sample Identification	Date Collected	Analysis	Date Analyzed	Number of Days Past Holding Time

Reviewer: _____

Data Summary Table
TSS and TDS

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Instrument ID For:	

ANALYTE	Calibration		Blanks		
	Initial	Continuing	Method ID	Method ID	Method ID
TSS	NA	NA			
TDS	NA	NA			
Applies to Samples (Client ID):					

Laboratory Control Samples **(80 - 120% R)**

Sample ID	TSS		TDS	
	% R	% RPD	% R	% RPD

Field Duplicates **(Water = < 25% RPD)**

ANALYTE	Sample ID	Sample ID	Sample ID	Sample ID
TSS				
TDS				

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-010

Explosives Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for explosives analysis performed by USEPA Method SW8330. These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

EPA. 1986. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. November 1986.

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory data package. Missing data is requested from the laboratory as soon as it is noted. If missing data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.

- Results of the equipment rinsate, ambient, sample bank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.

- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- Initial calibration - at least one RF and %RSD.
- Continuing calibration - at least one RF and %D.
- Surrogate - at least one set of surrogates from one sample.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.

R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
N	Presumptive evidence of presence of material.
NJ	Presumptive evidence of the presence of the material at an estimated quantity.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration
H	Holding time exceeded
K	Qualified due to surrogate recovery
S	Qualified due to matrix spike recovery
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Holding Time: Verify the samples were extracted and analyzed holding times specified in Method A-010 of the Methods Compendium. Qualify results as estimated with (J) and (UJ) if holding times were exceeded. If the holding time was grossly exceeded, professional judgment may be used for qualifying the data as unusable (R).

Calibration: Verify the Percent Relative Standard Deviation (% RSD) of the Response Factors (RF) for the initial 5-point calibration was ≤ 20%. Verify that a continuing calibration check was run every 10 samples and that the percent difference (%D) in RFs was within 15% of average RF from the initial calibration. If any samples were analyzed with a non-compliant calibration standard, then the positive results associated with a non-compliant standard must be estimated (J).

If %D or %RSD was > 50% then all positive and non-detected values for that analyte must be estimated (J) and unusable (R), respectively. If the %D or %RSD is grossly outside criteria, professional judgment may be used for qualifying the data unusable (R).

Blanks:

Method blank must be analyzed for each batch of samples extracted for each matrix and contamination must be less than PQL.

Equipment blank sample are qualified using qualification guidance levels derived from method blank contamination. Action Level for samples are determined from the maximum contamination levels in method and equipment blank contamination. If the contamination is greater than the PQL, then for each affected analyte:

Qualification Guidance Level = 5x contamination in blank.

Qualification Guidelines:

- If results < PQL, then no action is taken.
- If results < 5*x* contamination, then report the value with a (U).
- If results > 5*x* contamination, then no action is taken.

Matrix Spike:

Recoveries are compound dependant and should correspond to those specified in Method A-010 of the Methods Compendium for soils and water.

Matrix Spike Duplicate:

The RPDs are compound dependant and should correspond to those specified in Method A-010 of the Methods Compendium for soils and water.

The positive values for those compounds that fail MS/MSD criteria but have recoveries > 10% should be estimated in the unspiked sample.

For a matrix spike analyte which is outside recovery criteria, but whose recovery is greater than 10%, then positive analyte sample results in associated samples are qualified estimated (J). Non-detected analyte results in associated samples are evaluated on professional judgment.

For a matrix spike analyte which is outside recovery criteria, with a recovery less than 10%, then positive analyte sample results in associated samples are qualified estimated (J). Non-detected analyte results in associated samples are rejected (R).

Surrogates:

Two surrogates are used in this analysis:

- Nitroglycerin is the surrogate corresponding to PETN. If recovery for nitroglycerin is not within QC limits as specified in Method A-010 of the Methods Compendium, the sample must be re-extracted and reanalyzed. If the recovery is still outside QC limits, but > 10%, then a positive result for PETN is qualified as estimated (J) and non-detected PETN result is evaluated on professional judgment. If nitroglycerin recovery is < 10%, then positive PETN result is estimated and non-detected PETN result is rejected (R).

  Verify that the wavelength of the detector is set 220nm for PETN and nitroglycerin analysis. Unless supporting documentation for selecting a wavelength other than 220nm is present, reject (R) all data at a different wavelength other than 220nm.

- 4-Nitrotoluene is used as the surrogate for all the remaining analytes. If recovery for 4-nitrotoluene is not within QC limits as specified in Method A-010 of the Methods Compendium, the sample must be re-extracted and re-analyzed. If the recovery is still outside QC limits, but > 10%, then positive results are qualified as estimated (J) and non-detected results are evaluated on professional judgment. If 4-nitrotoluene recovery is < 10%, then positive results are estimated and non-detected results are rejected (R).

Laboratory Control Sample:

Laboratory control sample (LCS) recoveries must be within QC limits in Method A-010 or all associated samples must be re-extracted with another LCS. If the criteria was not met, then:

- If the recovery is below criteria and greater than 10%, qualify positive results estimated, (J) and non-detects unusable, (R).

- If the recovery is less than or equal to 10 percent, then qualify all results unusable, (R).

- For recoveries greater than the specified criteria, no action is taken for non-detects and positive results will be qualified estimated, (J). For recoveries that grossly exceed the specified criteria, positive results may be qualified unusable, (R), based on the validator's professional judgment.

Field Duplicate: Verify a field duplicate sample was analyzed for every 10 field samples or less of each matrix. The RPD must be ≤ 35% for water and ≤ 50% for soils. If criteria were not met, then estimate (J) the positive results for that analyte(s) in both samples. If one of the sample result is > PQL and the other is non-detected, then both results are estimated.

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Surrogate Recovery Forms

Complete header section. Complete remainder of table only for those samples which have surrogate outliers. If no outliers, then write 'None'.

3.5.3 Matrix Spike Recovery Forms

Complete header section. Complete remainder of table only for those samples which have MS/MSD outliers. Enter the percent recovery as appropriate. If no outliers, write 'None'. This form will also be used for laboratory control sample outliers.

3.5.4 Blank Data Summary Forms

Complete the appropriate form from Attachment III. Enter blank outliers only and note appropriate action level based on $5x$ or $10x$. Enter the associated sample IDs.

3.5.5 Calibration Forms

Complete header section, instrument, and date and time for each calibration run. Enter the outlying RF, RRF, RSD, or %D as necessary. List all associated sample IDs at bottom of form.

3.5.6 Field Duplicate Forms

Complete header section and sample IDs. Enter outlying RPD values for analytes. If no values are outside the required range, so state.

3.5.7 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for surrogate recovery, matrix spike recovery, calibration, field duplicate, and replicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- Initial calibration - at least one RF and %RSD.
- Continuing calibration - at least one RF and %D.
- Surrogate - at least one set of surrogates from one sample.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.8 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

EXPLOSIVES

- ☐ Table of Contents (Check List)
  - I. Case Narrative
    - ☐ Cover Page
    - ☐ Case Narrative
    - ☐ Cross Reference of Sample ID to Laboratory ID
    - ☐ Description of Data Qualifiers used in the Report
    - ☐ Applicable Laboratory SOP and Revision Date
    - ☐ Summary of Sample Results
    - ☐ Summary of Quality Control Results
    - ☐ Summary of Analysis and Preparation Dates
  - II. Raw Data
    - IIA. Sample Data
      - ☐ Example Calculation
      - ☐ Instrument Settings
      - ☐ Analytical Raw Data (Behind Each Result)
    - IIB. Standards Data
      - ☐ Standards Preparation Data
      - ☐ Initial and Continuing Calibration
      - ☐ Retention Time Windows
      - ☐ Instrument Calibration / Tuning
      - ☐ Raw Data Behind Each Result
    - IIC. Raw QC Data
      - ☐ Quality Control Results (Blanks, LCSs, MS/MSDs)
      - ☐ Quality Control Raw Data (Behind Each Result)
      - ☐ Instrument Run Log
      - ☐ Sample Preparation / Extraction / Digestion Logs
      - ☐ Percent Solids Determination Logs
  - III. Sample Receipt
    - ☐ Traffic Reports
    - ☐ Sample Log In Record
- ☐ IV. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I	-	Data Outlier Summary Forms
ATTACHMENT II	-	Qualified Data Summary Reports *
ATTACHMENT III	-	Data Completeness Checklist
ATTACHMENT IV	-	Laboratory Case Narrative
ATTACHMENT V	-	Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
Explosive Compounds - SW8330

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Laboratory Sample Identification	Mound Sample Identification	Date Collected	Date Extracted	Date Analyzed	Number of Days Past Holding Time

Reviewer: _____

Surrogate Recovery Outliers
Explosive Compounds - SW8330

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Soil Quality Control Limits - Nitroglycerin (50 - 150%) - 4-Nitrotoluene (50 - 150%)	**Water Quality Control Limits** - Nitroglycerin (50 - 150%) - 4-Nitrotoluene (50 - 150%)

Laboratory Sample Id	Mound Sample Id	Sample Matrix	Nitroglycerin		4-Nitrotoluene	
			C1	C2	C1	C2

Reviewer: _____

Matrix Spike Recovery Outliers
Explosives Compounds - SW8330

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Soil Quality Control Limits (high concentration)

- RDX (40 - 160%) / RPD<30%)
- 1,3,5-TNB (40 - 160%) / RPD<30%)
- 2,4,6-TNT (40 - 160%) / RPD<30%)
- 2,6-DNT (40 - 160%) / RPD<30%)
- 2,4-DNT (40 - 160%) / RPD<30%)
- PETN (40 - 160%) / RPD<30%)

Water Quality Control Limits (low concentration)

- RDX (62 - 87%) / RPD<20%)
- 1,3,5-TNB (85 - 100%) / RPD<20%)
- 2,4,6-TNT (78 - 102%) / RPD<20%)
- 2,6-DNT (66 - 102%) / RPD<20%)
- 2,4-DNT (74 - 99%) / RPD<20%)
- PETN (75 - 100%) / RPD<20%)

Laboratory Sample ID	Mound Sample ID	Sample Matrix	RDX			1,3,5-TNB			2,4,6-TNT			2,6-DNT			2,4-DNT			PETN		
			%R	%R	RPD	%R	%R	RPD	%R	%R	RPD	%R	%R	RPD	%R	%R	RPD	%R	%R	RPD

Reviewer: _____

Blank Data Outlier Summary Table
Explosive Compounds - SW8330

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

ANALYTE	Method Blank ID	Instrument Blank ID	Equip. Blank ID		Action Level
HMX					
RDX					
1,3,5-TNB					
1,3-DNB					
NB					
Tetryl					
TNT					
2A,4,6-DNT					
2,6-DNT					
2,4-DNT					
PETN					
Applies to Samples:					

Reviewer: _____

Calibration Outlier Summary Table
Explosive Compounds - SW8330

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Instrument ID:

ANALYTE	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
		RSD ≤ 20	*		%D ≤ 15%	*		%D ≤ 15%	*
HMX									
RDX									
1,3,5-TNB									
1,3-DNB									
NB									
Tetryl									
2,4,6-TNT									
2A,4,6-DNT									
2,6-DNT									
2,4-DNT									
PETN									
Affected Samples: (Client ID)									

* These flags should be applied to the analytes on the sample data sheets.

Reviewer: _____

Field Duplicate Data Summary Table
Explosive Compounds - SW8330

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC LIMITS
Water = ≤ 35% RPD

ANALYTE	Mound Sample ID	Mound Sample ID	Mound Sample ID	Mound Sample ID
HMX				
RDX				
1,3,5-TNB				
1,3-DNB				
NB				
Tetryl				
TNT				
2A,4,6-DNT				
2,6-DNT				
2,4-DNT				
PETN				

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-014

Tritium Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for tritium analysis. These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

EPA. "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," U.S. Environmental Protection Agency, EPA-600/4-80-032, latest version.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory data package. Missing data is requested from the laboratory as soon as it is noted. If missing data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.

- Results of the equipment rinsate, ambient, sample bank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.

- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- MS/MSD
- Method Spike

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
N	Presumptive evidence of presence of material.
NJ	Presumptive evidence of the presence of the material at an estimated quantity.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration
H	Holding time exceeded
S	Qualified due to matrix spike recovery
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Holding Time: Verify the samples were analyzed within the holding time specified in Method A-014 of the Methods Compendium. If holding time was exceeded, the sample results should be qualified as estimated (J) or (UJ). If the holding time was grossly exceeded, professional judgment may be used for qualifying the data as unusable (R).

Calibration: Verify the instrument being used has been calibrated according to the requirements of Method A-014 of the Methods Compendium. The frequency of calibration should be at a minimum of once a year.

Source Check: Verify the check source was counted on the same day before or during the run of samples being measured and that its value is within 3σ of its mean value. If this criterion is not met, qualify all of the results measured on that day as estimated, (J).

Background: Verify the background was counted each day, that the background was within 3σ of its mean and that the appropriate background was used to subtract from the sample counts. If the background was $> 3\sigma$, qualify all of the results measured on that day as estimated, (J).

Efficiency: Verify the correct efficiency was used in the calculation.

Method Blank: Verify the method blank activity is $\leq 2x$ the MDA. If the blank activity is greater than $2x$ the MDA, then use professional judgment. If the blank activity is greater than $2x$ the MDA and the sample activity is less than $3x$ the blank activity, then the result is

rejected (R). If the blank activity is greater than 2*x* the MDA and the sample activity is greater than 3*x* the blank activity, the result is estimated (J).

Method Spike: Verify the method spike is within ± 3σ of the theoretical spike value. If the method spike is outside the specified criteria in Method A-014 of the Methods Compendium, results will be qualified estimated (J). If the spike recovery grossly exceeds the criteria, results may be rejected (R) based on professional judgment.

Matrix Spike/
Matrix Spike Duplicate: Verify the method spike is within ± 4σ of the theoretical spike value. If the spike is outside criteria, then qualify the result estimated (J, UJ). If the spike recovery grossly exceeds the criteria, results may be rejected (R) based on professional judgment.

Field Duplicate: Verify that a field duplicate sample was taken for every 10 or fewer field samples. For water the duplicate result must be within 4σ of the result for the original. If this criterion is not met, qualify the results for that sample as estimated (J).

Decay Correction: If applicable, verify that the result was properly decayed to the sample date.

Replicate: Verify a replicated sample was run 1 per 20 samples of a similar matrix and that the result is within 4σ of the normalized range. If this criterion is not met, qualify both original and replicate samples as estimated (J).

Result: Verify the result is properly reported with its associated error. Also verify that data qualification letters are present if required.

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Check Source Forms

Complete header section. Complete remainder of table only for those samples which have check source outliers. If there are no check source outliers, then write 'None' on the form.

3.5.3 Background Summary Forms

Complete header section. Complete the remainder of the table only for those samples associated with background results outside criteria. If no samples are associated with background results outside criteria, then write 'None' on the form.

3.5.4 Method Spike Summary Forms

Complete header section. Complete the remainder of the table only for those samples associated with method spike recoveries outside criteria. If no method spikes are outside criteria, then write 'None' on the form.

3.5.5 Matrix Spike Recovery Forms

Complete header section. Complete remainder of table only for those samples which have MS/MSD outliers. Enter the percent recovery as appropriate. If no outliers, write 'None'.

3.5.6 Replicate Summary Forms

Complete header section. If outliers, enter sample IDs, outlier value, and qualification designation.

3.5.7 Field Duplicate Forms

Complete header section and sample IDs. Enter outlying RPD values for analytes. If no values are outside the required range, so state.

3.5.8 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for surrogate recovery, matrix spike recovery, calibration, field duplicate, and replicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- MS/MSD
- Method Spike

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.9 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

RADIOISOTOPES

- ☐ Table of Contents (Check List)
  - I. Case Narrative
    - ☐ Cover Page
    - ☐ Case Narrative
    - ☐ Cross Reference of Sample ID to Laboratory ID
    - ☐ Description of Data Qualifiers used in the Report
    - ☐ Applicable Laboratory SOP and Revision Date
    - ☐ Summary of Sample Results
    - ☐ Summary of Quality Control Results
    - ☐ Summary of Analysis and Preparation Dates
  - II. Raw Data
    - IIA. Sample Data
      - ☐ Example Calculation Page
      - ☐ Tritium Calculation and Result Sheet (Raw Data Part of Standards Data)
      - ☐ Gamma - Spectrometry (Results followed by Raw Data)
      - ☐ Alpha - Spectrometry (Results followed by Raw Data)
      - ☐ Sr-90 (Raw Data part of Standards Data)
      - ☐ Ra-226 (Raw Data part of Standards Data)
      - ☐ Am-241 (Raw Data part of Standards Data)
      - ☐ Sample Preparation / Extraction / Digestion Logs
      - ☐ Percent Solids Determination
      - ☐ Instrument Run Logs
    - IIB. Standards Data

      MONTHLY COMPILATION PROVIDED SEPARATELY
    - IIC. Raw QC Data
      - ☐ Quality Control Results (Blanks, MS/MSD, Replicate)
      - ☐ Quality Control Raw Data (Behind Each Result)
  - III. Sample Receipt
    - ☐ Traffic Reports
    - ☐ Sample Log In Record
  - ☐ IV. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I	-	Data Outlier Summary Forms
ATTACHMENT II	-	Qualified Data Summary Reports *
ATTACHMENT III	-	Data Completeness Checklist
ATTACHMENT IV	-	Laboratory Case Narrative
ATTACHMENT V	-	Chain-of-Custody

- Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Times Summary
Tritium Analyses

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
QC Limits: None	

Laboratory Sample Identification	Mound Sample Identification	Sample Date	Count Date	Days	Comments

Reviewer: _____

Check Source Summary
Tritium Analyses

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC Limits: ± 3σ from Mean Value

Laboratory Sample Identification	Mound Sample Identification	Count Date	Result	Comments

Reviewer: _____

Background Summary
Tritium Analyses

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC Limits: $\pm 3\sigma$ from Mean Value

Laboratory Sample Identification	Mound Sample Identification	Sample Count Date	Background Count Date	Comments

Reviewer: _____

Method Spike Summary
Tritium Analyses

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC Limits: ± 3σ of Known Value

Laboratory Sample Identification	Mound Sample Identification	Amount Added pCi/	Amount Found pCi/	Diff Std Dev	Comments

Reviewer: _____

Matrix Spike Summary
Tritium Analyses

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC Limits: ± 4σ of Known Value

Laboratory Sample Identification	Mound Sample Identification	Amount Added pCi/	Amount Found pCi/	Diff Std Dev	Comments

Reviewer: _____

Replicates Summary
Tritium Analyses

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC Limits: ± 4σ of Normalized Range

Laboratory Sample Identification	Mound Sample Identification	Orig Result pCi/	Rep Result pCi/	Diff Std Dev	Comments

Reviewer: _____

Field Duplicate Summary
Tritium Analyses

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC Limits: ± 4σ of Normalized Range

Laboratory Sample Identification	Mound Sample Identification	Orig Result pCi/	Rep Result pCi/	Diff Std Dev	Comments

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-015

Gamma Spectrometry Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for gamma spectrometry analysis. These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

DOE. "EML Procedures Manual", HASL-300, Environmental Measurements Laboratory, U.S. Department of Energy, 27th Edition.

ND9900 VAX/VMS Spectroscopy Application Package User's Manual (09-0196), Nuclear Data, Inc., Schaumberg, IL. August 1986.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, or two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, and two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory data package. Missing data is requested from the laboratory as soon as it is noted. If missing data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.
- Results of the equipment rinsate, ambient, sample bank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.
- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
N	Presumptive evidence of presence of material.
NJ	Presumptive evidence of the presence of the material at an estimated quantity.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Holding Time: Verify the samples were analyzed within the holding time specified in Method A-015 of the Methods Compendium. If holding time was exceeded, the sample results should be qualified as estimated (J) or (UJ).

Calibration: Verify each counter being used has been calibrated for each isotope of interest either by counting a standard of the isotope prepared in the same geometry as is used for samples or by calculation from an efficiency vs. energy calibration curve. The data reduction used must be the same as is used to calculate the sample data. This calibration should be done at least annually. Also verify that the correct branching ratio for each isotope was used in the calculations.

Mixed Standard: Verify a standard containing a mixture of isotopes covering the energy range of interest was counted and that the results obtained were within ± 5% of the known value.

Source Check: Verify the check source was counted each day and its value is within 3σ of its mean value for each counter used. If this criterion is not met, qualify all of the results measured, before the next acceptable check source count was obtained as estimated (J).

Background: Verify the background was counted for each counter at least once each month and the appropriate background was used to subtract from the sample counts.

Replicate: Verify a replicate sample was run 1 per 20 samples of a similar matrix and the result is within 4σ of the normalized range. If this criterion is not met, qualify both original and replicate samples as estimated (J).

Field Duplicate: Verify a field duplicate sample was taken for every 10 or fewer field samples. For water the duplicate result must be within 4σ of the result for the original. If this criterion is not met, qualify the results for that sample as estimated, (J).

Result: Verify the results are properly reported with their associated errors. This is generally 2σ based on counting statistics. Also verify that any data qualification letters are present.

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Check Source Forms

Complete header section. If samples were associated with check sources outside criteria, then complete the table. If no samples are associated with check sources outside criteria, then write 'None' on the form.

3.5.3 Background Summary Forms

Complete header section. Complete the remainder of the table only for those samples associated with background measurements outside criteria. If no samples are associated with background results outside criteria, then write 'None' on the form.

3.5.4 Replicate Summary Forms

Complete header section. If outliers, enter sample IDs, outlier value, and qualification designation.

3.5.5 Field Duplicate Summary Forms

Complete header section and sample IDs. Enter outlying standard deviation values for analytes. If no values are outside the required range, so state.

3.5.6 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for the field duplicate and replicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result.

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.7 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

RADIOISOTOPES

☐ Table of Contents (Check List)

I. Case Narrative
- ☐ Cover Page
- ☐ Case Narrative
- ☐ Cross Reference of Sample ID to Laboratory ID
- ☐ Description of Data Qualifiers used in the Report
- ☐ Applicable Laboratory SOP and Revision Date
- ☐ Summary of Sample Results
- ☐ Summary of Quality Control Results
- ☐ Summary of Analysis and Preparation Dates

II. Raw Data

IIA. Sample Data
- ☐ Example Calculation Page
- ☐ Tritium Calculation and Result Sheet (Raw Data Part of Standards Data)
- ☐ Gamma - Spectrometry (Results followed by Raw Data)
- ☐ Alpha - Spectrometry (Results followed by Raw Data)
- ☐ Sr-90 (Raw Data part of Standards Data)
- ☐ Ra-226 (Raw Data part of Standards Data)
- ☐ Am-241 (Raw Data part of Standards Data)
- ☐ Sample Preparation / Extraction / Digestion Logs
- ☐ Percent Solids Determination
- ☐ Instrument Run Logs

IIB. Standards Data

MONTHLY COMPILATION PROVIDED SEPARATELY

IIC. Raw QC Data
- ☐ Quality Control Results (Blanks, MS/MSD, Replicate)
- ☐ Quality Control Raw Data (Behind Each Result)

III. Sample Receipt
- ☐ Traffic Reports
- ☐ Sample Log In Record

☐ IV. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I - Data Outlier Summary Forms

ATTACHMENT II - Qualified Data Summary Reports *

ATTACHMENT III - Data Completeness Checklist

ATTACHMENT IV - Laboratory Case Narrative

ATTACHMENT V - Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
Gamma Analyses

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
QC Limits: None	

Laboratory Sample Identification	Mound Sample Identification	Sample Date	Count Date	Days	Comments

Reviewer: _____

Check Source Summary
Gamma Analyses

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
QC Limits: ± 3σ from Mean Value	

Laboratory Sample Identification	Mound Sample Identification	Sample Count Date	Result	Comments

Reviewer: _____

Background Summary
Gamma Analyses

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC Limits: Counted within one week of sample.
No peaks identified with ± 20% error.

Laboratory Sample Identification	Mound Sample Identification	Sample Count Date	Background Count Date	Comments

Reviewer: _____

Replicates Summary
Gamma Analyses

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC Limits: ±4 σ of Normalized Range

Laboratory Sample Identification	Mound Sample Identification	Orig Result pCi/	Rep Result pCi/	Diff Std Dev	Comments

Reviewer: _____

Field Duplicate Summary
Gamma Analyses

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC Limits: ±4 σ of Normalized Range

Laboratory Sample Identification	Mound Sample Identification	Orig Result pCi/	Rep Result pCi/	Diff Std Dev	Comments

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-018

Volatile Organic Analysis (VOA)/Method SW8030 Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for acetonitrile and acrylonitrile analysis. These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

EPA. 1986. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. November 1986.

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation:

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory data package. Missing data is requested from the laboratory as soon as it is noted. If missing data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.

- Results of the equipment rinsate, ambient, sample bank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.

- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
N	Presumptive evidence of presence of material.

NJ	Presumptive evidence of the presence of the material at an estimated quantity.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration
H	Holding time exceeded
S	Qualified due to matrix spike recovery
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Holding Time: Verify that the samples were analyzed within the holding time period of 14 days. Qualify results as estimated with (J) and (UJ) if the holding time is exceeded. If the holding time was grossly exceeded professional judgment should be used to determine whether to qualify the data as unusable (R).

Calibration: Verify that the initial 5 point calibrations had a relative standard deviation (%RSD) < 20%. Verify that a continuing calibration check was analyzed every 10 samples and that its response was within 15% of true value. If any samples were analyzed with a non-compliant calibration standard, then the results associated with the non-compliant standard must be estimated (J). If %D or %RSD was > 50%, then all positive and non-detected values for that analyte must be estimated (J) and (UJ).

Verify retention time windows: $\pm 3\sigma$ of three retention times for each analyte as per SW846.

Blanks: Method blanks must be analyzed one per 20 samples of a given matrix per day and contamination must be less than PQL. Verify

that equipment blanks are performed one per 10 samples; sample bank blanks and ambient blanks are performed one per 20 samples. These field blanks are qualified using qualification guidance levels derived form method blank contamination. Qualification level for samples are determined from the maximum contamination levels in method and equipment blank contamination.

Qualification Guidance Level = 5x contamination in blank.

Qualification Guidelines:

- If results <PQL, report PQL as U.
- If results >PQL and < 5*x* contamination, then report the value as U.
- If results >PQL and > 5*x* contamination, then report the value unqualified.

Laboratory Control Sample (LCS):

Verify that an LCS was performed 1 per 20 samples of a given matrix per day. Verify that LCS recoveries are within QC limits:

acrylonitrile	*(70 to 135%)*
acetonitrile	*(88 to 118%)*

If LCS recoveries are not within QC limits, then all associated samples must be re-extracted with another LCS. Professional judgment should be used to evaluate data associated with failed LCS analysis. If recoveries are below 10%, then associated results could be considered as unusable (R).

Matrix Spike (MS)/ Matrix Spike Duplicate(MSD):

Verify that an MS/MSD was performed one per 20 samples of a given matrix. Verify that MS/MSD recoveries are within QC limits:

acrylonitrile	*(70 to 135%)*
acetonitrile	*(70 to 136%)*

RPD (≤ 15%)

The positive values for those compounds that fail MS/MSD criteria should be estimated in the unspiked sample. If recoveries are below 10%, then associated results could be considered as unusable (R).

Field Duplicate: Verify that a field duplicate sample was analyzed for every 10 field samples or less of each matrix. The RPD must be ≤ 35% for waters. If a criterion was not met, then estimate (J) the positive results for that analyte(s) in both samples. If one of the sample results is > PQL and the other is non-detected, then both results are estimated (J,UJ).

Compound Quantitation Limits: Verify that the following quantitation limits were met:

Analyte	Water	Soil
acetonitrile	10 ug/L	100 ug/Kg
acrylonitrile	10 ug/L	100 ug/Kg

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Surrogate Recovery Forms

No surrogates are currently required. Complete header section. If a surrogate is used, the form may be used to document a problem. If no outliers, then write 'None'.

3.5.3 Matrix Spike Recovery Forms

Complete header section. Complete remainder of table only for those samples which have MS/MSD outliers. Enter the percent recovery as appropriate. The RPD should be entered in parentheses. If no outliers, write 'None'.

3.5.4 Blank Data Summary Forms

Complete the appropriate form from Attachment III. Enter blank outliers only and note appropriate action level based on 5*x* or 10*x*. Enter the associated sample IDs.

3.5.5 Calibration Forms

Complete header section, instrument, and date and time for each calibration run. Enter the outlying RF, RRF, RSD, or %D as necessary. List all associated sample IDs at bottom of form.

3.5.6 Field Duplicate Forms

Complete header section and sample IDs. Enter outlying RPD values for analytes. If no values are outside the required range, so state.

3.5.7 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for the matrix spike recovery, calibration, field duplicate, and replicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- Initial calibration - at least one RRF and %RSD.
- Continuing calibration - at least one RRF and %D.
- Surrogate - at least one set of surrogates from one sample.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.8 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC ANALYSIS

- ☐ Table of Contents (Check List)
  - I. Case Narrative
    - ☐ Cover Page
    - ☐ Case Narrative
    - ☐ Cross Reference of Sample ID to Laboratory ID
    - ☐ Description of all Qualifiers used by the Laboratory
    - ☐ Applicable Laboratory SOP and Revision Date
  - II. QC Summary
    - ☐ Surrogate Recoveries Summary
    - ☐ Matrix Spike/Matrix Spike Duplicate Summary
    - ☐ Method Blank Summary
    - ☐ Laboratory Control Sample
  - III. Raw Data
    - IIIA. Sample Data
      - ☐ Example Calculation
      - ☐ Target Compound Results
      - ☐ Chromatograms
      - ☐ Quantitation / Calculation of Concentrations
    - IIIB. Standard(s) Data
      - ☐ Initial Calibration Data
      - ☐ Standard(s) Chromatograms and Quantitation Reports
      - ☐ Continuing Calibration
      - ☐ Standard(s) Chromatograms and Quantitation Reports

DATA PACKAGE COMPLETENESS CHECKLIST (Con't)
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC ANALYSIS

IIIC. Raw QC Data

- ☐ Blank Data
  - ☐ Chromatogram
  - ☐ Quantitation / Calculation of Concentrations
- ☐ Matrix Spike Data
  - ☐ Tabulated Results
  - ☐ Chromatogram(s)
- ☐ Matrix Spike Duplicate Data
  - ☐ Tabulated Results
  - ☐ Chromatogram(s)
- ☐ Instrument Settings
- ☐ Instrument Run Logs
- ☐ Percent Solids Determination Logs
- ☐ Sample Preparation/Extraction Logs

IV. Sample Receipt

- ☐ Traffic Reports
- ☐ Sample Log In Record

☐ V. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I - Data Outlier Summary Forms

ATTACHMENT II - Qualified Data Summary Reports *

ATTACHMENT III - Data Completeness Checklist

ATTACHMENT IV - Laboratory Case Narrative

ATTACHMENT V - Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
Volatile Organics - Method 8030

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Laboratory Sample Identification	Mound Sample Identification	Date Collected	Date Extracted	Date Analyzed	Number of Days Past Holding Time

Holding Time: Analysis within 14 days from collection

Reviewer: _____

Surrogate Recovery Outliers
Volatile Organics - Method 8030

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Soil Quality Control Limits - None Specified	**Water Quality Control Limits** - None Specified

Laboratory Sample Id	Mound Sample Id	Sample Matrix			

Use the laboratory acceptance window (do not reject unless the recovery is less than 10%, per Weston Mound).

Reviewer: _____

Matrix Spike Recovery Outliers
Volatile Organics - Method 8030

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Soil Quality Control Limits

-	Acrylonitrile	N/A
-	Acetoonitrile	N/A

Water Quality Control Limits

-	Acrylonitrile	(70 - 135%)	(15%)
-	Acetonitrile	(70 - 136%)	(15%)

Laboratory Sample Id	Mound Sample Id	Sample Matrix	Acrylonitrile	Acetonitrile

Reviewer: _____

Blank Data Summary Table
Volatile Organics - Method 8030

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

ANALYTE	Method Blank ID	Method Blank ID	Trip Blank ID	Ambient Blank ID	Equip. Blank ID	Action Level
Acrylonitrile						
Acetonitrile						
Applies to Samples:						

Reviewer: _____

Calibration Outliers
Volatile TCL Compounds - Method 8030

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Instrument ID:	Matrix (Soil/Water):

	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
ANALYTE		RSD < 20%	*		D ± 15%	*		D ± 15%	*
Acrylonitrile									
Acetonitrile									
Affected Samples: (Client ID)									

Was a continuing calibration run every 10 samples? ☐ Yes ☐ No
Were retention times acceptable? ☐ Yes ☐ No

* These flags should be applied to the analytes on the sample data sheets.

Reviewer: _____

Field Duplicate Data Summary Table
Volatile Organics - Non-CLP Method 8030

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC LIMITS
Water = ≤ 35% RPD Soil = NA

ANALYTE	Mound Sample ID	Mound Sample ID	Mound Sample ID	Mound Sample ID
Acrylonitrile				
Acetonitrile				

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-019

Hexavalent Chromium Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for hexavalent chromium analysis. These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B, and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1987. Data Quality Objectives for Remedial Response Activities. U.S. Environmental Protection Agency. Office of Emergency and Remedial Response and Office of Waste Programs Enforcement. Report No. EPA 540/G-87/003. Washington, D.C. March 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B, and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory

data package. Missing data is requested from the laboratory as soon as it is noted. If missing data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.
- Results of the equipment rinsate, ambient, sample blank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.
- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
N	Presumptive evidence of presence of material.

NJ	Presumptive evidence of the presence of the material at an estimated quantity.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration
H	Holding time exceeded
S	Qualified due to matrix spike recovery
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Holding Time: Verify the samples were prepared and analyzed within the holding time specified in Method A-019 of the Methods Compendium. If the holding time is exceeded, qualify results as estimated (J, UJ). If the holding time was grossly exceeded, professional judgment may be used to qualify the data as unusable (R).

Calibration: Verify the 5-point initial calibration curve had a correlation coefficient ≥ 0.995. If the correlation coefficient is less than 0.995, then qualify all results estimated (J, UJ). If the correlation coefficient was grossly outside criteria, then reject (R) all results.

Verify the initial calibration verification (ICV) and continuing calibration verification are analyzed within the frequency specified in Method A-019 of the Methods Compendium and the results are within ± 10% of the true value. If the criteria for a calibration verification is not met, then qualify the associated sample data estimate (J, UJ). If the criteria is grossly exceeded, professional judgment must be applied to determine whether the data should be rejected (R).

Blanks:

Verify blanks have been analyzed at the frequency specified in Method A-019 of the Methods Compendium. If hexavalent chromium is detected in a blank, then apply the 5*x* rule.

Qualification Guidelines:

- If results < PQL, then no action is taken.
- If results < 5*x* the amount of hexavalent chromium in the blank, then qualify the result nondetect (U).
- If results > 5*x* the amount of hexavalent chromium in the blank, then apply no qualification.

Matrix Spikes:

Recoveries and relative percent differences should meet the requirements specified in Method A-019 of the Methods Compendium.

The positive sample results associated with an MS/MSD outside criteria, should be qualified estimated (J). If the MS/MSD recovery is extremely low (< 10%), then the positive result should be qualified rejected (R).

For a non-detect sample result associated with an MS/MSD recovery above acceptance criteria, no qualification is required. If the MS/MSD recovery associated with a non-detect result is less than 10%, the result should be qualified rejected (R). If the MS/MSD recovery associated with a sample result is less than acceptance limits and greater than 10%, the result should be qualified estimated (J, UJ).

Laboratory Control Sample:

If a laboratory control sample fails to meet acceptance criteria, the associated sample data should be qualified estimated (J, UJ). If the recovery is extremely poor or there are extenuating circumstances, the validator will use professional judgment to assign data qualifications.

Duplicate/Replicate:

Verify a field duplicate sample was analyzed for every 10 field samples of the same matrix. If the relative percent difference is greater than 25%, then qualify the associated data estimated (J, UJ). If one of the results is non-detect and the other result is greater than the PQL, then both the results should be estimated (J, UJ).

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Anion Data Summary Forms

Complete header section. Fill in remainder if criteria was exceeded.

3.5.3 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for surrogate recovery, matrix spike recovery, calibration, field duplicate, and replicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- Initial calibration - correlation coefficient is correct.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.4 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

HEXAVALENT CHROMIUM

- ☐ Table of Contents (Check List)
  - I. Case Narrative
    - ☐ Cover Page
    - ☐ Case Narrative
    - ☐ Cross Reference of Sample ID to Laboratory ID
    - ☐ Description of all Qualifiers used by the Laboratory
    - ☐ Applicable Laboratory SOP and Revision Date
    - ☐ Summary of Sample Results
    - ☐ Summary of Quality Control Results
    - ☐ Summary of Analysis and Preparation Dates
  - II. Raw Data
    - IIA. Sample Data
      - ☐ Example Calculation Sheet
      - ☐ Instrument Settings
      - ☐ Analytical Raw Data (Behind Each Result)
    - IIB. Standards Data
      - ☐ Standards Preparation Data
      - ☐ Initial and Continuing Calibration
    - IIC. Raw QC Data
      - ☐ Quality Control Results (Blanks, LCS, MS/MSD, Replicate)
      - ☐ Quality Control Raw Data (Behind Each Result)
      - ☐ Instrument Run Logs / Instrument Calibration / Tuning
      - ☐ Sample Preparation / Extraction / Digestion Logs
      - ☐ Percent Solids Determination Logs
  - III. Sample Receipt
    - ☐ Traffic Reports
    - ☐ Sample Log In Record
  - ☐ IV. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I - Data Outlier Summary Forms

ATTACHMENT II - Qualified Data Summary Reports *

ATTACHMENT III - Data Completeness Checklist

ATTACHMENT IV - Laboratory Case Narrative

ATTACHMENT V - Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
Hexavalent Chromium

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Laboratory Sample Identification	Mound Sample Identification	Date Collected	Date Analyzed	Number of Days Past Holding Time

Reviewer: _____

Data Summary Table
Hexavalent Chromium

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Instrument ID For:	

	Calibration		Blanks		(≤10 PQL)
	Initial	Continuing	Method ID	Method ID	Method ID
ANALYTE	COEFF > 0.995	% D < 10			
Chromium					
Applies to Samples (Client ID):					

Matrix Spike/Matrix Spike Duplicate / LCS

Sample ID	Matrix	Chromium MS/MSD		Chromium LCS
		85 - 115% R	% RPD	80 - 120%

Field Duplicates **(< 25% RPD)**

ANALYTE	Sample ID	Sample ID	Sample ID	Sample ID
Chromium				

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-020

Volatile Organic Analysis (VOA)/Method SW8020 Data Validation

(includes the criteria for SW8240/SW8260)

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: Compendium (February 1997)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for Method 8020 analysis. These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

EPA. 1986. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. November 1986.

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory

data package. Missing data is requested from the laboratory as soon as it is noted. If missing data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.
- Results of the equipment rinsate, ambient, sample blank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.
- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- Initial calibration - at least one RRF and %RSD.
- Continuing calibration - at least one RRF and %D.
- Surrogate - at least one surrogate from one sample.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
N	Presumptive evidence of presence of material.

NJ	Presumptive evidence of the presence of the material at an estimated quantity.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration
H	Holding time exceeded
K	Qualified due to surrogate recovery
S	Qualified due to matrix spike recovery
I	Qualified due to internal standard
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Holding Time: Verify the samples were extracted within the holding time specified in Method A-020 of the Methods Compendium. Qualify results as estimated (J, UJ) if the holding time was exceeded. If the holding time was grossly exceeded, professional judgment may be used for qualifying the data as unusable (R).

Calibration (GC only): Verify the Percent Relative Standard Deviation (RSD) of the relative response factors for each of the four targets was less than 20 percent. If the RSD > 20 percent and the RSD < 50 percent then estimate (J, UJ) both positive and negative results. If the RSD > 50 percent, then reject the results.

Verify a continuing calibration check (CCC) was introduced after every 10 samples. If the CCC was not run, then qualify the associated results estimated (J, UJ). If the CCC's exceed 15% difference, then qualify the results estimated (J, UJ).

Calibration (GC/MS only):

Verify the Percent Relative Standard Deviation (RSD) for each of the four targets was less than 30 percent. If the RSD > 30 percent and the RSD < 50 percent then estimate (J, UJ) both positive and negative results. If the RSD > 50 percent, then reject the results.

Verify the (GC/MS) Relative Response Factor (RRF) for each of the target compounds is greater than 0.05. If the RRF < 0.05, then reject both positive and negative results.

Verify a continuing calibration check was run every 12 hours. If the continuing calibration check was not run, then qualify all affected results estimated (J, UJ).

Continuing Calibration Qualification Criteria:

- If the percent difference is greater than 25%, qualify the associated positive analyte result estimated (J).
- If the percent difference is between -25 and -50%, then qualify both positive or negative associated analyte result estimated (J, UJ).
- If the negative percent difference is greater than -50%, then reject (R) the associated negative analyte result, or qualify the associated positive analyte result estimated (J).

Note: The laboratory is using BTEX compounds for SPCC's and CCC's.

Blanks:

A method blank must be analyzed for each batch of samples analyzed and the contamination must be less than the PQL. In addition to the laboratory method blank, field blanks and trip blanks will be submitted for testing and the contamination must be less than the PQL. If the method blanks contain target contaminants, the action level will be based on the largest result reported in the three blanks for each compound. The action level will be based on the 5*x* rule. Sample results less than 5 times the blank contaminant level will be qualified non-detect (U) at the reported value.

- If the sample result < PQL, then no action is taken.

- If the sample result > PQL and less than 5*x* contaminant level, then report the value with a 'U' qualifier.
- If the sample result > PQL and greater than 5*x* contaminant level, then no action is taken.

Internal Standards (GC/MS only):

Internal standards must be added to each sample prior to analysis. If the internal standard is below criteria, then the associated positive sample results will be estimated (J). If the internal standard is above criteria, then the associated positive and negative sample results will be estimated (J, UJ). If the internal standard criteria are grossly exceeded, then professional judgment may be used to reject data results.

Matrix Spike:

Recoveries and relative percent differences should correspond to those specified in Method A-020 of the Methods Compendium for soils and water.

If the MS/MSD recovery is below acceptance units and greater than 10%, then qualify associated sample results estimated (J, UJ).

If the MS/MSD recovery is greater than criteria, then qualify the associated positive sample results estimated.

If the recovery is less than 10%, then qualify positive results estimated and non-detect results rejected (R).

For RPD's which exceed criteria, qualify positive results estimated (J).

Laboratory Control Sample:

Laboratory control sample (LCS) recoveries must be within QC limits or all associated samples must be re-extracted with another LCS. If the criteria was not met, then:

- If the recovery is below criteria and greater than 10 percent, qualify positive results estimated (J).
- If the recovery is less than or equal to 10 percent, then qualify all results unusable (R).
- For recoveries greater than the specified criteria, no action is taken for non-detects and positive results will be qualified estimated, (J). For recoveries that grossly exceed the specified

criteria, positive results may be qualified unusable, (R), based on the validator's professional judgment.

Surrogates: If surrogate recoveries do not meet requirements as found in Method A-020 of the Methods Compendium, then the samples should be re-analyzed. If the surrogate recovery is below criteria and greater than 50 percent, then associated positive and negative results will be qualified estimated (J, UJ). If the percent recovery is less than 50 percent, then associated negative results will be rejected (R) and positive results will be qualified estimated. If the surrogate recovery exceeds criteria, then associated positive sample results will be qualified estimated (J).

Field Duplicate: Verify a field duplicate sample was analyzed for every 10 field samples or less of each matrix. The RPD must be ≤ 25% for water and ≤ 50% for soils. If criteria was not met, then estimate (J) the positive results for that analyte(s) in both samples. If one of the sample results is greater than the PQL and the other is non-detected, then both results are estimated (J, UJ).

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Surrogate Recovery Forms

Complete header section. Complete remainder of table only for those samples which have surrogate outliers. If no outliers, then write 'None'.

3.5.3 Matrix Spike Recovery Forms

Complete header section. Complete remainder of table only for those samples which have MS/MSD outliers. Enter the percent recovery as appropriate. If no outliers, write 'None'.

3.5.4 Blank Data Summary Forms

Complete the appropriate form from Attachment III. Enter blank outliers only and note appropriate action level based on 5*x* or 10*x*. Enter the associated sample IDs.

3.5.5 Calibration Forms

Complete header section, instrument, and date and time for each calibration run. Enter the outlying RF, RRF, RSD, or %D as necessary. List all associated sample IDs at bottom of form.

3.5.6 Internal Standard Forms

Complete header section. In area limits box, write actual area values from Form VIII. For IS outliers, enter the sample ID and enter the actual outlying area value.

3.5.7 Field Duplicate Forms

Complete header section and sample IDs. Enter outlying RPD values for analytes. If no values are outside the required range, so state.

3.5.8 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for surrogate recovery, matrix spike recovery, calibration, field duplicate, and replicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- Initial calibration - at least one RRF and %RSD.
- Continuing calibration - at least one RRF and %D.
- Surrogate - at least one surrogate from one sample.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.9 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC ANALYSIS

- ☐ Table of Contents (Check List)
  - I. Case Narrative
    - ☐ Cover Page
    - ☐ Case Narrative
    - ☐ Cross Reference of Sample ID to Laboratory ID
    - ☐ Description of all Qualifiers used by the Laboratory
    - ☐ Applicable Laboratory SOP and Revision Date
  - II. QC Summary
    - ☐ Surrogate Recoveries Summary
    - ☐ Matrix Spike/Matrix Spike Duplicate Summary
    - ☐ Method Blank Summary
    - ☐ Laboratory Control Sample
  - III. Raw Data
    - IIIA. Sample Data
      - ☐ Example Calculation
      - ☐ Target Compound Results
      - ☐ Chromatograms
      - ☐ Quantitation / Calculation of Concentrations
    - IIIB. Standard(s) Data
      - ☐ Initial Calibration Data
      - ☐ Standard(s) Chromatograms and Quantitation Reports
      - ☐ Continuing Calibration
      - ☐ Standard(s) Chromatograms and Quantitation Reports

DATA PACKAGE COMPLETENESS CHECKLIST (Con't)
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC ANALYSIS

IIIC. Raw QC Data

- ☐ Blank Data
  - ☐ Chromatogram
  - ☐ Quantitation / Calculation of Concentrations
- ☐ Matrix Spike Data
  - ☐ Tabulated Results
  - ☐ Chromatogram(s)
- ☐ Matrix Spike Duplicate Data
  - ☐ Tabulated Results
  - ☐ Chromatogram(s)
- ☐ Instrument Settings
- ☐ Instrument Run Logs
- ☐ Percent Solids Determination Logs
- ☐ Sample Preparation/Extraction Logs

IV. Sample Receipt

- ☐ Traffic Reports
- ☐ Sample Log In Record

☐ V. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC/MS VOLATILES ORGANIC COMPOUNDS (VOCs)

- ☐ Table of Contents (Check List)
  - I. Case Narrative
    - ☐ Cover Page
    - ☐ Case Narrative
    - ☐ Cross Reference of Sample ID to Laboratory ID
    - ☐ Description of all Qualifiers used by the Laboratory
    - ☐ Applicable Laboratory SOP and Revision Date
  - II. QC Summary
    - ☐ System Monitoring Compound Summary (Form II VOA)
    - ☐ Matrix Spike/Matrix Spike Duplicate Summary (Form III VOA)
    - ☐ Method Blank Summary (Form IV VOA)
    - ☐ GC / MS Instrument Performance Check (Form V VOA)
    - ☐ Internal Standard Area and RT Summary (Form VIII VOA)
    - ☐ Laboratory Control Sample
  - III. Raw Data
    - IIIA. Sample Data
      - ☐ Example Calculation
      - ☐ Target Compound Results - Organic Analysis Data Sheet (Form I VOA)
      - ☐ Tentatively Identified Compounds (Form I VOA - TIC)
      - ☐ Reconstructed Total Ion Chromatograms (RIC)
      - ☐ Quantitation Report
      - ☐ Raw Spectra and Background - Subtracted Mass Spectra of Target Compounds Identified
      - ☐ GC / MS Library Search Spectra for TIC (3 Best Library Matches)
      - ☐ Quantitation / Calculation of TIC Concentrations
    - IIIB. Standard(s) Data
      - ☐ Initial Calibration Data (Form VI VOA)
      - ☐ VOA Standard(s) Reconstructed Ion Chromatograms and Quantitation Reports
      - ☐ Continuing Calibration (Form VII VOA)
      - ☐ VOA Standard(s) Reconstructed Ion Chromatograms and Quantitation Reports

DATA PACKAGE COMPLETENESS CHECKLIST (Con't)
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC/MS VOLATILES ORGANIC COMPOUNDS (VOCs)

IIIC. Raw QC Data

- ☐ BFB / Bar Graph Spectrum / Mass Listing
- ☐ Blank Data
  - ☐ Reconstructed Total Ion Chromatogram and Quantitation Reports
  - ☐ Target Compound Spectra
  - ☐ GC / MS Library Search Spectra for TIC
  - ☐ Quantitation / Calculation of TIC Concentrations
- ☐ Matrix Spike Data
  - ☐ Tabulated Results (Form I VOA)
  - ☐ Reconstructed Ion Chromatogram(s)
- ☐ Matrix Spike Duplicate Data
  - ☐ Tabulated Results (Form I VOA)
  - ☐ Reconstructed Ion Chromatogram(s)
- ☐ Instrument Settings
- ☐ Instrument Run Logs
- ☐ Percent Solids Determination Logs

IV. Sample Receipt

- ☐ Traffic Reports
- ☐ Sample Log In Record

☐ V. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I	-	Data Outlier Summary Forms
ATTACHMENT II	-	Qualified Data Summary Reports *
ATTACHMENT III	-	Data Completeness Checklist
ATTACHMENT IV	-	Laboratory Case Narrative
ATTACHMENT V	-	Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
BTEX Analysis - SW8020 / SW8240 / SW8260

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Laboratory Sample Identification	Mound Sample Identification	Date Collected	Date Analyzed	Number of Days Past Holding Time

Reviewer: _____

Surrogate Recovery Outliers
BTEX Analysis - SW8020

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Soil Quality Control Limits - Fluorobenzene (80 - 120%) GC only	**Water Quality Control Limits** - Fluorobenzene (80 - 120%) GC only

Laboratory Sample Id	Mound Sample Id	Sample Matrix	Fluorobenzene		

Reviewer: _____

Surrogate Recovery Outliers
BTEX Analysis - SW8240

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Soil Quality Control Limits				Water Quality Control Limits			
-	Toluene-d8	(84 - 138%)	GC/MS only	-	Toluene-d8	(88 - 110%)	GC/MS only
-	Bromofluorobenzene	(59 - 113%)	GC/MS only	-	Bromofluorobenzene	(86 - 115%)	GC/MS only
-	1,2-Dichloroethane	(70 - 121%)	GC/MS only	-	1,2-Dichloroethane	(76 - 114%)	GC/MS only

Laboratory Sample Id	Mound Sample Id	Sample Matrix	Toluene-d8	Bromofluorobenzene	1,2-Dichloroethane-d4

Reviewer: _____

Matrix Spike Recovery Outliers
BTEX Analysis - SW8020 / SW8240 / SW8260

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Soil Quality Control Limits			Water Quality Control Limits		
-	Benzene	(80 - 120%/ RPD<30%)	-	Benzene	(80 - 120%/ RPD<15%)
-	Toluene	(80 - 120%/ RPD<30%)	-	Toluene	(80 - 120%/ RPD<15%)
-	Ethylbenzene	(80 - 120%/ RPD<30%)	-	Ethylbenzene	(80 - 120%/ RPD<15%)
-	Xylenes	(80 - 120%/ RPD<30%)	-	Xylenes	(80 - 120%/ RPD<15%)

Laboratory Sample Id	Mound Sample Id	Sample Matrix	Benzene			Toluene			Ethylbenzene			Xylenes		
			%R	%R	RPD	%R	%R	RPD	%R	%R	RPD	%R	%R	RPD

Reviewer: _____

Blank Data Outlier Summary Table
BTEX Analysis - SW8020 / SW8240 / SW8260

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

ANALYTE	Method Blank ID	Method Blank ID	Trip Blank ID	Ambient Blank ID	Equip. Blank ID	Action Level
Benzene						
Toluene						
Ethylbenzene						
Xylene (Total)						
Affected Samples:						

Reviewer: _____

Calibration Summary
BTEX Analysis - SW8020

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Instrument ID:	

ANALYTE	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
		RSD < 20%			%D < 15%			%D < 15%	
Benzene									
Toluene									
Ethylbenzene									
Xylene (Total)									

Affected Samples: (Client ID)			

Was a Continuing Calibration check run every 10 samples? ☐ Yes ☐ No

Reviewer: _____

Calibration Summary
BTEX Analysis - SW8240 / SW8260

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Instrument ID:

ANALYTE	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
	RRF > 0.05	RSD < 30%		RRF > 0.05	%D < 25%		RRF > 0.05	%D < 25%	
Benzene									
Toluene									
Ethylbenzene									
Xylene (Total)									

Affected Samples: (Client ID)			

Was a Continuing Calibration check run every 12 hours? ☐ Yes ☐ No

Reviewer: _____

Internal Standard Recovery Outliers
BTEX Analysis - SW8240 / SW8260 (GC/MS only)

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Quality Control Limits: Area: - 50% to + 100% RT: ± 30 seconds	**Internal Standard**	**Daily Standard Area**	**Lower Area Limit (- 50%)**	**Upper Area Limit (+ 100%)**
	Bromochloromethane			
	1,4-Difluorobenzene			
	Chlorobenzene-d5			

Laboratory Sample ID	**Mound Sample ID**	**Sample Matrix**	**Bromochloromethane**	**1,4-Difluorobenzene**	**Chlorobenzene**

Not required for method SW8020

Reviewer: _____

Field Duplicate Data Summary Table
BTEX Analysis - SW8020 / SW8240 / SW8260

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Field Sample Identification	
Field Duplicate Identification	

ANALYTE	Field Sample Result	Duplicate Result	RPD
Benzene			
Toluene			
Ethylbenzene			
Xylene (Total)			

Was a field duplicate sample submitted for every 10 field samples of each matrix? ☐ Yes ☐ No

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-021

Volatile Organic Analysis (VOA) by EPA 601 Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: Compendium (February 1997)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for volatile organic anlaysis (VOA) by EPA Method 601 analysis for Benzene, Toluene, Ethylbenzene, and Xylenes only. These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

EPA 1983. "Methods for Chemical Analysis of Water and Wastes", U.S. Environmental Protection Agency, EPA-600/4-79-020, March 1983.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory data package. Missing data is requested from the laboratory as soon as it is noted. If missing data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.

- Results of the equipment rinsate, ambient, sample blank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.

- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- Initial calibration - at least one %RSD.
- Continuing calibration - at least one %D.
- Surrogate - at least one set of surrogates from one sample.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration

H	Holding time exceeded
K	Qualified due to surrogate recovery
S	Qualified due to matrix spike recovery
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Holding Time: Verify the samples were extracted within the holding time specified in Method A-021 of the Methods Compendium. Qualify results as estimated (J, UJ) if the holding time was exceeded. If the holding time was grossly exceeded, professional judgment may be used for qualifying the data as unusable (R).

Calibration: Verify the Percent Relative Standard Deviation (RSD) of the response factors for each of the four was less than 10 percent. If the RSD is greater than 10 percent and the RSD is less than 50 percent then estimate (J, UJ) both positive and negative results. If the RSD is greater than 50 percent, then reject the results.

Verify a continuing calibration check was run every 10 samples. If the continuing calibration check was not run, then qualify all affected results estimated (J, UJ). If the analyte response percent difference for each of the target analytes is greater than 10 percent from the initial response, then qualify the associated positive sample result estimated (J).

Blanks: A method blank must be analyzed for each batch of samples analyzed and the contamination must be less than the PQL. In addition to the laboratory method blank, field blanks and trip blanks will be submitted for testing and the contamination must be less than the PQL. If the method blanks contain target contaminants, the action level will be based on the largest result reported in the three blanks for each compound. The action level will be based on the *5x* rule. Sample results less than *5x* the blank contaminant level will be qualified non-detect (U) at the reported value.

- If the sample result < PQL, then no action is taken.
- If the sample result > PQL and less than 5*x* contaminant level, then report the value with a (U) qualifier.
- If the sample result > PQL and greater than 5*x* contaminant level, then no action is taken.

Surrogates: If surrogate recoveries do not meet requirements as found in Method A-021 of the Methods Compendium, then the samples should be re-analyzed. If the surrogate recovery is below criteria and greater than 50 percent, then associated positive and negative results will be qualified estimated (J, UJ). If the percent recovery is less than 50 percent, then associated negative results will be rejected (R) and positive results will be qualified estimated. If the surrogate recovery exceeds criteria, then associated positive sample results will be qualified estimated (J).

Matrix Spike: Recoveries and relative percent differences should correspond to those specified in Method A-021 of the Methods Compendium for soils and water.

If the MS/MSD recovery is below acceptance units and greater than 10%, then qualify associated sample results estimated (J, UJ).

If the MS/MSD recovery is greater than criteria, then qualify the associated positive sample results estimated.

If the recovery is less than 10%, then qualify positive results estimated and non-detect results rejected (R).

For RPD's which exceed criteria, qualify positive results estimated (J).

Laboratory Control Sample: Laboratory control sample (LCS) recoveries must be within QC limits or all associated samples must be re-extracted with another LCS. If the criteria was not met, then:

- If the recovery is below criteria and greater than 10 percent, qualify positive results estimated (J).
- If the recovery is less than or equal to 10 percent, then qualify all results unusable, (R).

- For recoveries greater than the specified criteria, no action is taken for non-detects and positive results will be qualified estimated, (J). For recoveries that grossly exceed the specified criteria, positive results may be qualified unusable, (R), based on the validator's professional judgment.

Field Duplicate: Verify a field duplicate sample was analyzed for every 10 field samples or less of each matrix. The RPD must be ≤ 25% for water. If criteria was not met, then estimate (J) the positive results for that analyte(s) in both samples. If one of the sample result is greater than PQL and the other is non-detected, then both results are estimated.

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Surrogate Recovery Forms

Complete header section. Complete remainder of table only for those samples which have surrogate outliers. If no outliers, then write 'None'.

3.5.3 Matrix Spike Recovery Forms

Complete header section. Complete remainder of table only for those samples which have MS/MSD outliers. Enter the percent recovery as appropriate. If no outliers, write 'None'. The matrix spike form should also be used for the laboratory control sample recovery.

3.5.4 Blank Data Summary Forms

Complete the appropriate form from Attachment III. Enter blank outliers only and note appropriate action level based on $5x$ or $10x$. Enter the associated sample IDs.

3.5.5 Calibration Forms

Complete header section, instrument, and date and time for each calibration run. Enter the outlying RF, RRF, RSD, or %D as necessary. List all associated sample IDs at bottom of form.

3.5.6 Field Duplicate Forms

Complete header section and sample IDs. Enter outlying RPD values for analytes. If no values are outside the required range, so state.

3.5.7 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for surrogate recovery, matrix spike recovery, calibration, and field duplicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- Initial calibration - at least one %RSD.
- Continuing calibration - at least one %D.
- Surrogate - at least one set of surrogates from one sample.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.8 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC ANALYSIS

- ☐ Table of Contents (Check List)
  - I. Case Narrative
    - ☐ Cover Page
    - ☐ Case Narrative
    - ☐ Cross Reference of Sample ID to Laboratory ID
    - ☐ Description of all Qualifiers used by the Laboratory
    - ☐ Applicable Laboratory SOP and Revision Date
  - II. QC Summary
    - ☐ Surrogate Recoveries Summary
    - ☐ Matrix Spike/Matrix Spike Duplicate Summary
    - ☐ Method Blank Summary
    - ☐ Laboratory Control Sample
  - III. Raw Data
    - IIIA. Sample Data
      - ☐ Example Calculation
      - ☐ Target Compound Results
      - ☐ Chromatograms
      - ☐ Quantitation / Calculation of Concentrations
    - IIIB. Standard(s) Data
      - ☐ Initial Calibration Data
      - ☐ Standard(s) Chromatograms and Quantitation Reports
      - ☐ Continuing Calibration
      - ☐ Standard(s) Chromatograms and Quantitation Reports

DATA PACKAGE COMPLETENESS CHECKLIST (Con't)
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC ANALYSIS

IIIC. Raw QC Data

- ☐ Blank Data
  - ☐ Chromatogram
  - ☐ Quantitation / Calculation of Concentrations
- ☐ Matrix Spike Data
  - ☐ Tabulated Results
  - ☐ Chromatogram(s)
- ☐ Matrix Spike Duplicate Data
  - ☐ Tabulated Results
  - ☐ Chromatogram(s)
- ☐ Instrument Settings
- ☐ Instrument Run Logs
- ☐ Percent Solids Determination Logs
- ☐ Sample Preparation/Extraction Logs

IV. Sample Receipt

- ☐ Traffic Reports
- ☐ Sample Log In Record

☐ V. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I - Data Outlier Summary Forms

ATTACHMENT II - Qualified Data Summary Reports *

ATTACHMENT III - Data Completeness Checklist

ATTACHMENT IV - Laboratory Case Narrative

ATTACHMENT V - Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
BTEX Analysis - 601

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Laboratory Sample Identification	Mound Sample Identification	Date Collected	Date Analyzed	Number of Days Past Holding Time

Reviewer: _____

Surrogate Recovery Outliers
BTEX Analysis - 601

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Soil Quality Control Limits - NA	**Water Quality Control Limits** - Fluorobenzene (70 - 130%)

Laboratory Sample Id	Mound Sample Id	Sample Matrix	Fluorobenzene		

Reviewer: _____

Matrix Spike Recovery Outliers
BTEX Analysis - 601

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Soil Quality Control Limits

-

NA

Water Quality Control Limits

-	Benzene	(70 - 130%/ RPD<25%)
-	Toluene	(70 - 130%/ RPD<25%)
-	Ethylbenzene	(70 - 130%/ RPD<25%)
-	Xylenes	(70 - 130%/ RPD<25%)

Laboratory Sample Id	Mound Sample Id	Sample Matrix	Benzene			Toluene			Ethylbenzene			Xylenes		
			%R	%R	RPD	%R	%R	RPD	%R	%R	RPD	%R	%R	RPD

Reviewer: _____

Blank Data Outlier Summary Table
BTEX Analysis - 601

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

ANALYTE	Method Blank ID	Method Blank ID	Trip Blank ID	Ambient Blank ID	Equip. Blank ID	Action Level
Benzene						
Toluene						
Ethylbenzene						
Xylene (Total)						
Affected Samples:						

Reviewer: _____

Calibration Summary
BTEX Analysis - 601

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Instrument ID:	

ANALYTE	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
		RSD < 10%			%D < 10%			%D < 10%	
Benzene									
Toluene									
Ethylbenzene									
Xylene (Total)									

Affected Samples: (Client ID)			

Was a Continuing Calibration check run every 10 samples? ☐ Yes ☐ No

Reviewer: _____

Field Duplicate Data Summary Table
BTEX Analysis - 601

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Field Sample Identification	
Field Duplicate Identification	

ANALYTE	Field Sample Result	Duplicate Result	RPD
Benzene			
Toluene			
Ethylbenzene			
Xylene (Total)			

Was a field duplicate sample submitted for every 10 field samples of each matrix? ☐ Yes ☐ No

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-022

Gasoline Range Organic (GRO) Analysis by SW8015 Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: Compendium (February 1997)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for petroleum hydrocarbon analysis by EPA Method 8015 for gasoline range organics (GRO). These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1987. Data Quality Objectives for Remedial Response Activities. U.S. Environmental Protection Agency. Office of Emergency and remedial Response and Office of Waste Programs Enforcement. Report No. EPA 540/g-87/003. Washington, D.C. March 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory

data package. Missing data is requested from the laboratory as soon as it is noted. If missing data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.
- Results of the equipment rinsate, ambient, sample blank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.
- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- Initial calibration - at least one RRF and %RSD.
- Continuing calibration - at least one RF and %D.
- Surrogate - at least one surrogate from one sample.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.

NJ	Presumptive evidence of the presence of the material at an estimated quantity.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration
H	Holding time exceeded
K	Qualified due to surrogate recovery
S	Qualified due to matrix spike recovery
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Holding Time: Verify that samples were analyzed within the holding time period specified in Method A-022 of the Methods Compendium. If the holding time was exceeded, qualify results as estimated with (J) and (UJ). If the holding time was grossly exceeded, professional judgment should be used for qualifying the data and determining whether the data are unusable (R).

Calibration: Verify that the percent relative standard deviation (%RSD) of the response factors (RF) for the initial 5 point calibration was less than 20%.

Verify that a continuing calibration was run every 10 samples and that the percent difference (%D) in RFs was within 15% of average RF from the initial calibration.

If any samples were analyzed with a non-compliant calibration standard, then the results associated with the non-compliant standard must be estimated (J, UJ).

Verify retention time windows: ± 3σ of three retention times for each analyte as per SW846.

Blanks:

Method blanks must be analyzed one per 20 samples of a given matrix per day and contamination must be less than PQL.

Verify that trip blanks are performed one per shipping container to laboratory. The qualification level for samples is determined from the maximum target analyte concentration in the method blank, equipment blank and field blank.

Verify that equipment blanks (field blanks) are performed one every 10 samples or less. The qualification level for samples is determined from the maximum target analyte concentration in the method blank, equipment blank and field blank.

Qualification Guidance Level is 5x contamination in blank

Qualification Guidelines:

- If results < PQL, report PQL as U.
- If results > PQL and < guidance level, then report the value as U.
- If results > PQL and > guidance level, then report the value unqualified.

Surrogates:

The recoveries for the surrogates should be within QC limits as specified in Method A-022 of the Methods Compendium. If recoveries are outside the QC limits, then the associated positive sample results should be estimated (J). If recoveries are below 10%, then associated sample results should be considered as unusable (R).

Laboratory Control Sample (LCS):

Verify that an LCS was performed 1 per 20 samples of a given matrix per day. Verify that LCS recoveries are within QC limits as specified in Method A-022 of the Methods Compendium.

If LCS recoveries are not within QC limits, then all associated samples may be qualified estimated (J, UJ). If recoveries are below 10%, then associated results should be considered as unusable (R).

Matrix Spike (MS)/ Matrix Spike Duplicate (MSD): Verify that an MS/MSD was performed one per 20 samples of a given matrix. Verify that MS/MSD recoveries are within QC limits as specified in Method A-022 of the Methods Compendium. If the MS or MSD recovery is less than the criteria and greater than 10%, qualify positive and non-detect results estimated (J, UJ). If the recovery is greater than criteria, then qualify positive results estimated (J). If the recovery is less than 10%, then qualify the non-detect results rejected (R) and the positive results estimated (J).

Field Duplicate: Verify that a field duplicate sample was analyzed for every 10 field samples or less of each matrix. The RPD must be < 35% for waters and < 50% for soils. If criteria was not met, then estimate (J) the positive results for that analyte(s) in both samples. If one of the sample results is > PQL and the other is non-detected, then both results are estimated (J, UJ).

Quantitation Limits: Verify that quantitation limits stated in Method A-022 of the Methods Compendium were met.

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Surrogate Recovery Forms

Complete header section. Complete remainder of table only for those samples which have surrogate outliers. If no outliers, then write 'None'.

3.5.3 Matrix Spike Recovery Forms

Complete header section. Complete remainder of table only for those samples which have MS/MSD outliers. Enter the percent recovery as appropriate. If no outliers, write 'None'. The matrix spike form should also be used to document deficient LCS results.

3.5.4 Blank Data Summary Forms

Complete the appropriate form from Attachment III. Enter blank outliers only and note appropriate action level based on 5*x* or 10*x*. Enter the associated sample IDs.

3.5.5 Calibration Forms

Complete header section, instrument, and date and time for each calibration run. Enter the outlying RF, RRF, RSD, or %D as necessary. List all associated sample IDs at bottom of form.

3.5.6 Field Duplicate Forms

Complete header section and sample IDs. Enter outlying RPD values for analytes. If no values are outside the required range, so state.

3.5.7 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for surrogate recovery, matrix spike recovery, calibration, and field duplicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- Initial calibration - at least one RRF and %RSD.
- Continuing calibration - at least one RF and %D.
- Surrogate - at least one surrogate from one sample.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.8 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC ANALYSIS

- ☐ Table of Contents (Check List)
  - I. Case Narrative
    - ☐ Cover Page
    - ☐ Case Narrative
    - ☐ Cross Reference of Sample ID to Laboratory ID
    - ☐ Description of all Qualifiers used by the Laboratory
    - ☐ Applicable Laboratory SOP and Revision Date
  - II. QC Summary
    - ☐ Surrogate Recoveries Summary
    - ☐ Matrix Spike/Matrix Spike Duplicate Summary
    - ☐ Method Blank Summary
    - ☐ Laboratory Control Sample
  - III. Raw Data
    - IIIA. Sample Data
      - ☐ Example Calculation
      - ☐ Target Compound Results
      - ☐ Chromatograms
      - ☐ Quantitation / Calculation of Concentrations
    - IIIB. Standard(s) Data
      - ☐ Initial Calibration Data
      - ☐ Standard(s) Chromatograms and Quantitation Reports
      - ☐ Continuing Calibration
      - ☐ Standard(s) Chromatograms and Quantitation Reports

DATA PACKAGE COMPLETENESS CHECKLIST (Con't)
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC ANALYSIS

IIIC. Raw QC Data

- ☐ Blank Data
  - ☐ Chromatogram
  - ☐ Quantitation / Calculation of Concentrations
- ☐ Matrix Spike Data
  - ☐ Tabulated Results
  - ☐ Chromatogram(s)
- ☐ Matrix Spike Duplicate Data
  - ☐ Tabulated Results
  - ☐ Chromatogram(s)
- ☐ Instrument Settings
- ☐ Instrument Run Logs
- ☐ Percent Solids Determination Logs
- ☐ Sample Preparation/Extraction Logs

IV. Sample Receipt

- ☐ Traffic Reports
- ☐ Sample Log In Record

☐ V. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I	-	Data Outlier Summary Forms
ATTACHMENT II	-	Qualified Data Summary Reports *
ATTACHMENT III	-	Data Completeness Checklist
ATTACHMENT IV	-	Laboratory Case Narrative
ATTACHMENT V	-	Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
Gasoline Range Organics - Method 8015

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Laboratory Sample Identification	Mound Sample Identification	Date Collected	Date Extracted	Date Analyzed	Number of Days Past Holding Time

Holding Time: Analysis within 14 days from collection

Reviewer: _____

Surrogate Recovery Outliers
Gasoline Range Organics - Method 8015

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Soil Quality Control Limits - Fluorobenzene (50 - 150%)	**Water Quality Control Limits** - Fluorobenzene (60 - 140%)

Laboratory Sample Id	Mound Sample Id	Sample Matrix	Fluorobenzene

Reviewer: _____

Matrix Spike Recovery Outliers
Gasoline Range Organics - Method 8015

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Soil Quality Control Limits - Gasoline (50 - 150%) (≤ 30%)	**Water Quality Control Limits** - Gasoline (60 - 140%) (≤ 20%)

Laboratory Sample Id	Mound Sample Id	Sample Matrix	Gasoline

Reviewer: _____

Blank Data Summary Table
Gasoline Range Organics - Method 8015

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

ANALYTE	Method Blank ID	Method Blank ID	Trip Blank ID	Ambient Blank ID	Equip. Blank ID	Action Level
Gasoline						
Applies to Samples:						

Reviewer: _____

Calibration Outliers
Gasoline Range Organics - Method 8015

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Instrument ID:	Matrix (Soil/Water):

	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
ANALYTE		RSD ≤ 20%	*		D ± 15%	•		D ± 15%	•
Gasoline									

Affected Samples: (Client ID)			

Was a continuing calibration check analyzed every 10 samples? ☐ Yes ☐ No
Were retention times acceptable? ☐ Yes ☐ No

* These flags should be applied to the analytes on the sample data sheets.

Reviewer: _____

Field Duplicate Data Summary Table
Gasoline Range Organics - Method 8015

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC LIMITS
Water = ≤ 35% RPD Soil = ≤ 50% RPD

ANALYTE	Mound Sample ID	Mound Sample ID	Mound Sample ID	Mound Sample ID
Gasoline				

Was a duplicate introduced for every 10 samples? ☐ **Yes** ☐ **No**

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-023

Total Petroleum Hydrocarbons (TPH) by EPA 418.1 Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: Compendium (February 1997)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for Total Petroleum Hydrocarbon (TPH) by USEPA Method 418.1. These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

EPA. 1986. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. November 1986.

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

EPA. 1993. "Methods for Chemical Analysis of Water and Waste," U.S. Environmental Protection Agency, EPA-600/4-79-020 revised March 1983.

DOE. 1994. "Fire Fighting Training Area Removal Action Operable Unit 5 Work Plan," U.S. Department of Energy, Mound Plant, June 1994.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory data package. Missing data is requested from the laboratory as soon as it is noted. If missing data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.

- Results of the equipment rinsate, ambient, sample bank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.

- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- Initial calibration - verify the calculated correlation coefficient.
- Continuing calibration - verify the continuing calibration check.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
N	Presumptive evidence of presence of material.
NJ	Presumptive evidence of the presence of the material at an estimated quantity.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

SUBQUALIFIERS - ORGANICS

B	Qualified due to method blank or a field blank
C	Qualified due to calibration
H	Holding time exceeded
S	Qualified due to matrix spike recovery
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Holding Time: Verify the samples were extracted and analyzed holding times specified in Method A-023 of the Methods Compendium. Qualify results as estimated with (J) and (UJ) if holding times were exceeded. If the holding time was grossly exceeded, professional judgment may be used for qualifying the data as unusable (R).

Calibration:

Verify the initial 5-point calibration curve had a correlation coefficient ≥ 0.9996. If the correlation coefficient was less than 0.9996, then qualify all results estimated (J, UJ). If the correlation coefficient was grossly outside criteria, then reject (R) all results.

Verify that a continuing calibration check (CCC) was analyzed at the frequency described in Method A-023 of the Methods Compendium. If the CCC was not run at the frequency specified in the method, then qualify the affected data estimated (J,UJ). If the CCC recovery was below the criteria specified in Method A-023 and greater than 50 percent, then qualify all results estimated. If the CCC recovery was below 50 percent reject all results reported non-detect and qualify all positive results estimated. If the CCC recovery exceeded recovery, then qualify positive results estimated (J).

Blanks:

Method blank must be analyzed for each batch of samples extracted for each matrix and contamination must be less than PQL.

Equipment blank sample are qualified using qualification guidance levels derived from method blank contamination. Action Level for samples are determined from the maximum contamination levels in method and equipment blank contamination. If the contamination is greater than the PQL, then for each affected analyte:

Qualification Guidance Level = 5x contamination in blank.

Qualification Guidelines:

- If results < PQL, then no action is taken.
- If results < 5*x* contamination, then report the value with a (U).
- If results > 5*x* contamination, then no action is taken.

Matrix Spike:

Recoveries and relative percent differences should correspond to those specified in Method A-023 of the Methods Compendium for soils and water.

If the MS/MSD recovery is below acceptance units and greater than 10%, then qualify associated sample results estimated (J, UJ).

If the MS/MSD recovery is greater than criteria, then qualify the associated positive sample results estimated.

If the recovery is less than 10%, then qualify positive results estimated and non-detect results rejected (R).

For RPD's which exceed criteria, qualify positive results estimated (J).

Laboratory Control Sample:

Laboratory control sample (LCS) recoveries must be within QC limits or all associated samples must be re-extracted with another LCS. If the criteria was not met, then:

- If the recovery is below criteria and greater than 10 percent, qualify positive results estimated (J).
- If the recovery is less than or equal to 10 percent, then qualify all results unusable, (R).
- For recoveries greater than the specified criteria, no action is taken for non-detects and positive results will be qualified estimated, (J). For recoveries that grossly exceed the specified criteria, positive results may be qualified unusable, (R), based on the validator's professional judgment.

Field Duplicate:

Verify a field duplicate sample was analyzed for every 10 field samples or less of each matrix. The RPD must be ≤ 35% for water and ≤ 35% for soils. If a criterion was not met, then estimate (J) the positive results in both samples. If one of the sample result is > PQL and the other is non-detected, then both results are estimated.

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Data Summary Forms

Complete the header section. Fill in remainder if criteria was exceeded.

3.5.3 Blank Data Summary Forms

Complete the appropriate form from Attachment III. Enter blank outliers only and note appropriate action level based on 5*x* or 10*x*. Enter the associated sample IDs.

3.5.4 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for the matrix spike recovery, calibration, field duplicate, and replicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- Initial calibration - verify the calculated correlation coefficient.
- Continuing calibration - verify the continuing calibration check.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.5 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

TOTAL PETROLEUM HYDROCARBON

- ☐ Table of Contents (Check List)
  - I. Case Narrative
    - ☐ Cover Page
    - ☐ Case Narrative
    - ☐ Cross Reference of Sample ID to Laboratory ID
    - ☐ Description of all Qualifiers used by the Laboratory
    - ☐ Applicable Laboratory SOP and Revision Date
  - II. QC Summary
    - ☐ Matrix Spike/Matrix Spike Duplicate Summary
    - ☐ Method Blank Summary
    - ☐ Laboratory Control Sample
  - III. Raw Data
    - IIIA. Sample Data
      - ☐ Example Calculation
      - ☐ Target Compound Results - Organic Analysis Data Sheet
      - ☐ Spectra
      - ☐ Quantitation Report
      - ☐ Quantitation / Calculation
    - IIIB. Standard(s) Data
      - ☐ Initial Calibration Data
      - ☐ Continuing Calibration

DATA PACKAGE COMPLETENESS CHECKLIST (Con't)
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

• TOTAL PETROLEUM HYDROCARBON

IIIC. Raw QC Data

- ☐ Blank Data
  - ☐ Target Compound Spectra
  - ☐ Quantitation / Calculation
- ☐ Matrix Spike Data
  - ☐ Tabulated Results
  - ☐ Spectra
- ☐ Matrix Spike Duplicate Data
  - ☐ Tabulated Results
  - ☐ Spectra
- ☐ Instrument Settings
- ☐ Instrument Run Logs
- ☐ Percent Solids Determination Logs

IV. Sample Receipt

- ☐ Traffic Reports
- ☐ Sample Log In Record

☐ V. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I	-	Data Outlier Summary Forms
ATTACHMENT II	-	Qualified Data Summary Reports *
ATTACHMENT III	-	Data Completeness Checklist
ATTACHMENT IV	-	Laboratory Case Narrative
ATTACHMENT V	-	Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
Total Petroleum Hydrocarbons - 418.1

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Laboratory Sample Identification	Mound Sample Identification	Date Collected	Date Extracted	Date Analyzed	Number of Days Past Holding Time

Reviewer: _____

Data Summary Table
Total Petroleum Hydrocarbon - 418.1

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Instrument Identification:	

ANALYTE	Calibration		Blank Evaluation		
	Initial Coeff ≥ 0.9996	Continuing %D < 20	Method Blank ID	Method Blank ID	Equipment Blank ID
TPH					
Applies to Samples (Client Id)					

Matrix Spike/Matrix Spike Duplicate / LCS **(75 - 125% R, < 15% RPD)**

Sample ID	Matrix	TPH	
		% R	% RPD

Field Duplicates **(Water ≤ 35% RPD)**

ANALYTE	Sample ID	Sample ID	Sample ID	Sample ID
TPH				

Reviewer: _____

MOUND

Environmental
Restoration
Program

Method: DV-024

Diesel Range Organic (DRO) Analysis by SW8015 for Data Validation

Revision 2.0

Mound Plant
Miamisburg, OH

Source Document: Compendium (February 1997)

1. INTRODUCTION

1.1 Purpose

This procedure describes the data validation requirements for petroleum hydrocarbon analysis by EPA Method 8015 for diesel range organics (DRO). These guidelines are presented to ensure that data validation is performed consistently for chemical data collected for the Mound ER Program. However, these guidelines may not be inclusive of all Quality Control Check (QCC) deficiencies or conditions, and validators may need to use professional judgment in these limited cases. These cases may include, but are not limited to, evaluations of a grossly exceeded QCC criteria, QCCs not described in these procedures that could impact data quality, evaluations of trends related to QCC results from multiple field batches, or multiple QCC deficiencies.

1.2 References

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1987. Data Quality Objectives for Remedial Response Activities. U.S. Environmental Protection Agency. Office of Emergency and remedial Response and Office of Waste Programs Enforcement. Report No. EPA 540/g-87/003. Washington, D.C. March 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

2. RESPONSIBILITIES

Data Validator - A data validator must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>or</u> two years of experience performing data validation.

Data Validation Reviewer - A data validation reviewer must have a minimum of a Bachelors degree in chemistry, two years of work experience in an environmental analytical laboratory, <u>and</u> two years of experience performing data validation.

3. PROCEDURES

3.1 General Guidelines

The following procedures apply to all data packages submitted for data validation.

- The data package is first reviewed for completeness by the data validator. The Data Completeness Checklist, in Attachment I of this method, is completed for each laboratory

data package. Missing data is requested from the laboratory as soon as it is noted. If missing data is identified during data validation, the validation effort will continue as feasible until the data is provided by the laboratory.

- Data validation is performed using the guidelines presented in Section 3.4 of this document.
- Results of the equipment rinsate, ambient, sample blank, and trip blanks are used to qualify sample data. Results of laboratory blanks are used in qualifying field blank data and sample data, as with any investigative sample.
- Any QCCs not specifically found in the following procedures but outlined in the Methods Compendium will be evaluated against the Method Compendium criteria.

3.2 Calculations

A portion of all laboratory sample result calculations will be re-calculated and verified following the guidelines described below.

- For manual calculations, 10 percent of all calculations in the batch will be verified.
- For automated calculations, one calculation per batch will be verified.

If a calculation error is found, the data set will be submitted to the laboratory for verification of all the sample calculations. The laboratory will be required to make all necessary corrections to the data.

In addition to verifying sample calculations, the validators verify:

- Initial calibration - at least one RRF and %RSD.
- Continuing calibration - at least one RF and %D.
- Surrogate - at least one surrogate from one sample.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

3.3 Data Qualifiers

The primary data qualifiers to be applied, as described in the CLP Functional Guidelines are the following:

U	The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J	The associated numerical value is an estimated quantity.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
UJ	The material was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

Subqualifiers are used to assist in data assessment by indicating the source of the primary qualifier. These subqualifiers are attached to the primary qualifier, separated by a hyphen. The following is a list of allowable subqualifiers:

B	Qualified due to method blank or a field blank
C	Qualified due to calibration
H	Holding time exceeded
K	Qualified due to surrogate recovery
S	Qualified due to matrix spike recovery
(+)	Potential positive bias (added after subqualifier with a parentheses)
(-)	Potential negative bias (added after subqualifier with a parentheses)

Examples of final qualification might be J-C, UJ-S(+), UJ-BC(-), etc.

The Mound Project Manager must approve the use of additional qualifiers and sub-qualifiers.

3.4 Analysis Specific Validation Instructions

Holding Time: Verify that samples were analyzed within the holding time specified in Method A-024 of the Methods Compendium. If the holding time was exceeded, qualify results as estimated with (J) and (UJ). If the holding time was grossly exceeded, professional judgment should be used for qualifying the data and determining whether the data are unusable (R).

Calibration: Verify that the percent relative standard deviation (%RSD) of the response factors (RF) for the initial 5-point calibration was less than 20%.

Verify that a continuing calibration was run every 10 samples and that the percent difference (%D) in RFs was within 15% of average RF from the initial calibration.

If any samples were analyzed with a non-compliant calibration standard, then the results associated with the non-compliant standard must be estimated (J, UJ).

Verify retention time windows: $\pm 3\sigma$ of three retention times for each analyte as per SW846.

Blanks:

Method blanks must be analyzed one per 20 samples of a given matrix per day and contamination must be less than PQL.

Verify that trip blanks are performed one per shipping container to laboratory. Verify that equipment blanks (field blanks) are performed one every 10 samples or less. The qualification level for samples is determined from the maximum contamination levels in method, trip, and equipment blank.

Qualification Guidance Level is 5x contamination in blank

Qualification Guidelines:

- If results < PQL, report PQL as U.
- If results > PQL and < guidance level, then report the value as U.
- If results > PQL and > guidance level, then report the value unqualified.

Surrogates:

The recoveries for the surrogates should be within QC limits specified in Method A-024 of the Methods Compendium. If recoveries are outside limits specified in Method A-024, then associated positive sample results should be estimated (J). If recoveries are below 10%, then associated sample results should be considered as unusable (R).

Laboratory Control Sample (LCS):

Verify that an LCS was performed 1 per 20 samples of a given matrix per day. Verify that LCS recoveries are within QC limits as specified in Method A-024 of the Methods Compendium.

If LCS recoveries are not within QC limits, then all associated samples may be qualified estimated (J, UJ). If recoveries are below 10%, then associated results should be considered as unusable (R).

Matrix Spike (MS)/ Matrix Spike Duplicate (MSD):

Verify that an MS/MSD was performed one per 20 samples of a given matrix. Verify that MS/MSD recoveries are within QC limits as specified in Method A-024 of the Methods Compendium. If the MS or MSD recovery is less than the criteria and greater than 10%, qualify positive and non-detect results estimated (J, UJ). If the recovery is greater than criteria, then qualify positive results estimated (J). If the recovery is less than 10%, then qualify the non-detect results rejected (R) and the positive results estimated (J).

Field Duplicate: Verify that a field duplicate sample was analyzed for every 10 field samples or less of each matrix. The RPD must be < 35% for waters and < 50% for soils. If criteria was not met, then estimate (J) the positive results for that analyte(s) in both samples. If one of the sample results is > PQL and the other is non-detected, then both results are estimated (J,UJ).

Quantitation Limits: Verify that quantitation limits stated in Method A-024 of the Methods Compendium were met.

3.5 Data Validation Reporting Requirements

Validation report contents must be in a narrative format and must follow the outline presented in Attachment II.

Data outlier summary forms presented in Attachment III are to be completed as described in this section. All forms associated with the analytical method must be completed for each data validation report and must be signed and dated by the data validator and reviewer.

3.5.1 Holding Time Forms

Complete the appropriate form from Attachment III for all samples with date of sampling, date of extraction, date of analysis, and write number of days between each event in the right-hand corner and note whether extraction or analysis holding time was exceeded.

3.5.2 Surrogate Recovery Forms

Complete header section. Complete remainder of table only for those samples which have surrogate outliers. If no outliers, then write 'None'.

3.5.3 Matrix Spike Recovery Forms

Complete header section. Complete remainder of table only for those samples which have MS/MSD outliers. Enter the percent recovery as appropriate. If no outliers, write 'None'. The matrix spike form should also be used to document deficient LCS results.

3.5.4 Blank Data Summary Forms

Complete the appropriate form from Attachment III. Enter blank outliers only and note appropriate action level based on 5*x* or 10*x*. Enter the associated sample IDs.

3.5.5 Calibration Forms

Complete header section, instrument, and date and time for each calibration run. Enter the outlying RF, RRF, RSD, or %D as necessary. List all associated sample IDs at bottom of form.

3.5.6 Field Duplicate Forms

Complete header section and sample IDs. Enter outlying RPD values for analytes. If no values are outside the required range, so state.

3.5.7 Sample Calculations

A sample calculation should be provided in the upper right hand corner of tables for surrogate recovery, matrix spike recovery, calibration, field duplicate, and replicate tables along with the data validators initials and date. This calculation should show the formula, actual sample values, and final result. Use the following as a guideline for calculations to show:

- Initial calibration - at least one RRF and %RSD.
- Continuing calibration - at least one RF and %D.
- Surrogate - at least one surrogate from one sample.
- MS/MSD - at least one compound recovery and RPD for each analyte group.
- LCS - at least one compound.

The first draft report of data validation results is delivered to the Data Validation Reviewer or his/her designee within 20 days from date of data receipt for a senior technical review. Requested corrections to the first draft reports must be submitted within 10 calendar days, unless otherwise noted.

3.5.8 Qualified Data Summary Forms

Form 1s for CLP or result summary sheets for Non-CLP analyses should be copied and the proper qualifiers/subqualifiers added as discussed in Section 3.4.

Attachment I

Laboratory Completeness Checklist

DATA PACKAGE COMPLETENESS CHECKLIST
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC ANALYSIS

- ☐ Table of Contents (Check List)
  - I. Case Narrative
    - ☐ Cover Page
    - ☐ Case Narrative
    - ☐ Cross Reference of Sample ID to Laboratory ID
    - ☐ Description of all Qualifiers used by the Laboratory
    - ☐ Applicable Laboratory SOP and Revision Date
  - II. QC Summary
    - ☐ Surrogate Recoveries Summary
    - ☐ Matrix Spike/Matrix Spike Duplicate Summary
    - ☐ Method Blank Summary
    - ☐ Laboratory Control Sample
  - III. Raw Data
    - IIIA. Sample Data
      - ☐ Example Calculation
      - ☐ Target Compound Results
      - ☐ Chromatograms
      - ☐ Quantitation / Calculation of Concentrations
    - IIIB. Standard(s) Data
      - ☐ Initial Calibration Data
      - ☐ Standard(s) Chromatograms and Quantitation Reports
      - ☐ Continuing Calibration
      - ☐ Standard(s) Chromatograms and Quantitation Reports

DATA PACKAGE COMPLETENESS CHECKLIST (Con't)
EG&G MOUND PROJECT

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

GC ANALYSIS

IIIC. Raw QC Data

- ☐ Blank Data
  - ☐ Chromatogram
  - ☐ Quantitation / Calculation of Concentrations
- ☐ Matrix Spike Data
  - ☐ Tabulated Results
  - ☐ Chromatogram(s)
- ☐ Matrix Spike Duplicate Data
  - ☐ Tabulated Results
  - ☐ Chromatogram(s)
- ☐ Instrument Settings
- ☐ Instrument Run Logs
- ☐ Percent Solids Determination Logs
- ☐ Sample Preparation/Extraction Logs

IV. Sample Receipt

- ☐ Traffic Reports
- ☐ Sample Log In Record

☐ V. Nonconformance Records / Correspondence / Telephone Communications Records / ETC.[1]

Laboratory Reviewer:	Date:
WESTON Verified by:	Date:
Comments:	

[1] Any records not specified, but pertaining to the sample analysis.

Attachment II

Example Report Format

DATA VALIDATION REPORT FORMAT
EG&G MOUND ER PROGRAM

I. TITLE - "Report of Data Validation Results"

II. HEADER - Name of Project, Operable Unit, Description of Task (e.g., Groundwater monitoring), Work Order Number, and date of report.

III. CASE SUMMARY

State the following:

- Case/Batch number
- Number of samples and type of matrix
- Date of Collection
- Chain of Custody identifier, if any
- Type of analysis (Discuss impacts if incorrect method used)
- Condition of samples when received by the laboratory, any lost data
- List the client sample IDs applicable to the report.

IV. HOLDING TIMES

Discuss results of holding times and those outside the required holding time.

V. RESULTS OF LABORATORY QUALITY CONTROL CHECKS

For each laboratory quality control check required to be performed, discuss whether the frequency, acceptance criteria, and corrective actions were met. Summarize which samples were affected and how the data was impacted.

The laboratory quality control checks for each analysis are listed below:

<u>Volatiles and Semivolatiles</u>

a) GC/MS Tuning
b) Calibration
c) Blanks
d) Surrogate Spike Recoveries
e) Matrix spike/Matrix Spike Duplicate Recoveries
f) Internal Standards
g) Compound Identification
h) System Performance
i) Compound Quantitation and Contract Required Quantitation Limits (CRQLs)
j) Tentatively Identified Compounds
k) Laboratory Control Sample

Pesticides/PCBs

a) GC/ECD Performance Check
b) Performance Evaluation Mixture
    i) Resolution
    ii) Retention Time
    iii) Recovery
    iv) DDT/Endrin Breakdown
c) Calibration
d) Blanks
e) Surrogate Spike Recoveries
f) Matrix spike/Matrix Spike Duplicate Recoveries
g) Pesticide Cleanup Checks
    i) GPC Recoveries
    ii) Florosil Cartridge Cleanup
h) Compound Identification
i) System Performance
j) Compound Quantitation and Reported CRQLs
k) Laboratory Control Sample

Inorganic Metals and Cyanide

a) Calibration and CRDL
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Interference Check Sample
d) Laboratory Control Sample
e) Duplicate Sample Analysis
f) Matrix Spike Sample Analysis
    i) ICP Metals
    ii) GFAA Metals
    iii) Cyanide
g) Furnace Atomic Absorption QC
h) ICP Serial Dilution
i) Sample Result Verification

Anions

a) Calibration
b) Blanks
    i) Preparation Blanks
    ii) Calibration Blanks
c) Laboratory Control Sample
d) Matrix Spike/Matrix Spike Duplicate Sample Analysis
e) Sample Result Verification

VI. RESULTS OF ASSOCIATED FIELD QUALITY CONTROL CHECKS

A. Field Duplicates

B. Field Blanks (equipment blanks, etc.)

VII. OVERALL ASSESSMENT OF DATA

Discuss overall assessment of data and categorize data in one or more of the following (use these as subsections to this section):

- Data had no problem/or qualified due to minor problems.
- Data qualified due to major problems.
- Data unacceptable.
- Problems, but does not affect data.

Discuss how all findings in the review could impact the data usability.

ATTACHMENT I - Data Outlier Summary Forms

ATTACHMENT II - Qualified Data Summary Reports *

ATTACHMENT III - Data Completeness Checklist

ATTACHMENT IV - Laboratory Case Narrative

ATTACHMENT V - Chain-of-Custody

* Form is for CLP analyses, data summary reports from laboratory for non-CLP analyses

Attachment III

Data Validation Worksheets

Holding Time Summary
Diesel Range Organics - Method 8015

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

Laboratory Sample Identification	Mound Sample Identification	Date Collected	Date Extracted	Date Analyzed	Number of Days Past Holding Time

Holding Time: Analysis within 14 days from collection

Reviewer: _____

Surrogate Recovery Outliers
Diesel Range Organics - Method 8015

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Soil Quality Control Limits - p-terphenyl (50 - 150%)	**Water Quality Control Limits** - p-terphenyl (50 - 150%)

Laboratory Sample Id	Mound Sample Id	Sample Matrix	p-terphenyl

Reviewer: _____

Matrix Spike Recovery Outliers
Diesel Range Organics - Method 8015

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Soil Quality Control Limits - Diesel Fuel (50 - 150%) (≤ 30%)	**Water Quality Control Limits** - Diesel Fuel (50 - 150%) (≤ 20%)

Laboratory Sample Id	Mound Sample Id	Sample Matrix	Diesel Fuel

Reviewer: _____

Blank Data Summary Table
Diesel Range Organics - Method 8015

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

ANALYTE	Method Blank ID	Method Blank ID	Trip Blank ID	Ambient Blank ID	Equip. Blank ID	Action Level
Diesel Fuel						
Applies to Samples:						

Reviewer: _____

Calibration Outliers
Diesel Range Organics - Method 8015

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:
Instrument ID:	Matrix (Soil/Water):

	Initial Calibration			Continuing Calibration			Continuing Calibration		
	Date: Time:			Date: Time:			Date: Time:		
ANALYTE		RSD ≤ 20%	*		D ± 15%	*		D ± 15%	*
Diesel Fuel									

Affected Samples: (Client ID)			

Was a continuing calibration check analyzed every 10 samples? ☐ **Yes** ☐ **No**
Were retention times acceptable? ☐ **Yes** ☐ **No**

* These flags should be applied to the analytes on the sample data sheets.

Reviewer: _____

Field Duplicate Data Summary Table
Diesel Range Organics - Method 8015

Prepared by:	WESTON Work Order:
Date:	Mound Project Name:
Laboratory Batch Number:	Operable Unit: NA
Laboratory Name/Location:	Collection Date:

QC LIMITS
Water = ≤ 35% RPD Soil = ≤ 50% RPD

ANALYTE	Mound Sample ID	Mound Sample ID	Mound Sample ID	Mound Sample ID
Diesel Fuel				

Was a duplicate specified for every 10 samples? ☐ **Yes** ☐ **No**

Reviewer: _____

FIELD STANDARD OPERATING PROCEDURES

Table of Contents

Method Number	Method Description	Document
S-001	General Instructions for Field Personnel	QAPP
S-002	Soil Sampling with a Spade and Scoop	QAPP
S-003	Soil and Rock Borehole Logging and Sampling	QAPP

FIELD STANDARD OPERATING PROCEDURES

The field procedures were included in the Compendium to provide continuity between the Potential Release Site Investigations and the OU9 Site-Wide Investigation. Many of the field procedures, as identified in the table of contents for this section, were adapted from the SOPs in Appendix A of the RI/FS Operable Unit 9, Site-Wide Quality Assurance Project Plan. The procedures were revised to incorporate new information and remove redundancies.

As new procedures are required, the procedures will be introduced into the Compendium. Each new procedure will clearly reference the first approved sampling plan and date where the new procedure was first used. When a new procedure is added, the revised table of contents and the procedure will be distributed to the copy holders of the Compendium.

MOUND

Environmental
Restoration
Program

Method: S-001

General Instructions for Field Personnel

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. APPLICABILITY

This procedure provides field personnel with general instructions regarding activities to be performed before, during, and after field investigations. These general instructions are intended to supplement the information supplied in the project plan and associated standard operating procedures (SOPs) and clarify the role of field personnel during investigations. These instructions will ensure that field personnel take the proper precautions to understand the site, the objective and the schedule for the field program, their authority, and their responsibilities described in the project plans. Review of the SOPs will ensure that the work performed in the field is legally defensible, well documented, and cost-effective.

This procedure was formerly identified as ER Program SOP 1.1 (March 1992).

2. RESPONSIBILITIES

In most field programs, there are three levels of responsibility. The levels have been identified as project manager, field team leader, and field technician. On larger projects, one person may be assigned to each position. However, for small projects, a single person may fulfill the responsibilities for all three job responsibilities.

Project Manager – The project manager (PM) has overall responsibility for the project. The PM must approve all changes and deviations to the project plans. The PM has ultimate responsibility for schedule, quality and budget.

Field Team Leader – The Field Team Leader (FTL) is responsible for field functions. These functions include compliance to project plans (schedule, quality, etc.) and directing field activities. The FTL is responsible for communicating status and changes or deviations to the project manager.

Field Technician – The Field Technician is responsible for performing the tasks assigned by the FTL. The FT is responsible for knowing the procedures required by the FTL and communicating status and problems to the FTL.

In addition to the responsibilities defined above, each person in any of the levels is responsible for implementing corrective actions and corrective action reports as defined in Q-001 when a plan deficiency is identified.

2.1 Before Starting a Project

2.1.1 Review Project Documents

Personnel should review the project plans and associated documentation for a specific operation and obtain all information related to the purpose and intent of the field program. This may include (but is not limited to) the documents listed below.

- The scope of work or work plan described in the project plans;
- Previous reports related to the site;
- Reports related to the area;
- Site maps;
- Area maps;
- Access agreements;
- The subcontractor's work plan;
- Data collection forms and equipment checklists; and
- Associated SOPs.

2.1.2 Pre-Job Meeting

For some Mound projects, a pre-job meeting is required. If a pre-job meeting is required, then prepare for the meeting and attend the meeting.

2.1.3 Equipment

Order the field equipment which will be needed to collect the samples and to perform any required field analyses. Table 3.1 includes examples of equipment which may be needed during a typical field activity. Before going into the field to collect samples, verify the equipment is working properly. If the equipment is not working properly, either repair or replace the equipment as appropriate.

2.1.4 Logbooks and Data Sheets

Before starting the project, obtain a field logbook and any required data collection forms. Other documents which may be required include:

- Chain-of-Custody forms;
- Excavation Permit;
- Burn permits;
- Rad. Worker II Certification; or
- Safety Checklists.

2.2 Documentation

2.2.1 Document Inventory during Field Effort

If the following project or reference documents are available, the field team will have the documents at field site:

- A copy of the Statement of Work;
- A copy of the Quality Assurance Project Plan;
- A copy of the applicable methods from the Methods Compendium;
- A copy of the SAP; and
- A signed copy of the HASP.

In addition to the project and reference documents, the field team must have copies of applicable permits and licenses.

2.2.2 Record Keeping

The field team, at a minimum, is required to maintain a field logbook for every sampling or construction effort. In addition to the field logbook, the field team, as required by the project, often must complete the following forms or records:

- Chain-of-custody records;
- Field measurement forms;
- Equipment calibration records;
- Field data forms; or
- Corrective action reports.

2.3 Field Activities

2.3.1 Decontamination Zones and Public Barricades

As required by the project plan, establish any required decontamination zones or public barricades.

2.3.2 Equipment Calibration and Checks

Each day before sampling begins check and calibrate equipment as necessary. If a piece of equipment can't be calibrated or fails an instrument check, then tag the equipment as unusable and make arrangements to repair or replace the piece of equipment. If the equipment defect may have impacted field data results collected from the previous day, then initiate a corrective action report to document which samples may be affected and any corrective actions taken (Q-001 of the Methods Compendium).

2.3.3 Sample Locations

Sample locations will be identified by Mound, the field team leader, or the project manager. In some instances, the field team leader or project manager may authorized a field team member to identify sample locations based on the project documents.

If a selected sample location must be moved, the new sample location must meet all the approval requirements of the primary location and the deviation must be recorded following the corrective action procedure described in procedure Q-001.

2.3.4 Documentation

Logbooks will be completed as described in Q-003. In addition to the field logbook, data collection forms, chain-of-custody forms, corrective action reports, etc. will be completed as required by the project. A list of possible forms and documents are listed in Section 3.1.

2.3.5 Communication

Field team members will provide a progress report at the end of each day to the site manager or office headquarters.

2.3.6 Subcontractor Oversight

The field team is responsible for ensuring the compliance of all subcontractors with the project requirements and any site requirements, such as permits and licenses. In the event a subcontractor is not complying with project requirements, the field team member will initiate corrective actions which may include stopping work.

Table 3.1 – Commonly Required Equipment and Supplies

- ❑ Overshoes
- ❑ Work gloves (2 pairs)
- ❑ Acid (10% HCl) bottle
- ❑ Clipboard case
- ❑ Strapping Tape
- ❑ Pin hammer
- ❑ Tape measure (tenths)
- ❑ Protractor
- ❑ Hat
- ❑ First aid kit
- ❑ Sun Screen
- ❑ Thermoluminescent dosimeter (TLD)
- ❑ Safety shoes/boots
- ❑ Ziplock ® bags
- ❑ Pre-printed labels
- ❑ Distilled (organic-free) water
- ❑ Methanol (nanograde)
- ❑ Freight forms (Federal Express forms)
- ❑ Telephone directories
- ❑ Chain-of-Custody forms
- ❑ Hard Hat
- ❑ Pieces of wood (2 inches x 2 inches x 8 inches) to indicate core loss intervals

Table 3.1 – Commonly Required Equipment and Supplies (cont'd)

- ❑ Stamped addressed envelopes
- ❑ Phone and gas credit cards
- ❑ Calculator
- ❑ Pens, pencils, and permanent markers
- ❑ Package cord
- ❑ Flagging
- ❑ Hand lens
- ❑ Tool box
- ❑ Rain suit
- ❑ Camera
- ❑ Ear plugs
- ❑ Stopwatch
- ❑ Cold-weather gear
- ❑ Alpha meter
- ❑ Safety glasses
- ❑ Kitchen screen (determine lithology)
- ❑ Ice chest
- ❑ Bound logbooks
- ❑ Data collection forms
- ❑ Caution Tape
- ❑ Custody Seals

MOUND

Environmental
Restoration
Program

Method: S-002

Soil Sampling with a Spade and Scoop

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. APPLICABILITY

This method describes collecting a grab soil sample from less than 4-feet below ground surface. The Sampling and Analysis Plan (SAP) or other project plan may contain specific details about the procedures and equipment for this SOP. Refer to the SAP for the type of samples to be collected. Collection and measurement of samples and documentation of data will be performed as described in the associated procedures. Collection of samples from near the ground surface can be accomplished with hand tools, i.e. spades, shovels, and scoops.

2. RESPONSIBILITIES

See Procedure S-001.

3. PROCEDURE

3.1 Preparation

Prior to sampling, the following tasks must be completed:

1. Order the equipment which will be needed for the task. Table 3.1 lists typically equipment which may be required for this procedure. Verify the equipment is working properly and if necessary, decontaminate the equipment.

2. Order any required bottles, preservatives, packing materials, or coolers. Verify the correct materials are received. For bottles and preservatives, retain any bottle lot check or preservative lot checks data. The lot check data will be included in the project file.

3. Review Procedure S-001 *General Instructions for Field Personnel* in the Methods Compendium.

4. Review the SAP and determine whether the SAP includes specific sampling instructions which supersede the requirements specified in this procedure.

5. If sampling will be performed off-site, verify that permission has been granted to access the property.

6. Prior to collecting samples, verify that sample locations have been cleared of underground utilities.

7. Prior to digging on the Mound site, verify that you have been given a current and signed excavation permit or been told an excavation permit is not required.

8. Assuming samples will be sent for off-site analysis, verify that the analyses have been scheduled with the laboratory.

9. Verify that the sample locations have been staked and surveyed. If surveying will be performed at the completion of the task, make sure that the surveyor is scheduled to perform the work immediately after completing the sampling task.

3.2 Documentation

Obtain a field notebook. The field notebook will be used to collect information on site conditions, personnel at the sample location during sampling, sample specific measurements that may be required, equipment calibrations, and anomalies observed during sampling. Sample identity information will also be entered into the notebook. Compendium Method Q-003 describes the standard documentation requirements. In addition to the notebook entries, the SAP or applicable field procedures may also require collecting information on a data sheet. For instance, if soil logging is required, a borehole log description form may be needed.

3.3 Operation

1. Label the sample bottles. Be sure the label is clearly written and the appropriate analyses are specified.

2. If a sod layer is present at the sample location, then remove the layer of sod from the soil with a shovel.

3. Using an appropriately decontaminated stainless steel or Teflon® scoop or trowel, remove and discard a thin layer of soil from the area that came in contact with the shovel.

4. Begin collecting the soil sample with the scoop or trowel.

5. ***For a sample being submitted for organic volatiles analysis, go to step 5.*** Otherwise, transfer the soil sample to a stainless steel bowl and homogenize the sample thoroughly.

6. Transfer the sample into an appropriate sample bottle.

   a. For volatile sample fractions, the soil should be placed into the bottle directly from the sample location and the bottle should be filled so that there is little headspace.

   b. For other sample fractions, the homogenized soil sample should be transferred from the stainless steel bowl to the appropriate bottle.

6. Preserve the sample. For most analyses, soil samples are kept cool at 4 degrees Celsius; however, be sure to review the project plans for preservation requirements.

7. Be sure the Teflon liner is present in the cap. Secure the cap tightly.

8. Record the pertinent sample information into the logbook. The entries should include: the sample location, the sample identification, the fractions collected for analysis, preservation used on the samples, and any anomalies. The documentation requirements

are presented in greater detail in Compendium Method Q-003. An anomaly might include discoloration of the soil or problems obtaining a homogenous sample.

9. Place the samples into a cooler with blue ice or double bagged ice to keep the soil cool. Be sure bags are sealed to prevent leakage as the ice melts.

10. Complete the required chain-of-custody forms, see Compendium Method Q-002. Typically the chain-of-custody form will also be used as a laboratory analysis request form. Be sure to properly correct all errors on the chain-of-custody by drawing a line through the error, writing the correct entry next to the lined out entry, and dating and initialing the change.

11. Follow the requirements for handling investigation-derived material (IDM) and specific instructions in the project plans for backfilling. If sod was removed from the sample location, then replace the sod.

12. Decontaminate sample equipment before using the equipment to collect a sample.

13. Prepare the sample for transportation according the project plan requirements or the methods specified in the Methods Compendium.

14. Verify the completeness and accuracy of the logbook entries, and sign/initial and date all pages. Verify that all data collection forms are complete and sign/initiate and date any applicable forms.

Table 3.1

Examples of Typical Equipment and Supplies

- ❑ Stainless steel scoop or lab spoons (scoopulas)
- ❑ Stainless steel shovel or flat pointed mason trowel
- ❑ Stainless steel spade
- ❑ Tape measure (tenths of feet)
- ❑ Teflon sheets or stainless steel sampling trays
- ❑ Plastic sheet
- ❑ Alconox
- ❑ Brushes (long handle, scrub, and wire)
- ❑ Galvanized tub
- ❑ Trash bags
- ❑ Buckets (galvanized, stainless steel, and plastic)
- ❑ Garden pressure sprayer
- ❑ Cleaning wipes
- ❑ Kim wipes
- ❑ Storage containers for waste decontamination solutions
- ❑ Blue ice or equivalent
- ❑ Disposable laboratory gloves
- ❑ Camera and film
- ❑ Sample containers and preservatives
- ❑ Custody Seals

MOUND

Environmental
Restoration
Program

Method: S-003

Soil and Rock Borehole Logging and Sampling

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. APPLICABILITY

To describe the physical nature of consolidated or unconsolidated subsurface earthen materials encountered during auger, rotary, or other drilling activities and collect samples of the earthen materials for further evaluation.

Most sites where soil logging is conducted include unconsolidated deposits of varying thickness over consolidated bedrock at depth. Standard forms, such as Figure 1, may be used to collect soil and rock data.

2. RESPONSIBILITIES

See Compendium Method S-001.

3. PROCEDURE

Prior to sampling, the following tasks must be completed:

1. Order the equipment which will be needed for the task. Verify the equipment is working properly and if necessary, decontaminate the equipment.

2. Order any required sample containers, packing or shipping materials. Verify the ordered materials meet the project quality control requirements.

3. Review Procedure S-001 *General Instructions for Field Personnel* in the Methods Compendium.

4. Review the SAP and determine whether the SAP includes specific sampling instructions which supersede the requirements specified in this procedure.

5. If sampling will be performed off-site, verify that permission has been granted to access the property.

6. Prior to going to the sample site and drilling, verify that sample locations have been cleared of underground utilities.

7. Prior to drilling on the Mound site, verify that you have been given a current and signed excavation permit or been told an excavation permit is not required.

8. Verify that the sample locations have been staked and surveyed. If surveying will be performed at the completion of the task, make sure that the surveyor is scheduled to perform the work immediately after completing the sampling task.

3.1 Documentation

Obtain a field notebook. The field notebook will be used to collect information on site conditions, personnel at the sample location during sampling, sample specific measurements that

may be required, equipment calibrations, and anomalies observed during sampling. Sample identity information will also be entered into the notebook. Compendium Method Q-003 describes the standard documentation requirements. In addition to the notebook entries, the SAP or applicable field procedures may also require collecting information on a data sheet. For instance, a borehole log description form may be needed.

3.2 Operation

1. When a soil sample will be collected and stored for later evaluation, the sample container should be labeled and prepared before the sample is collected.

2. The geologist will evaluate the gross properties of the sample by unaided visual observation (naked eye) or inspection under a hand lens. Whenever feasible, the sample description will be based on observing fresh cuttings or surfaces. These descriptions should either be recorded neatly in the notebook or in the appropriate field forms.

3. The ASTM method D2488-93 can be used to classify soils and ASTM Method D2113-83 can be used to classify rock. These ASTM procedures describe in detail how to apply the Unified Soil Classification System designations and the Unified Rock Classification System designations. Record the soil descriptions in the field logbook or on a standard form.

4. Blow counts for unconsolidated deposits may be counted for each of the 0.5 ft. penetrations of the Standard Penetration test.

5. Record all linear measurements in feet or tenths of feet, not inches or meters, in either the logbook or on the appropriate form.

6. If the core will be retained, place the core in a labeled box after inspection. The box should be further labeled to indicated the top, bottom and interval of the core sample. Mark the core and box with colored markers. Two parallel lines in different colors should be marked on the core and box so that the relative position of the core can be preserved between observations. Mark unrecovered core intervals in the box with blocks of wood.

3.3 Post-operation activities

After completing the excavation, restore the sample location to pre-job conditions including abandoning borings per the project plan requirements.

Verify the logbook entries are complete and all required forms have been completed properly. Record any anomalies noted during the operation or site closure. If any errors must be corrected, correct the errors by drawing a single line through the error, write the correction nearby, date and initial the change. Sign and date completed logbook pages each day.

Figure 1

BORING LOG

SITE ID:

CLIENT:
SITE NAME:
PROJECT NO.:
DRILLING CO.:
LOGGED BY:

ELEV. (GRND):
NORTHING:
EASTING:
DEPTH TO WATER (bgs):
TOTAL BORING DEPTH (bgs):

(TOC):

Page 1 of 1
DATE:

DRILL METHOD:
SITE TYPE Well ( ) Boring ( ) Piezo. ( ) Other:

SAMPLE INTERVAL (ft) FROM	TO	RECOVERY (ft)	BLOW COUNT (per 6 inches)				DEPTH (ft)	GRAPHIC COLUMN	FIELD MONITORING PID	ODOR	MOISTURE CONDITION	HEADSPACE (ppm) PID	CONSISTENCY/STRENGTH	PLASTICITY	COLOR	LITHOLOGIC DESCRIPTION
							0									
							5									
							10									
							15									
							20									
							25									

LEGEND

Graphic	Soil Type	Strength		Plasticity	
		n	none	np	none
		l	low	l	low
		m	medium	m	medium
		h	high	h	high
		vh	v high		

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