WP 02-EM3003

Revision 5

Data Validation and Verification of RCRA Constituents

Management Control Procedure

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APPROVED FOR USE

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INTRODUCTION [1, 2]

This procedure provides instructions on performing verification and validation of laboratory data containing the analysis results of groundwater monitoring samples. This procedure is applied only to the non-radiological analyses results for compliance data associated with the groundwater monitoring sampling around the Waste Isolation Pilot Plant (WIPP) site.

The basis for verification and validation is provided in WP 13-1, Washington TRU Solutions LLC Quality Assurance Program Description. Performance requirements for the analysis are also defined in U.S. Environmental Protection Agency (EPA) methods published in EPA SW-846 and laboratory standard operating procedures. The USEPA Contract Laboratory Program (CLP) National Functional Guidelines for Organic Data Review and Inorganic Data Review (EPA-540/R-94-012 and EPA-540/R-94-013, respectively) may be used for supporting information during the verification and validation, however, the chemical analyses are required to be in compliance with EPA SW-846 protocols.

The results of the data verification and validation are documented in a report that includes a summary narrative with discussion of any anomalies and resolutions. Data anomalies include data points reported as being below the method detection limit or otherwise censored over a specific range of values, missing data points occurring randomly in the data set, and outliers associated with Resource Conservation and Recovery Act (RCRA) regulated constituents as identified in the Hazardous Waste Facility Permit (HWFP). If a determination is made that there is statistically significant evidence of contamination, notification to the New Mexico Environment Department (NMED) will be made in accordance with WP 02-PC.03, WIPP Hazardous Waste Facility Permit Reporting and Notifications Compliance Plan. Changes in the general water chemistry as it affects the predicted repository performance, are evaluated by Sandia National Laboratory.

One or more of the following records are generated by the use of this procedure.

- Verification and Validation Report including:
  - Summary Narrative
  - Attachment 2, Preliminary Review
  - Attachment 3, Alkalinity Data Review
  - Attachment 4, Anions (Chloride, Sulfate, and Nitrate) Data Review
  - Attachment 5, Nitrate by Spectrophotometric Cadmium Reduction Data Review
  - Attachment 6, Total Kjeldahl Nitrogen Data Review
  - Attachment 7, pH Data Review

- Attachment 8, Specific Gravity Data Review
- Attachment 9, Specific Conductance Data Review
- Attachment 10, Total dissolved Solids Data Review
- Attachment 11, Total Suspended Solids Data Review
- Attachment 12, Total Organic Carbon Data Review
- Attachment 13, Total Organic Halogen Data Review
- Attachment 14, Metals Data Review
- Attachment 15, Volatile Organic Data Review
- Attachment 16, Semivolatile Organic Data Review

- Statistical Analysis
- Telephone Conference Log(s)

REFERENCES

BASELINE DOCUMENTS

- 40 Code of Federal Regulations (CFR) Part 264, Subpart F, "Releases from Solid Waste Management Units"
- 40 CFR Part 265, Subpart F, "Groundwater Monitoring"
- DOE Order 450.1, *Environmental Protection Program*
- DOE/WIPP 98-2285, *Waste Isolation Pilot Plant RCRA Background Groundwater Quality Baseline Report*
- DOE/WIPP 99-2194, *Waste Isolation Pilot Plant Environmental Monitoring Plan*
- *Waste Isolation Pilot Plant RCRA Background Groundwater Quality Baseline Report*, Addendum-1, July 2000
- WP 13-1, Washington TRU Solutions LLC Quality Assurance Program Description
- WP 15-PR, WIPP Records Management Program

REFERENCED DOCUMENTS

- Hazardous Waste Facility Permit, Waste Isolation Pilot Plant, Permit No. NM4890139088-TSDF, issued by the New Mexico Environment Department
- EPA-540/R-94-012, *EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*
- EPA-540//R-94-013, *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*
- EPA SW-846, *Test Methods for Evaluating Solid Waste Physical/Chemical Methods*, Methods 6010B, 7000, 7470, 8000B, 8260B, and 8270C.
- EPA Method 120.1, *Conductivity*
- EPA Method150.1, *pH*
- EPA Method 160.1, *Residue, Filterable (Gravimetric, Dried at 180 °C)*
- EPA Method 160.2, *Residue, Non-Filterable (Gravimetric, Dried at 103 - 105 °C)*
- EPA Method 300.0, *Determination of Inorganic Anions by Ion Chromatography*
- EPA Method 310.1, *Alkalinity (Titrimetric, pH 4.5)*
- EPA Method 351.1, *Nitrogen, Kjeldahl Total (Colorimetric, Automated Phenate)*
- EPA Method 353.3, *Nitrogen, Nitrate-Nitrite (Spectrophotometric, Cadmium Reduction)*
- EPA Method 415.1, *Total Organic Carbon in Water (Combustion or Oxidation)*
- EPA Method 9020B, *Total Organic Halides (TOX)*
- EPA Standard Method 4500-NO_3, *Nitrogen (Nitrate)*
- *Statistical Analysis of Ground-Water Monitoring Data at RCRA Facilities*, Interim Final Guidance. EPA. April 1989
- *Statistical Analysis of Ground-Water Monitoring Data at RCRA Facilities*, Addendum to Interim Final Guidance. EPA. July 1992
- ASTM D854-92, *Density*

- WP 02-1, WIPP Groundwater Monitoring Program Plan
- WP02-PC3002, *WIPP Hazardous Waste Facility Permit Change Request and Modification Processing*
- WP 02-PC.03, WIPP Hazardous Waste Facility Permit Reporting and Notifications Compliance Plan
- WP15-PC3041, Approval/Variation Request Processing

PRECAUTIONS AND LIMITATIONS

- Personnel performing verification and validation of inorganic and organic data shall have at least two years of experience with EPA SW-846 methods including generating, compiling, and reporting metals and/or organic analytical data and must be familiar with the USEPA CLP National Functional Guidelines for Organic Data Review and the USEPA CLP National Functional Guidelines for Inorganic Data Review (EPA-540/R-94-012 and -013). (Inorganic implies metals for these guidelines)
- Environmental Monitoring & Hydrology (EM&H) Manager shall be contacted if this procedure cannot be performed as written.
- EM&H Manager shall be contacted if abnormal conditions are found during the performance of this procedure.
- Acceptance criteria shall be consistent with the following EPA methods (or equivalent) identified in subsequent sections of this procedure.
- A contract laboratory submitting data with errors will be required to correct errors and resubmit the data. If any data quality objectives were not met that could affect the usability of the data, the laboratory may be required to reanalyze the samples. The only exception to this situation is when the sample matrix precludes achieving the data quality objectives on the associated Quality Control (QC) duplicate and matrix spike samples in which case, appropriate allowances will be made and documented.
- Holding times are determined by comparing the sampling date on the chain-of-custody (COC) form with the dates of analysis on the laboratory analysis summary and in the laboratory raw data.
- Sample integrity can be assumed if the Sample Delivery Group (SDG) narrative or the sample records do not indicate problems. If problem(s) are detected, then sample integrity may have been compromised. In this case, validators/verifiers will use their judgment to evaluate the possible effects on sample analysis.

PREREQUISITE ACTIONS

- This verification and validation applies to compliance data as defined in Attachment 1. Verify that Attachment 1 is current based on changes that may have been requested in accordance with WP 02-PC3002.
- Obtain complete data package for verification and validation and print Attachments 2 - 16 of this procedure.
- Obtain baseline report with 95 percent upper tolerance limit value (UTLV) or 95th percentile for constituents listed in Attachment 1.

PERFORMANCE

1.0 PRELIMINARY DATA REVIEW

1.1 Complete Section A of Attachment 2.

1.2 Perform review of data package and place a check (✓) mark in the appropriate column (YES or NO) in Sections B and C of Attachment 2. Record N/A for any item not applicable and provide justification. Section C may be completed after completion of analysis-specific checklist.

1.3 **IF** one or more items are either missing or incomplete,
THEN notify the cognizant laboratory representative via telephone **AND** request the missing items.

1.3.1 Document phone conversation on telephone conference log.

1.3.2 Contact EM&H manager if deficiencies cannot be resolved.

1.3.3 When data package is determined to be complete **GO TO** Step 1.4.

1.4 Determine the type of data review required based on data package contents, and **GO TO** the appropriate section listed below:

- Section 2.0, Indicator Parameters Review
- Section 3.0, Metals Data Review
- Section 4.0, Volatile Organic Data Review
- Section 5.0, Semivolatile Organic Data Review

2.0 INDICATOR PARAMETERS REVIEW

NOTE

The following references are applicable to indicator constituents and parameters data review and evaluation: EPA Method 310.1, EPA Method 300.0, EPA Method 351.1, EPA Method 150.1, EPA Method 120.1, EPA Method 160.1, EPA Method 351.1, EPA Method 353.3 or Equivalent Standard Method 4500-NO_3, EPA Method 415.1, EPA Method 9020B, EPA Method 160.2, and ASTM D854-92.

2.1 Review the data package and place a check (✓) mark on Attachments 3 - 13 for each question in the appropriate column (YES or NO). Record N/A for any section or question that is not applicable and provide justification.

2.2 Evaluate and document observed discrepancies on Attachments 3 -13.

2.3 User shall either qualify or reject the data and document conclusion in the narrative summary of the Verification and Validation Report.

3.0 METALS DATA REVIEW

NOTE

The following references are applicable to metals data review and evaluation: EPA SW-846 Methods 6010B, *Inductively Conducted Plasma-Atomic Emission Spectrometry* for metals analysis; 7470A *Mercury in Liquid Waste* (Manual Cold-Vapor Technique) for mercury analysis including Method 7000B, *Flame Atomic Absorption Spectrophotometry*; and/or EPA-540/R-94-013.

3.1 Review the data package and place a check (✓) mark on Attachment 14 for each question in the appropriate column (YES or NO). Record N/A for any section or question in Attachment 14 that is not applicable, and provide justification.

3.2 Evaluate and document observed discrepancies on Attachment 14.

3.3 User shall either qualify or reject the data and document conclusion in the narrative summary of the Verification and Validation Report.

4.0 VOLATILE ORGANIC DATA REVIEW

NOTE

The following references are applicable to volatile organic data review and evaluation: EPA SW-846 Method 8260B, *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)* including the supplemental information in Method 8000B, *Determinative Chromatographic Separations*, if necessary and/or EPA-540/R-94-012.

4.1 Review the data package and place a check (✓) mark on Attachment 15 for each question in the appropriate column (YES or NO). Record N/A for any section or question in Attachment 15 that is not applicable, and provide justification.

4.2 Evaluate and document observed discrepancies on Attachment 15.

4.3 User shall either qualify or reject the data and document conclusion in the narrative summary of the Verification and Validation Report.

5.0 SEMIVOLATILE ORGANIC DATA REVIEW

NOTE

The following references are applicable to semivolatile organic data review and evaluation: EPA SW-846 Method 8270C, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)* including the supplemental information in Method 8000B, *Determinative Chromatographic Separations*, if necessary and/or EPA-540/R-94-012.

5.1 Review the data package and place a check (✓) mark on Attachment 16 for each question in the appropriate column (YES or NO). Record N/A for any section or question in Attachment 16 that is not applicable, and provide justification.

5.2 Evaluate and document observed discrepancies on Attachment 16.

5.3 User shall either qualify or reject the data and document conclusion in the narrative summary of the Verification and Validation Report.

6.0 OUTLIER AND POTENTIAL CONTAMINATION DETERMINATION

NOTE

Statistical tests for outliers and evidence of contamination are performed for constituents listed in Attachment 1.

6.1 For each of the constituents listed in Attachment 1, identify qualified data points that exceed the 95 percent UTLV or 95th percentile depending on the distribution type. If no data points meet this criteria, report as such in the Verification and Validation Report and proceed to Section 7.0.

6.2 Data points that meet the criteria in Step 6.1 shall be compared with the baseline groundwater quality utilizing methods described in *Statistical Analysis of Ground-Water Monitoring Data at RCRA Facilities* (EPA 1989 and EPA 1992). This test will be used to determine if the data point is an outlier or represents a statistically significant evidence of contamination. Prepare statistical results for inclusion in the Verification and Validation Report.

7.0 REPORTING REQUIREMENTS

NOTE

The WIPP Groundwater Detection Monitoring Semiannual Report, which includes the data Verification and Validation Report, is submitted to the NMED within 120 days after the last sample was collected in accordance with WP 02-PC.03. The semiannual report provides the record copy of the data Verification and Validation Report as required in the HWFP.

NOTE

The Permittees are to notify the NMED Secretary in writing of statistically significant evidence of contamination within seven calendar days of discovery. Discovery is defined as completion of peer review of the draft Verification and Validation Report. Reporting is performed in accordance with WP 02-PC.03. Resampling of wells found to have potential contamination is conducted in accordance with the HWFP, Module V.

7.1 Within 90 days following the date the last sample was collected, complete the data Verification and Validation Report. An example outline of this report is provided in Attachment 17.

7.2 Perform one of the following:

7.2.1 If data verification and validation has been contracted, distribute the data Verification and Validation Report to EM&H Manager, Field Team Leader, Quality Assurance (QA) Manager, and Subcontract Technical Representative (STR) in accordance with WP15-PC3041.

7.2.2 If data verification and validation has been done internally, obtain EM&H Manager or designee review and approval as indicated by signature and date on the report, and distribute the data Verification and Validation Report to the EM&H Manager, Field Team Leader, and QA Manager.

Attachment 1 - WIPP Groundwater Monitoring Program Parameters and Constituents

Indicator Parameters		
Total Dissolved Solids	Calcium	Alkalinity
Total Suspended Solids	Iron	Nitrate (as N)
Total Organic Carbon	Magnesium	Chloride
Total Organic Halogens	Potassium	Sulfate
pH	Sodium	
Density		
Specific Conductance		
Constituents		
Volatile Organic Compounds	**Semivolatile Organic Compounds**	**Trace Metals**
Isobutanol	1,2-Dichlorobenzene	Antimony
Carbon tetrachloride	1,4-Dichlorobenzene	Arsenic
Chlorobenzene	2,4-Dinitrophenol	Barium
Chloroform	2,4-Dinitrotoluene	Beryllium
1,1-Dichloroethane	Hexachlorobenzene	Cadmium
1,2-Dichloroethane	Hexachloroethane	Chromium
1,1-Dichloroethylene	Cresols (2-, 3-, & 4-Methylphenol)	Lead
cis-1,2-Dichloroethylene	Nitrobenzene	Mercury
trans-1,3-Dichloroethylene	Pentachlorophenol	Nickel
Methyl ethyl ketone	Pyridine	Selenium
Methylene chloride		Silver
1,1,2,2-Tetrachloroethane		Thallium
Tetrachloroethylene		Vanadium
1,1,1-Trichloroethane		
1,1,2-Trichloroethane		
Toluene		
Trichloroethylene		
Trichlorofluoromethane		
Vinyl chloride		
Xylenes		

Attachment 2 - Preliminary Review

SECTION A

Sample Location: _____

Laboratory Name: _____

Laboratory Work Order #: _____

Sample Number(s): _____

Reviewer Name (print): _____

Reviewer Signature: _____

SECTION B	YES	NO
Is the SDG narrative present and certification page signed by an authorized representative of the laboratory?		
Does the SDG narrative explain problems associated with processing and analysis of the samples?		
Were the original or copy of the COC and the Request for Analysis (RFA) present and complete?		
Is the sample analysis summary table present?		
Were sufficient data as required by the Statement of Work (SOW) available to evaluate the analysis results?		
Is the electronic data package present and in the proper format?		

SECTION C GENERAL VERIFICATION AND VALIDATION	YES	NO
Were data entries free of transcription error?		
Were calculations correct?		
Were the correct number of significant figures applied to reported data?		
Were trip (VOC only), field and method blanks (MB) free of contaminants?		
Note: The consistent detection of any anthropogenic compounds should be reported to the EM&H Manager, as well as documented and discussed in the Verification and Validation Report.		
Were any anthropogenic compounds starting to appear consistently in the samples?		
Were concentration results consistently reported in units of micrograms per liter (ug/L)?		
Were the applicable EPA SW-846 procedures used?		
Note: The EM&H Manager should be contacted if the data completeness objective of 100% is not met for any Verification and Validation Report since resampling and/or reanalysis of detection monitoring program (DMP) samples may be required.		
Were 100% of the samples usable and valid?		

Attachment 3 - Alkalinity Data Review

HOLDING TIME	YES	NO
Were the samples preserved at <6°C?		
Were the samples analyzed within 14 days of collection?		

REAGENTS	YES	NO
Was the pH meter standardized with at least two certified buffer solutions?		
Were the pH measurements within 98-102% of the buffer concentration?		
Was a standardized acid titrant used for the analyses?		

ANALYSIS QUEUE	YES	NO
Was a MB analyzed with the sample set?		
Was an Initial Calibration Verification (ICV) analyzed prior to the samples?		
Did the ICV meet the accuracy criteria of 90-110% recovery?		
Was a duplicate sample analyzed with the sample set?		
Was the duplicate precision ≤20 RPD (relative percent difference)?		

Attachment 4 - Anions (Chloride, Sulfate, and Nitrate) Data Review

Note: Nitrate may alternatively be measured by Spectrophotometric Cadmium Reduction Method 353.3, or Equivalent.

HOLDING TIMES	YES	NO
Were the samples preserved at <6°C?		
Were the samples analyzed within 28 days of collection (48 hours for nitrate)?		

ANALYSIS QUEUE	YES	NO
Was a 5-point calibration curve analyzed for both target anions?		
Was an ICV analyzed at the beginning of the sample queue?		
Was a Continuing Calibration Verification (CCV) analyzed periodically throughout the sample set?		
Was a MB analyzed with the sample set?		
Did the sample queue contain a duplicate sample?		
Was a Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) analyzed with the sample set?		
Was a matrix spike/matrix spike duplicate (MS/MSD) analyzed with the sample set?		
Was a DMP sample used for the MS/MSD?		

METHOD PERFORMANCE	YES	NO
Was the MB concentration of the anions <Reporting Limit?		
Did the ICV and CCV analyses meet the accuracy criteria of 90-110% recovery?		
Did the duplicate precision meet the ≤20 RPD criterion for the anions?		
Did the LCS/LCSD analyses meet the accuracy criteria of 90-110% recovery?		
Did the MS/MSD analyses meet the accuracy criteria of 80-120% recovery?		

QUANTITATION	YES	NO
Were any of the samples diluted to bring the anion concentrations within the calibration range of the instrument?		
Were the appropriate dilution factors applied to the results?		

Attachment 5 - Nitrate by Spectrophotometric Cadmium Reduction Data Review

Note: Nitrate may alternatively be measured by Anion analysis Method 300.0 along with chloride and sulfate.

HOLDING TIMES	YES	NO
Were the samples maintained at <6°C prior to analysis?		
Were the samples analyzed within 24 hours of collection or preserved with sulfuric acid and refrigerated for later analysis?		

CALIBRATION	YES	NO
Was a standard curve prepared with at least three points?		
Was the correlation coefficient for the curve at least 0.995?		
Was a nitrite curve analyzed without the cadmium reduction step for use in subtracting out nitrite to determine nitrate?		

ANALYSIS QUEUE		
Did the analysis queue include an ICV, LCS, LCSD, MS, MSD, and CCV?		
Was a DMP sample used for the MS/MSD?		

METHOD PERFORMANCE (a)		
Was the accuracy objective of 90-110% recovery met for the ICV and CCV?		
Was the accuracy objective of 80-120% recovery met for the LCS and LCSD?		
Was the precision objective of $\leq$20RPD met for the LCS/LCSD?		
Was the accuracy objective of 70-130% recovery met for the MS and MSD?		
Was the precision objective of $\leq$30RPD met for the MS and MSD?		

(a) **Note** that EPA Method 353.3 does not define data quality objectives for the accuracy and precision of the data. The method performance limits are based on data review experience with Method 353.3 and other EPA Methods for Chemical Analysis of Water and Wastes.

Attachment 6 - Total Kjeldahl Nitrogen Data Review

HOLDING TIMES	YES	NO
Were the samples maintained at <6°C prior to analysis?		
Were the samples analyzed within 24 hours of collection or preserved with sulfuric acid and refrigerated for later analysis within 28 days?		

CALIBRATION	YES	NO
Were five (5) calibration points used for the initial calibration?		
Was the correlation coefficient for the curve at least 0.995?		
Was an ICV from a separate source analyzed prior to the samples?		
Was a CCV analyzed prior to the samples?		

ANALYSIS QUEUE		
Were initial and continuing calibration blanks analyzed?		
Was a MB analyzed with the samples?		
Was one of the samples analyzed in duplicate?		
Was a LCS analyzed with the samples?		
Was a MS analyzed with the samples?		

METHOD PERFORMANCE		
Was the accuracy objective of 90-110% recovery met for the ICV and CCV?		
Were the blank analysis results less than the detection limit of 0.10 mg/L?		
Was the accuracy objective of 80–120% recovery met for the LCS?		
Was the accuracy objective of 75-125% recovery met for the MS?		
Was the precision objective of $\leq$20RPD met for the duplicate sample analysis?		

Attachment 7 - pH Data Review

HOLDING TIME	YES	NO
Were the samples preserved at <6°C?		
Were the samples analyzed within one day of receipt?		

CALIBRATION	YES	NO
Were the samples allowed to warm to room temperature prior to analysis?		
Was the temperature recorded for each pH measurement?		
Were at least two standard buffer solutions used to calibrate the instrument?		

SAMPLE ANALYSIS	YES	NO
Did the standard buffers bracket the pH range of the samples?		
Was the sample analysis temperature within 2°C of the buffer analysis temperature?		
Was the pH recorded to the nearest 0.1 pH unit?		
Did the buffer checks yield 98-102% accuracy following calibration?		
Was the pH of one of the samples measured in duplicate?		

Attachment 8 - Specific Gravity Data Review

SAMPLE ANALYSIS	YES	NO
Were the samples allowed to warm to near room temperature?		
Was the temperature of the density measurements recorded?		
Was the mass of at least 5.0 mL of the water sample measured?		
Were the weights recorded to the nearest 0.1 mg?		

Attachment 9 - Specific Conductance Data Review

HOLDING TIME	YES	NO
Were the samples maintained at <6°C prior to analysis?		
Were the samples allowed to warm to room temperature prior to analysis?		
Were the samples analyzed within 28 days of collection?		

CALIBRATION	YES	NO
Was the instrument calibrated with 0.01N KCl?		
Was the temperature of each measurement recorded?		
Was the proper temperature correction factor applied to the conductivity measurements?		

ANALYSIS QUEUE	YES	NO
Was an ICV analyzed prior to the samples?		
Did the ICV consist of 0.1 N KCl?		
Did the ICV provide a conductivity reading between 90-110% of 1412 µmhos/cm?		
Was a second source KCl solution used for the CCV?		
Was one of the samples analyzed in duplicate?		

METHOD PERFORMANCE	YES	NO
Did the CCV analyses yield a concentration within 90-110% of 1412 µmhos/cm?		
Did the duplicate precision meet the ≤20 RPD criterion?		

Attachment 10 - Total Dissolved Solids Data Review

HOLDING TIME	YES	NO
Were the samples maintained at <6°C prior to analysis?		
Were the samples analyzed within 7 days of collection?		

ANALYSIS QUEUE	YES	NO
Was an ICV analyzed at the beginning of the sample queue?		
Was a MB analyzed with the sample set?		
Was a CCV analyzed every 10 samples?		
Did the ICV and CCV consist of two different sources of NaCl?		
Was a MB analyzed with the sample set?		
Was the difference in the first and second weights of the dried residues <0.5 mg?		

METHOD PERFORMANCE	YES	NO
Did the ICV and CCV analyses meet the accuracy criteria of 90-110% recovery?		
Was the RPD of the duplicate analyses $\leq$10?		

Attachment 11 - Total Suspended Solids Data Review

HOLDING TIME	YES	NO
Were the samples maintained at <6°C prior to analysis?		
Were the samples analyzed within 7 days of collection?		

ANALYSIS QUEUE	YES	NO
Was a MB analyzed with the sample set?		
Were a LCS/LCSD analyzed with the sample set?		
Was one of the samples analyzed in duplicate?		

METHOD PERFORMANCE	YES	NO
Was the MB result <1.0 mg/L?		
Did the LCS and LCSD analyses meet the 90-110% recovery accuracy criteria?		
Was the precision of the duplicate analyses ≤20 RPD?		

Attachment 12 - Total Organic Carbon Data Review

HOLDING TIME	YES	NO
Were the samples maintained at <6°C prior to analysis?		
Were the samples preserved with acidification to pH<2?		
Were the samples analyzed within 28 days of collection?		

ANALYSIS QUEUE	YES	NO
Was the instrument calibrated daily with a 5-point calibration curve?		
Was an ICV analyzed using a second source standard?		
Was a MB analyzed with the sample set?		
Was a CCV analyzed after every 10 samples and at the end of the queue?		
Were Initial Calibration Blanks (ICB) and Continuing Calibration Blanks (CCB) analyzed after the ICV and CCV?		
Were quadruplicate measurements taken for each sample?		
Were both the average and the range reported for the quadruplicate measurements?		
Was one of the samples analyzed in duplicate?		
Was at least one lab control spike analyzed?		
Was at least one matrix spike analyzed?		
Was a DMP sample used for the matrix spike?		

METHOD PERFORMANCE	YES	NO
Did the calibration curve provide a 0.995 correlation coefficient?		
Did the ICV meet the accuracy criteria of 90-110% recovery?		
Did the CCVs meet the 90-100% recovery accuracy criteria?		
Were the results for the MB, the ICB and CCB less than the low calibration standard?		
Was the precision of the duplicate analyses ≤20 RPD? (for samples with concentrations >5 times the low calibration standard)		
Did the lab control sample meet the accuracy criteria of 80-120% recovery?		
Did the matrix spike meet the accuracy criteria of 75-125% recovery?		

Attachment 13 - Total Organic Halogen Data Review

HOLDING TIME	YES	NO
Were the samples collected with zero headspace?		
Were the samples preserved with acidification to pH<2 with sulfuric acid?		
Were the samples stored at <6°C?		
Were the samples analyzed within 28 days of collection ?		

ANALYSIS QUEUE	YES	NO
Were duplicate calibration standards analyzed each day?		
Were duplicate calibration blanks analyzed each day?		
Were at least two MBs analyzed to establish the repeatability of the method background?		
Were all samples analyzed in duplicate?		
Were a LCS/LCSD analyzed with the sample set?		
Was a matrix spike analyzed with the sample set?		
Was a DMP sample used for the matrix spike ?		

METHOD PERFORMANCE	YES	NO
Was >90% of the TOC on the front column for each standard?		
Was >90% of the TOC on the front column for each sample?		
Was the precision of the duplicate sample analyses <20 RPD?		
Did the lab control sample meet the accuracy criteria of 80-120% recovery?		
Did the matrix spike sample meet the accuracy criteria of 70-130% recovery?		

Attachment 14 - Metals Data Review

HOLDING TIMES	YES	NO
Based on the COC and/or SDG narrative, were aqueous samples preserved to ≤ pH2 with nitric acid?		
Were holding times met for all sample digestion/analysis? Maximum holding time is 28 days for Mercury and 6 months for metals. (EPA SW-846, Table 3-2).		
Was the integrity of each sample intact?		
Were the analysis dates on the laboratory analysis summary form and the raw data sheets identical?		

INDUCTIVELY COUPLED PLASMA (ICP) CALIBRATION	YES	NO
Was a calibration blank and at least one calibration standard used for daily calibration and each setup?		
Was the initial ICV analyzed immediately after calibration and prior to sample analysis?		
Was the ICV prepared from a second source standard and at a mid-range concentration of the calibration curve?		
Did the ICV analysis meet the accuracy criteria of 90 - 110% recovery for all target metals?		
Did the ICV analysis meet the accuracy criteria of 90 - 110% recovery for all target metals?		
Were CCV standards analyzed at a frequency of every 10 samples and at the end of the sample queue?		
Did the CCV analysis meet the accuracy criteria of CCV results within 90 - 110% recovery for all target metals?		
Did the recalculated percent recovery (%R) values agree within 1% of the reported values?		

Attachment 14 - Metals Data Review

ICP BLANKS	YES	NO
Was an ICB analyzed immediately after the ICV?		
Were CCBs analyzed in the sample queue every 10 samples, immediately after the CCV, to evaluate drift, sensitivity, and contamination?		
Were the ICB, CCB, and PB analysis results all <RL (Reporting Limit)?		
Was a field blank analyzed (minimum frequency of 20 samples)?		

ICP INTERFERENCE CHECK SAMPLE	YES	NO
Were Interference Check Standards (ICA & ICSAB) analyzed daily prior to sample analysis and near the end of the sample queue?		
Were any target metals detected in the samples at a concentration >Instrument Detection Limit (IDL) that were not present in the Interference Check Sample (ICS)?		
Were the recoveries for all the target metals in the range of 80 - 120% of true value?		
Did the recalculated percent recovery (%R) values agree within 1% of the reported values?		

ICP LABORATORY CONTROL SAMPLES	YES	NO
Were LCS/LCSD prepared for each SDG batch of $\leq$20 samples?		
Were the LCS/LCSD recoveries within the range of 80 - 120% for each metal analyte?		
Did the recalculated percent recovery (%R) of one of the target metals agree within 1% of the reported values?		

ICP DUPLICATE ANALYSIS	YES	NO
Was at least one sample analyzed in duplicate for each SDG batch?		
Was the precision objective of $\leq$20 RPD met for all target metals detected in the duplicate samples?		
Did the recalculated RPD of one of the target metals agree within 1% of the reported values?		

Attachment 14 - Metals Data Review

ICP MATRIX SPIKED SAMPLE ANALYSIS	YES	NO
Was a matrix spike sample analyzed for each SDG batch?		
Was a Washington TRU Solutions LLC (WTS) sample used for the matrix spike?		
Did the matrix spike recovery meet the 75 - 125% recovery objective?		
Was a post-digestion spike analysis performed on an aliquot of the sample used for the matrix spike to check recoveries in case the matrix spike recovery does not meet the recovery objective?		
Did the post-digestion spike meet the 80 - 120% recovery objective?		
Did the recalculated percent recovery (%R) of the spiked metals agree within 1% of the reported values ?		

COLD VAPOR ATOMIC ABSORPTION MERCURY ANALYSIS	YES	NO
Was the instrument calibrated daily for mercury with a minimum of 3 calibration standards and a calibration blank?		
Did the calibration curve meet the linearity criteria of a 0.995 correlation coefficient ®?		
Was the ICV standard prepared from a second source standard and at a mid-range concentration of the calibration curve.		
Did the ICV analysis meet the accuracy criteria of 90-110% recovery?		
Were CCV standards analyzed at a frequency of every 10 samples and at the end of the sample queue?		
Did the CCV analyses meet the accuracy criteria of 80-120% recovery?		
Was an LCS/LCSD prepared for each SDG batch of $\leq$20 samples?		
Were the LCS/LCSD recoveries within the range of 80-120% recovery?		
Was a MS/MSD analyzed for each SDG batch?		
Did the MS/MSD analyses meet the accuracy criteria of 80-120% recovery?		
Did the recalculated percent recovery (%R) of the lab control spike and matrix spike samples agree within 1% of the reported values?		

Attachment 15 - Volatile Organic Data Review

HOLDING TIMES	YES	NO
Based on the COC and/or SDG narrative, were aqueous samples preserved to ≤ pH2 with hydrochloric acid (HCl)?		
Based on the COC and/or SDG narrative, were the samples received and maintained at ≤ 6° C?		
Was the holding time of 14 days met for all sample?		
Were the analysis dates on the laboratory analysis summary form and the raw data sheets identical?		
Was the integrity of each sample intact?		

GC/MS INSTRUMENT PERFORMANCE CHECK	YES	NO
Was a BFB tuning compound analysis performed at the beginning and every 12 hours during VOC instrument operation?		
Was the calibration mass assignment correct?		
Was the ion abundance listing normalized to m/z 95?		
Were the ion abundance criteria met?		

Attachment 15 - Volatile Organic Data Review

INITIAL CALIBRATION	YES	NO
Were a minimum of 5 calibration standards used for the initial calibration?		
Was the low standard at or below the RL for the method, e.g., 5.0 ug/L?		
Is the precision of the Relative Response Factors (RRF) over the calibration range ≤15% relative standard deviation (RSD)?		
Were all RRFs ≥ 0.05 for all target analytes and the System Monitoring Compounds (SMCs)?		
If sample results were calculated using the initial calibration curve were samples analyzed within 12 hours of the associated instrument performance checks?		
Note: The %RSD check shall be completed for one or more volatile target compounds associated with each internal standard.		
Note: If the %RSD for any of the response factors is greater than 30%, judgment should be used to determine the need to check the points on the curve for the cause of the nonlinear response. The verification may be performed by eliminating either the high point or the low point and recalculating the %RSD. Document calculation and the basis for changes in validation and verification report.		
Were recalculated #RSDs of the response factors ≤30%?		
If sample results were calculated using an initial ICV standard, do the reported RFFs of the ICV agree to within 20 percent difference (bias) of the average RRFs from the initial calibration?		
Did recalculated avg. RRFs agree within 1% with the laboratory reported values? This check shall be completed for the RRFs from the initial calibration curve of one or more volatile target compounds associated with each internal standard.		
Did recalculated %RSDs agree within 1% of the laboratory reported value(s)? This check shall be completed for the RRFs from the initial calibration curve of one or more volatile target compounds associated with each internal standard.		

CONTINUING CALIBRATION	YES	NO
Were CCV standards analyzed prior to samples and every 12 hours thereafter during instrument operation?		
Note: The RRF and percent difference checks shall be completed for one or more volatile target compound(s) associated with each internal standard.		
Did the reported RRFs of the CCVs agree to within 20 percent difference of the average RRFs from the initial calibration?		
Did recalculated RRFs in the CCVs agree within 1% with the laboratory reported values?		
Did recalculated percent differences agree to within 1% of the laboratory reported values?		

Attachment 15 - Volatile Organic Data Review

BLANKS	YES	NO
Was a MB prepared for each 12-hour period of GC/MS instrument operation?		
Note: Review shall include both forms and raw data chromatograms.		
If any contaminants were detected in the MB samples, were they present at concentrations <RL?		
Were instrument blanks analyzed following any instances when target compounds were detected at concentrations above the linear range of the GC/MS instrument?		
Was a trip blank (one per shipment) analyzed?		
Was a field blank analyzed (minimum frequency of 20 samples)		

SYSTEM MONITORING COMPOUNDS	YES	NO
Note: Flag the data if SMCs are outside criteria with no evidence of sample reanalysis.		
Were a minimum of three (3) SMCs added to all samples and blanks prior to GC/MS analysis?		
Were SMC recoveries reported on the SMC recovery form consistent with the raw data?		
Did the SMC recoveries meet the 70-130% recovery objective or the QC recovery chart limits established historically by the laboratory?		
Did the lab reanalyze any samples within the required holding time when the SMC recovery objective was not met?		
Note: This check shall be completed for one or more SMCs.		
Did recalculated recoveries agree within 1% with the laboratory reported values?		

Attachment 15 - Volatile Organic Data Review

MATRIX SPIKE/MATRIX SPIKE DUPLICATE	YES	NO
Were a MS/MSD prepared and analyzed for each SDG batch?		
Were the MS/MSD samples spiked with all the target VOC compounds?		
Was a DMP sample used to generate the MS/MSD samples?		
Were the MS/MSD recoveries calculated and reported correctly?		
Did the MS/MSD recoveries for target DMP target VOCs meet the recovery objective specified in the EPA Functional Guidelines? (60 - 140% recovery) or QC recovery control chart limits established historically by the laboratory.		
Did the precision of the MS/MSD recoveries meet the precision objective of ≤30 RPD?		
Note: This check shall be completed for one or more spike compounds.		
Did recalculated recoveries agree within 1% with the laboratory reported values?		

LABORATORY CONTROL SAMPLES	YES	NO
Were a LCS/LCSD prepared and analyzed for each SDG batch of ≤20 samples?		
Were the LCS/LCSD samples spiked with all the DMP target VOC compounds?		
Did the LCS/LCSD recoveries meet the 70 -130% recovery objective or QC recovery control chart limits established historically by the laboratory.?		
Did the precision of the LCS/LCSD recoveries meet the precision objective of ≤20 RPD?		
Note: This check shall be completed for one or more LCS/LCSD compounds.		
Did recalculated recoveries agree to within 1% of the laboratory reported values?		

INTERNAL STANDARDS	YES	NO
Were the retention times of the internal standards within ±30 seconds of the retention times of the internal standards in the associated CCV?		
Were the areas of the internal standards within -50% to +100% of the areas in the associated CCV?		

Attachment 15 - Volatile Organic Data Review

TARGET COMPOUND IDENTIFICATION	YES	NO
Were the Relative Retention Times (RRTs) of the target VOC compounds detected in the samples within ±0.06 RRT units of the RRT of the compounds in the nearest CCV?		
Did the relative intensities of the characteristic mass spectral ions agree within 30% of the relative intensities of these ions in the reference mass spectra?		
Were all the major peaks in the GC/MS total ion chromatogram identified either on the chromatogram or on the associated quantitation report?		

TENTATIVELY IDENTIFIED COMPOUNDS	YES	NO
Was a reverse search of the mass spectral library performed on all samples and blanks?		
Were any VOC Tentatively Identified Compounds (TICs) detected in the samples?		
Note: When a low-level nontarget compound that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks that are less than 10% of the internal standard height, but present in the blank chromatogram at similar RRTs.		
If any TICs were detected in the samples, were they also present in the MBs at similar concentrations?		

Attachment 16 - Semivolatile Organic Data Review

HOLDING TIMES	YES	NO
Based on the COC and/or SDG narrative, were the samples received and maintained at ≤ 6° C?		
Were the samples extracted within 7 days of sample collection?		
Were the extracts analyzed within 40 days of sample extraction?		
Were the analysis dates on the laboratory analysis summary form and the raw data sheets identical?		
Was the integrity of each sample intact?		

GC/MS INSTRUMENT PERFORMANCE CHECK	YES	NO
Was a DFTPP tuning compound analysis performed at the beginning and every 12 hours during semivolatile organic compounds (SVOC) instrument operation?		
Was the mass assignment correct?		
Was the ion abundance listing normalized to m/z 198?		
Were the ion abundance criteria met?		

Attachment 16 - Semivolatile Organic Data Review

INITIAL CALIBRATION	YES	NO
Were a minimum of 5 (five) calibration standards used for the initial calibration?		
Was the low standard at or below the RL for the method, e.g., 5.0 ug/mL in the standard?		
Is the precision of the RRFs over the calibration range ≤15% relative percent deviation?		
Were all RRFs ≥ 0.05 for all target compounds and surrogates?		
If sample results were calculated using the initial calibration curve were samples analyzed within 12 hours of the associated instrument performance checks?		
Note: The %RSD check shall be completed for one or more semi-volatile target compounds associated with each internal standard.		
Note: If the %RSD for any of the response factors is greater than 30%, judgment should be used to determine the need to check the points on the curve for the cause of the nonlinear response. The verification may be performed by eliminating either the high point or the low point and recalculating the %RSD. Document calculation and the basis for changes in the Verification and Validation Report.		
Were recalculated %RSDs of the response factors ≤ 30%?		
If sample results were calculated using an initial ICV standard, do the reported RFFs of the ICV agree to within 20 percent difference of the average RRFs from the initial calibration?		
Note: The RFFs check shall be completed for the RRFs from the initial calibration curve of one or more semivolatile target compounds associated with each internal standard.		
Did recalculated RRFs for initial calibration and target compounds agree within 1% with the laboratory reported values?		
Note: This check shall be completed for the RSDs from the initial calibration curve of one or more semivolatile target compounds associated with each internal standard.		
Did recalculated %RSDs agree within 1% with the laboratory reported value(s)?		

Attachment 16 - Semivolatile Organic Data Review

CONTINUING CALIBRATION	YES	NO
Were CCV standards analyzed prior to samples and every 12 hours thereafter during instrument operation?		
Did the reported RRFs of the CCVs agree to within 20 percent difference of the average of the RRFs from the initial calibration?		
Note: This check shall be completed for one or more semivolatile targets compounds associated with each internal standard.		
Did recalculated RRFs in the CCVs agree within 1% with the laboratory reported values?		
Did recalculated percent differences agree to within 1% of the laboratory reported values?		

BLANKS	YES	NO
Was a MB extracted along with each SDG batch?		
Note: Review shall include both forms and raw data chromatograms.		
If any contaminants were detected in the MB sample, were they present at concentrations $\leq$RL?		

SURROGATE SPIKES	YES	NO
Were three base-neutral and three acid surrogate spike recovery compounds spiked into the samples prior to extraction?		
Were the surrogate recoveries reported on the surrogate recovery form consistent with the raw data?		
Did the surrogates meet the recovery objective of 15 - 110% for acid surrogates and 30 - 130% for base neutrals or the QC recovery control chart limits established historically by the laboratory?		
Note: Flag the data if surrogates are outside criteria with no evidence of sample re-injection or re-extraction.		
Did the laboratory re-inject or re-extract any samples for which the surrogate recovery objective was not met?		
Note: This check shall be completed for one or more base-neutral and one or more acid spike compounds.		
Did recalculated recoveries agree within 1% with the laboratory reported values?		

Attachment 16 - Semivolatile Organic Data Review

MATRIX SPIKE/MATRIX SPIKE DUPLICATE	YES	NO
Were a MS/MSD prepared and analyzed for each SDG batch?		
Were the MS/MSD samples spiked with all the target SVOC compounds?		
Was a DMP sample used to generate the MS/MSD samples?		
Did the MS/MSD recoveries for the DMP target SVOCs meet the recovery objective of 40 -140% for base neutral compounds and 30 - 130% for acid compounds or the QC recovery control chart limits established historically by the laboratory?		
Was the precision of the MS/MSD recoveries calculated and reported?		
Did the MS/MSD recoveries meet the precision objective of ≤30 RPD?		
Note: This check shall be completed for one or more spike compounds.		
Did recalculated recoveries agree within 1% with the laboratory reported values?		

LABORATORY CONTROL SAMPLES	YES	NO
Were a LCS/LCSD prepared and analyzed for each SDG batch of ≤20 samples?		
Were the LCS/LCSD samples spiked with all the target SVOC compounds?		
Did the LCS/LCSD recoveries meet the recovery objectives of 40 -140% for base neutral compounds and 30 -130% for acidic compounds or the QC recovery control chart recoveries established historically by the laboratory?		
Did the precision of the LCS/LCSD recoveries meet the precision objective of 20 RPD?		
Note: This check shall be completed for one or more spike compounds.		
Did recalculated recoveries agree within 1% with the laboratory reported values?		

INTERNAL STANDARDS	YES	NO
Were a minimum of six internal standards spiked into extracts of samples and blanks prior to GC/MS analysis?		
Were the retention times of the internal standards within ±30 seconds of the retention times of the internal standards in the associated CCV?		
Were the areas of the internal stands within -50% to +100% of the areas in the associated CCV?		

Attachment 16 - Semivolatile Organic Data Review

TARGET COMPOUND IDENTIFICATION	YES	NO
Were the RRTs of the target SVOC compounds detected in the samples within ±0.06 RRT units of the RRT of the compounds in the nearest CCV?		
Did the relative intensities of the characteristic mass spectral ions agree within 30% of the relative intensities of these ions in the reference mass spectra?		
Were all the major peaks in the GC/MS total ion chromatogram identified either on the chromatogram or on the associated quantitation report?		

TENTATIVELY IDENTIFIED COMPOUNDS	YES	NO
Was a reverse search of the mass spectral library performed on all samples and blanks?		
Were any SVOC TICs detected in the samples?		
Note: When a low-level nontarget compound that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks that are less than 10% of the internal standard height, but present in the blank chromatogram at similar RRTs.		
If any TICs were detected in the samples, were they also present in the MBs at similar concentrations?		

Attachment 17 - Example of Data Verification and Validation Report Outline

Results Summary - Identify detects for the target DMP trace metals, volatile organic compounds and semivolatile organic compounds. Identify presence or absence of detects, and tentatively identified compounds. Include table for detects identifying sample number, analyte(s) detected, reported concentration, 95 percent UTLV or 95th percentile, minimum detection limit (MDL) and RL.

Summary for Trace Metals Analysis - Identify elements analyzed and procedures used. List observations and resolutions to discrepancies regarding the quality of the data as determined from the data verification and validation.

Summary for Volatile Organic Analysis - Identify constituents analyzed and procedures used. List observations and resolutions to discrepancies regarding the quality of the data as determined from the data verification and validation.

Summary for Semi-volatile Organic Analysis - Identify constituents analyzed and procedures used. List observations and resolutions to discrepancies regarding the quality of the data as determined from the data verification and validation.

Summary for Indicator Parameter Analysis - Identify analysis completed and procedures used. List observations and resolutions to discrepancies regarding the quality of the data as determined from the data verification and validation.

Potential Outlier and Contamination Determination - Identify presence or absence of data points exceeding 95 percent UTLV or 95^{th} percentile. If a statistical evaluation is required, report results.

Other Miscellaneous Data Package Issues

Attachment 18 - Acronyms/Abbreviations

COC	Chain of Custody
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CFR	Code of Federal Regulations
CLP	USEPA Contract Laboratory Program
DMP	Detection Monitoring Program
EM&H	Environmental Monitoring & Hydrology
EPA	U.S. Environmental Protection Agency
GC/MS	Gas Chromatograph/Mass Spectrometer
HWFP	Hazardous Waste Facility Permit
ICB	Initial Calibration blank
ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
MB	Method Blank (generally equivalent to Preparation Blank)
MDL	Minimum Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NMED	New Mexico Environment Department
QA	Quality Assurance
QC	Quality Control
RCRA	Resource Conservation and Recovery Act
RFA	Request for Analysis
RL	Reporting Limit
RPD	Relative Percent Difference
SDG	Sample Delivery Group
SMC	System Monitoring Compound (equivalent to surrogate recovery compound)
SOW	Statement of Work
STR	Subcontractor Technical Representative
TIC	Tentatively Identified Compounds
UTLV	upper tolerance limit value
WIPP	Waste Isolation Pilot Plant
WTS	Washington TRU Solutions LCC