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CCP-TP-185

Revision 1

CCP Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry

EFFECTIVE DATE: 11/18/2008

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APPROVED FOR USE

RECORD OF REVISION

Revision Number	Date Approved	Description of Revision
0	05/02/2007	Initial Issue.
1	11/18/2008	Revised to support addition of hexachlorobutadiene and bis(2-Ethylhexyl)phthalate as site-specific target analytes for Accelerated Retrieval Project (ARP).

1. ABSTRACT

This Central Characterization Project (CCP) procedure is used for analyzing semivolatile organic compounds (SVOCs) by gas chromatography/mass spectrometry (GC/MS) in extracts prepared from all types of solid waste matrices and soil samples.

2. APPLICABILITY

This procedure is based on Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry," and Method 8000B, "Determinative Chromatographic Separations," from EPA SW-846, Test Methods for Evaluating Solid Wastes, Third Edition.

This method is applicable to analysis of extracts prepared per CCP-TP-187, *CCP Sample Preparation for Semivolatile Organic Compounds.*

This method is suitable for characterization of homogeneous solids and soil/gravel per the requirements of the Waste Isolation Pilot Plant (WIPP) Waste Analysis Plan (WAP). Compounds routinely calibrated and determined by this procedure are listed in Table 2.

This procedure functions as in ICP Use Type 2 document for performing operations within the ALD facilities.

Personnel performing this procedure will be trained and qualified in accordance with CCP-QP-002, *CCP Training and Qualification Plan,* prior to performing this procedure.

3. DISCUSSION

Samples are prepared for analysis by GC/MS using appropriate sample preparation techniques from CCP-TP-187. The SVOCs are introduced into the GC/MS by injecting the sample extract into the GC equipped with a narrow bore fused silica capillary column. The GC column is temperature programmed to separate the analytes, which are then detected by the MS. Identification of the target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like spectra) of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a standard curve.

Samples analyzed are radioactive. Reduced sample sizes (see CCP-TP-187) are used as the best compromise between low method detection levels, minimal radiation exposure to laboratory personnel, and instrument damage.

The following compounds may require special treatment when determined by this method:

- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, and 4-chloro-3-methylphenol are subject to erratic behavior, especially if the GC system is contaminated with high boiling material.

4. SAFETY PRECAUTIONS

4.1 Chemical Hazards

4.1.1 Handle all chemicals per MCP-3635, “Chemical Hygiene Plan.”

4.1.2 Wear the following standard laboratory PPE in the lab:

A. Lab coat

B. Safety glasses with side shields

C. Substantial footwear

D. Appropriate gloves.

4.1.3 Change gloves immediately upon chemical contact.

4.1.4 Handle all spills per ACLP-0.24, “Laboratory Spill Cleanup.”

4.2 Radiological Hazards

4.2.1 Perform all work under an applicable RWP per MCP-7, “Radiological Work Permit.”

4.2.2 Handle nonroutine samples per ACLP-0.21, “Work Control Procedure for the Analysis of Nonroutine Samples.”

4.3 Compressed Gas Hazards

4.3.1 Handle compressed gases per MCP-3635.

4.3.2 Wear safety shoes when moving compressed gas cylinders.

4.4 Physical Hazards

4.4.1 Use a holding device or a passive reshielding technique for recapping syringes.

4.4.2 Dispose of syringe needles in appropriate containers.

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4.5 Thermal Hazards

4.5.1 Avoid contact with hot surfaces on drying oven, purge and trap concentrators, and gas chromatographs.

4.6 Waste Disposition

4.6.1 Manage all waste generated per ACLP-0.40, "Analytical Laboratories Waste Management."

5. APPARATUS AND REAGENTS

5.1 Apparatus

5.1.1 Gas chromatograph, Agilent 6890N, or equivalent

5.1.2 Analytical column, DB-5, Agilent HP-5MS SV, or equivalent

5.1.3 Guard column (optional)

5.1.4 Mass spectrometer, Agilent 5973, or equivalent

5.1.5 Data system, Enviroquant, or equivalent

5.1.6 Autosampler, Agilent 7683, or equivalent

5.1.7 Helium, ultra high purity (UHP)

5.1.8 Syringes, Hamilton, or equivalent, various sizes

5.1.9 Vials, autosampler, glass, 2-mL with Teflon-lined caps

5.1.10 Inserts, glass, 300-µL with poly feet, or equivalent, for 2-mL autosampler vials

5.2 Reagents

Use all purchased standards within the manufacturer-specified shelf life, or within one year of opening the supplier's vial, whichever occurs first. Use all prepared standards within six months of preparation, unless otherwise indicated.

Store Stock Solutions at ≤–10 °C in Teflon-sealed containers in the dark. Store all other solutions at 4 ± 2 °C in Teflon-sealed containers in the dark.

5.2.1 Methylene chloride (CH_2Cl_2). Pesticide quality or better

5.2.2 Stock Standard Solutions. Purchase certified, commercially prepared solutions containing the compounds routinely analyzed (see Table 2) in benzene or methylene chloride at 1000 µg/mL from two independent sources.

NOTE: *Multiple standard solutions may be needed to encompass all routinely analyzed SVOC analytes.*

5.2.3 Standard Solution, 200 µg/mL. Prepare by diluting the primary-source Stock Standard Solutions 5-fold with methylene chloride.

5.2.3.1 Calibration Standards

Prepare standards by diluting the Standard Solution with methylene chloride per the following recommended analyte concentrations.

Cal Std Conc. (µg/mL)	µL $MeCl_2$	µL Std
10	950	50
20	900	100
30	850	150
40	800	200
50	750	250
60	700	300

5.2.3.2 Continuing Calibration Verification (CCAL) Standards

Prepare standards near the midpoint concentration of the initial calibration range by diluting the Standard Solution with methylene chloride per the following recommended analyte concentrations, and assign a 1-week shelf-life.

CCAL Std Conc. (µg/mL)	µL $MeCl_2$	µL Std
20	900	100

5.2.4 Stock Internal Standard Solution. Purchase a certified, commercially prepared solution containing 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} in methylene chloride at 1000 µg/mL.

5.2.5 Stock Tuning Solution. Purchase a certified, commercially prepared solution containing decafluorotriphenylphosphine (DFTPP), 4,4'-DDT, pentachlorophenol, and benzidine in methylene chloride at 1000 to 5000 µg/mL.

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5.2.6 Tuning Solution

5.2.6.1 5 µg/mL. Prepare by diluting the Stock Tuning Solution in methylene chloride.

6. SAMPLE HANDLING

6.1 Prepare samples for analysis per CCP-TP-187.

6.2 Store sample extracts at ≤-10 °C until completion of analysis.

6.3 Analyze all extracts within 40 days of sample extraction.

6.4 Analyze samples in analytical batches. An analytical batch consists of a suite of samples of similar matrix that is processed as a unit, using the same analytical method, within a specific time period. An analytical batch can contain up to 20 samples, excluding laboratory QC samples, all of which must be received within 14 days of validated time of sample receipt (VTSR) of the first sample in the batch.

7. PROCEDURES

NOTE: *Individual samples may be at different steps in the process. Instrument operation may occur concurrently with, or independent from, sample or standard preparation activities. The sequence of instrument and data station step-up, QC sample preparation, and data evaluation and interpretation tasks may vary.*

7.1 GC/MS Instrument Operating Parameters (Recommended)

7.1.1 Set up GC/MSs according to the recommendations in Table 1.

7.2 Tuning

7.2.1 Inject 1 µL of the Tuning Solution (Item 5.2.6).

7.2.2 Assess the GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%.

7.2.2.1 Examine the chromatogram for discernible DDT breakdown products (DDD and DDE).

NOTE: *Elution order is normally DDD, DDE and then DDT for a DB-5 column. Primary and secondary ions for DDT, DDE, and DDD are:*

Compound	*Primary Ion*	*Secondary Ions*
DDT	*235*	*237, 165*
DDE	*246*	*248, 176*
DDD	*234*	*237, 165*

7.2.2.2 IF break down products are observed,
THEN calculate the % breakdown per guidance in EPA Method 8081A, "Organochlorine Pesticides by Gas Chromatography."

7.2.3 Ensure benzidine and pentachlorophenol are present, their responses are within normal ranges, and no excessive peak tailing is present.

7.2.4 IF degradation of DDT exceeds 20%
OR poor chromatography is noted,
THEN clean the injection port and change the injector liner, and if necessary, break off the first 6–12 inches of the capillary column.

7.2.5 Acquire and evaluate the DFTPP mass spectrum.

7.2.5.1 Acquire the DFTPP mass spectrum by performing the "Auto Find" command.

NOTE: *Auto Find acquires the DFTPP mass spectrum by averaging the peak apex scan and the scans immediately proceeding and following the apex, and background subtracting from the average.*

7.2.6 Evaluate the DFTPP mass intensities against the tuning acceptance criteria in Table 3.

7.2.6.1 IF the DFTPP fails to meet these criteria,
THEN evaluate the results and adjust the MS or clean the source, if necessary.

7.2.6.2 Re-inject the Tuning Solution and re-evaluate the DFTPP peak.

7.2.6.3 Repeat these steps until the DFTPP passes the tuning criteria.

NOTE: *A 12-hour time clock (tune period) starts at the injection time of the Tuning Solution that passes the tuning criteria.*

7.2.7 Continue with the analysis sequence after obtaining a passing DFTPP tune.

7.2.7.1 Perform subsequent injections in the analysis sequence within the 12-hour time frame from the DFTPP tune.

7.2.7.2 Perform all subsequent injections under the same MS instrument conditions as those associated with the DFTPP.

7.2.7.3 IF initial calibration is required,
THEN GO TO Section 7.3, Initial Calibration.

7.2.7.4 IF the initial calibration has already been performed,
THEN GO TO Section 7.4, Continuing Calibration.

7.3 Initial Calibration

NOTE: *Section 7.3 is performed initially, then as needed, based on instrument maintenance or QC results.*

7.3.1 Do not change the MS instrument conditions from those used to pass the DFTPP tuning criteria.

7.3.2 Use the identical GC/MS instrument conditions for all standards, samples, blanks and quality control samples associated with the DFTPP analysis.

NOTE: *A minimum of one calibration standard having concentrations below the PRQL is required. The PRQL values of 40 mg/kg and 2.6 mg/kg from Table 4 correspond to solution-equivalent concentrations of 200 µg/mL and 13 µg/mL, respectively, assuming a 5 g sample weight and a 1 mL final extract volume.*

7.3.3 Add internal standard (Item 5.2.4) to each calibration standard to provide a spiking ratio of 5 µL Internal Standard Solution to 200 µL calibration standard.

7.3.4 Inject 0.5–2 µL of each calibration standard. Injection volume may vary between the GC/MS systems.

7.3.5 Ensure that the calibration file correctly identifies the peaks, based on the following criteria:

A. The retention time (RT) window is ≤30 seconds. A 30-second window ensures that the SW-846 requirement of ±0.06 relative retention time (RRT) units is met. A RT window <30 seconds may be used to ensure that the proper peak is identified. This is especially important for structural isomers having very similar mass spectra and retention times.

B. The search method is based on the mass spectrum.

C. The relative intensities of the characteristic ions have a 30% window. The characteristic ions are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum.

D. The quantitation mass is the primary ion (see Table 2).

7.3.5.1 IF a peak is incorrectly identified,
THEN adjust the RT window as necessary to correct the identification, based on visual evaluation of the chromatogram.

7.3.6 Integrate the extracted ion current profile (EICP) using the calibration file.

7.3.7 Verify that all analytes are correctly integrated.

7.3.7.1 Manually integrate any peaks that are incorrectly integrated.

7.3.7.1.1 Print a copy of the manual integrations and document the justification for the manual integration (e.g., wrong retention time, integrated to baseline).

7.3.8 Check for interferences with the primary quantitation ions.

7.3.8.1 IF interferences are found,
THEN perform the following steps.

7.3.8.1.1 Print a hard copy of the mass spectrum showing the interferences.

7.3.8.1.2 Change the quantitation mass in the calibration file for that analyte to a secondary ion (see Table 2).

7.3.8.1.3 Re-integrate any previously integrated peaks.

7.3.9 Verify that the target analytes and surrogates reference the nearest internal standard, using the retention times from the mid-point standard; modify the calibration file if necessary.

NOTE: *Calculations are performed by the instrument data system.*

7.3.10 Calculate the relative response factors (RRFs) for each analyte (see Equation 9.1).

7.3.11 Calculate the average RRF and the percent relative standard deviation (%RSD) of the RRFs for each analyte (see Equations 9.2, 9.3, and 9.4).

7.3.12 Verify that all Calibration Check Compounds (CCCs) have a %RSD ≤30%. The CCCs are:

Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine (N-Nitrosodiphenylamine[a])	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

a. N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.

7.3.12.1 <u>IF</u> %RSD is >30% for any CCC,
<u>THEN</u> examine the graphical plot of the response versus the concentration.

7.3.12.1.1 <u>IF</u> a single standard appears to be the source of the nonlinearity,
<u>THEN</u> reprepare, reanalyze and reevaluate that standard.

7.3.12.1.2 <u>IF</u> the curve is <u>NOT</u> linear at the upper or lower end,
<u>THEN</u> delete the highest standard as appropriate and recalculate the average RRF and %RSD of the RRFs.

NOTE: *A minimum of five data points (standards) is required. A minimum of six data points (standards) is required for quadratic regressions.*

7.3.12.1.3 IF the %RSD is still >30%, THEN evaluate the GC/MS system for possible problems, including standard degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or detector system.

NOTE: *Changing the injection port septum or liner does not constitute a major change to the GC/MS system.*

7.3.12.1.3.1 Correct the problem(s). Refer to ACLP-9.01, "Equipment Maintenance for the Organic Laboratory."

7.3.12.1.3.2 GO TO Section 7.2, Tuning, OR Section 7.3, Initial Calibration, as appropriate for the action taken.

7.3.13 Verify that average RRFs meet acceptance criteria.

7.3.13.1 Verify that all System Performance Check Compounds (SPCCs) have average RRFs ≥0.050. The SPCCs are:

N-nitroso-di-n-propylamine

Hexachlorocyclopentadiene

2,4-dinitrophenol

4-nitrophenol.

7.3.13.2 Verify that all other (non-SPCC) compounds have average RRFs ≥0.01.

7.3.13.3 IF the average RRFs do NOT meet the acceptance criteria, THEN evaluate the GC/MS system for possible problems, including standard degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or detector system.

7.3.13.3.1 Correct the problem(s). Refer to ACLP-9.01.

7.3.13.3.2 GO TO Section 7.2 or Section 7.3, as appropriate for the action taken.

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7.3.14 Evaluate the %RSD of each calibrated compound for linearity.

7.3.14.1 IF the %RSD is ≤15%,
THEN use the average RRF for quantitation.

7.3.14.2 IF the %RSD is >15%,
THEN use a linear or quadratic regression (instrument response versus standard concentration) for quantitation.

NOTE: *Linear regressions require a minimum of five standards. Quadratic regressions require a minimum of six standards.*

7.3.14.2.1 Modify the calculation type (average, linear or quadratic) in the calibration file, as necessary.

7.3.14.2.2 Verify that the linear regression correlation coefficient (r) or the quadratic regression coefficient of determination (COD) is ≥0.990.

7.3.14.2.3 IF the r or COD fails to meet the criteria,
THEN remove the upper standard to narrow the calibration range and re-evaluate the calibration linearity using average RRF or linear regression.

7.3.14.2.4 IF the r still fails to meet the criteria,
THEN consult with the responsible technical lead.

7.3.15 IF analyzing samples within the same 12-hour shift as the ICAL,
THEN GO TO Section 7.5 for sample analysis.

7.4 Continuing Calibration

7.4.1 Do not change the MS instrument conditions from those used to pass the DFTPP tuning criteria.

7.4.2 Use GC instrument conditions identical to those of the ICAL for all standards, samples, blanks and quality control samples.

7.4.3 Verify that the GC/MS has met the DFTPP tuning criteria (Section 7.2).

7.4.4 Verify that the GC/MS has met the initial calibration criteria (Section 7.3).

7.4.5 Add internal standard (Item 5.2.4) to the CCAL standard to provide an internal standard spiking ratio consistent with that used for the ICAL (see Step 7.3.3).

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7.4.6 Analyze the continuing calibration standard, using the same injection volume as was used for the ICAL.

7.4.6.1 Set up the data station to accept data for the specified solution.

7.4.6.2 Start the GC/MS and data station to acquire analysis data.

7.4.7 Integrate the EICP, using the calibration file generated from the most recent initial calibration.

7.4.8 Verify that all analytes are correctly integrated.

7.4.9 Manually integrate any peaks that are incorrectly integrated.

7.4.9.1 Print a copy of the manual integrations and document the justification for the manual integration (e.g., wrong retention time, integrated to baseline).

7.4.10 Calculate the percent difference (%D) between the ICAL and the CCAL for each analyte.

7.4.10.1 IF average RRFs are used for quantitation, THEN calculate %D between the ICAL average RRF and the CCAL RRF.

7.4.10.2 IF regression fit models are used for quantitation, THEN calculate %D between the known and measured concentrations of the CCAL standard.

7.4.11 Verify that all CCCs have %D ≤20.

7.4.11.1 IF the %D is >20 for any CCC, THEN repeat Steps 7.4.5 through 7.4.11.

7.4.11.2 IF the %D is still >20 for any CCC, THEN GO TO Section 7.3, or prepare a fresh Standard Solution (Item 5.2.3) and either GO TO Section 7.3 or Section 7.4, Continuing Calibration.

7.4.12 Verify that CCAL RRFs meet acceptance criteria.

7.4.12.1 Verify that all SPCCs have a RRF ≥0.050.

7.4.12.2 Verify that all other compounds have a RRF ≥0.01.

7.4.12.3 IF any of the RRFs do NOT meet the acceptance criteria, THEN evaluate the GC/MS system for possible problems, including standard degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or detector system.

7.4.12.3.1 Correct the problem(s). Refer to ACLP-9.01.

7.4.12.3.2 GO TO Section 7.4 after corrective maintenance is completed.

7.4.12.3.3 IF the CCAL still fails acceptance criteria after corrective maintenance, THEN GO TO Section 7.3.

7.4.13 Check the internal standard retention times (RTs) against those of previously analyzed standards.

7.4.13.1 Verify that the CCAL RTs are within ±30 seconds of those in the last daily calibration standard.

7.4.13.2 IF the RTs have changed more than 30 seconds, THEN evaluate the system for potential problems and perform corrective maintenance as necessary.

7.4.13.2.1 Reanalyze the CCAL per Section 7.4.

7.4.13.2.2 IF the CCAL still fails, THEN GO TO Section 7.3.

7.4.14 Check the internal standard peak areas against those of previously analyzed standards.

7.4.14.1 Verify that the internal standard peak areas are within a factor of two (50 to 200%) of those in the last daily calibration standard.

7.4.14.2 IF the peak areas of the internal standards have changed by more than a factor of two (50 to 200%), THEN evaluate the system for potential problems, and perform corrective maintenance as necessary.

7.4.14.2.1 Reanalyze the CCAL per Section 7.4.

7.4.14.3 IF the CCAL still fails, THEN GO TO Section 7.3.

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7.5 Sample Analysis

7.5.1 Do not change the MS instrument conditions from those used to pass the DFTPP tuning criteria.

7.5.2 Use GC instrument conditions identical to those of the ICAL for all standards, samples, blanks and quality control samples.

7.5.3 Verify that the GC/MS has met the DFTPP tuning criteria (Section 7.2).

7.5.4 Verify that the GC/MS has met the initial calibration criteria (Section 7.3).

7.5.5 Verify that the GC/MS has met the continuing calibration criteria (Section 7.4) unless the samples are being analyzed within the same 12-hour shift as a valid ICAL.

7.5.6 Check extracts for coloration indicating high levels of organic contaminants.

7.5.6.1 IF an extract exhibits coloration,
THEN dilute it with methylene chloride for the initial analytical run when necessary to avoid instrument contamination.

7.5.7 Add internal standards to the extracts, using the same internal standard spiking ratio as was used for ICAL and CCAL standards (see Steps 7.3.3 and 7.4.5).

7.5.8 Analyze the LB, LCS, sample extracts, MS and MSD, using the same injection volume as was used for the ICAL.

NOTE: *Analysis of the LB and LCS extracts before analysis of the associated sample extracts is recommended.*

7.5.8.1 Set up the data station to accept data for the specified solution.

7.5.8.2 Start the GC/MS and data station to acquire analysis data.

7.5.9 Integrate the EICP, using the calibration file generated from the most recent initial calibration.

7.5.10 Verify that the peaks are correctly integrated.

7.5.10.1 Manually integrate any peaks that are incorrectly integrated.

7.5.10.1.1 Print a copy of the manual integrations and document the justification for the manual integration (e.g., wrong retention time, integrated to baseline).

7.5.11 Verify that concentrations of target compounds are within the ICAL calibration range.

7.5.11.1 IF compounds are quantitated using quadratic regressions, THEN use the peak area, not the concentration, to determine if the compound is within the ICAL range.

7.5.11.2 Examine the peak shape of all target analyte peaks for possible detector saturation.

7.5.11.2.1 IF any saturated peaks are found, THEN dilute the sample extract with methylene chloride and reanalyze.

7.5.11.3 IF concentration (or area for quadratic models) exceeds the calibration range,
THEN dilute the sample extract in methylene chloride such that the highest concentration target analyte is within the upper half of the calibration range and reanalyze.

7.5.11.3.1 Examine results of subsequent injections for potential carry-over (cross contamination) due to the over-range compound(s).

7.5.11.3.2 Reanalyze any samples having potential carry-over.

7.5.11.4 IF the initial analysis of an extract was performed diluted due to suspected high concentration levels,
AND results of the initial analysis indicate that the extract can be safely analyzed at a lesser dilution,
THEN reanalyze the sample at a lesser dilution such that the highest concentration target analyte is within the upper half of the calibration range or such that hydrocarbon contamination is no greater than twice the highest internal standard peak height.

NOTE: *Highly contaminated sample extracts will not be analyzed neat or at dilutions that would result in damage or gross contamination of the analytical system.*

7.5.12 Perform a mass spectral library search to tentatively identify sample components not associated with the calibration standards.

7.5.12.1 Use a spectral reference library that contains, at a minimum, all available spectra for compounds that appear in the 20.4.1.200 NMAC (incorporating 40 CFR Part 261) Appendix VIII list.

7.5.12.2 Perform a mass spectral library search on all non-target analyte sample components present with a minimum of 10% of the area of the nearest internal standard.

7.6 Data Evaluation

7.6.1 Verify that the injection time for sample extracts is within 12 hours of a passing DFTPP injection (see Section 7.2).

7.6.2 Verify that comparison of the sample internal standard RTs and areas to those of the CCAL meet the QC limits defined in Step 8.2.1.

7.6.2.1 IF samples are analyzed immediately after an ICAL (i.e., within the same 12-hour tune period), THEN use the mid-point standard of the ICAL as the CCAL.

7.6.3 Identify a target compound as present when the following criteria are met.

A. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criteria.

B. The relative retention time (RRT) of the sample component is within ±0.06 RRT units of the RRT of the standard component.

C. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

7.6.4 Identify a tentatively identified compound (TIC) as present based on the mass spectral library search per the following SW-846 guidance.

A. Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

B. The relative intensities of the major ions should agree within ±20% (e.g., for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30% and 70%).

C. Molecular ions present in the reference spectrum should be present in the sample spectrum.

D. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

E. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks.

7.6.5 Evaluate the LB and LCS results against the acceptance criteria defined in Steps 8.3.1 and 8.3.2, respectively, and implement the defined corrective actions in case of failures.

7.6.6 Evaluate surrogate recoveries against the acceptance criteria defined in Step 8.2.2 and implement the defined corrective actions in case of failures.

7.6.7 Calculate sample and MS/MSD results in reporting units, using the equations in Section 9.11.

7.6.8 Evaluate the MS/MSD results against the acceptance criteria defined in Step 8.3.3.

7.6.9 Assign data qualifying flags based on the data evaluation per CCP-TP-188, *CCP Analytical Data Recording, Review, and Reporting*."

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8. QUALITY CONTROL REQUIREMENTS

Table 5 summarizes QC requirements discussed in this section and identifies corrective actions.

8.1 Instrument-Specific QC Requirements

8.1.1 Tuning

Daily before analysis of standards or samples, ensure the hardware tune of the GC/MS system meets the criteria listed in Table 3 (see Section 7.2). An acceptable DFTPP tune starts a 12-hour tune period within which standards and samples may be injected. Do not proceed with sample analysis until a tune meeting these criteria is achieved.

8.1.2 Initial Calibration (ICAL)

8.1.2.1 Calibrate the GC/MS initially and then as needed for all target analytes and surrogate compounds.

8.1.2.2 Use a minimum of five standards for average RRF or linear regression calibration, and a minimum of six standards for quadratic regression calculation.

8.1.2.3 Verify that CCC %RSDs are ≤30%.

8.1.2.4 Verify that SPCC average RRFs are ≥0.050.

8.1.2.5 Verify that the average RRF of all other compounds is ≥0.01.

8.1.2.6 Use the average RRF to calculate concentration if the %RSD is ≤15%.

8.1.2.7 Use a linear or quadratic regression to calculate concentration if the %RSD is >15%.

8.1.2.8 For linear and quadratic regressions, verify that correlation coefficients or coefficients of determination are ≥0.990.

8.1.2.9 Perform a new initial calibration (ICAL) whenever the GC/MS system has undergone major changes (e.g., cleaning the MS, changing the GC column, etc.) or whenever indicated as corrective action for continuing calibration failure.

NOTE: *Changing the injection port septum or liner does not constitute a major change to the GC/MS system.*

8.1.3 Continuing Calibration (CCAL) Verification

8.1.3.1 Daily within the 12-hour tune period but before sample injections, verify the calibration of the GC/MS for all target analytes and surrogate compound using a midrange standard.

8.1.3.2 Verify that the CCC %Ds are ≤20%.

8.1.3.3 Verify that the SPCC RRFs are ≥0.050.

8.1.3.4 Verify that RRFs of all other compounds are ≥0.01.

8.1.3.5 Verify that the internal standards RTs are within 30 seconds of the RTs of the last daily calibration.

8.1.3.6 Verify that the internal standards areas are within 50 to 200% of the areas of the last daily calibration.

8.2 Sample-Specific QC Requirements

8.2.1 Internal Standard Areas and Retention Times

8.2.1.1 Verify the internal standard areas for all samples are within 50 to 200% of the associated CCAL internal standard areas (use the mid-point ICAL standard if samples are analyzed within the same 12-hour shift as an ICAL).

8.2.1.2 Verify internal standard RTs for all samples are within 30 seconds of the associated CCAL internal standard RTs (use the mid-point ICAL standard if samples are analyzed within the same 12-hour shift as an ICAL).

8.2.1.3 Reanalyze or dilute and reanalyze samples failing to meet these criteria, depending upon the suspected cause of the internal standard failure.

8.2.2 Surrogate Compounds

8.2.2.1 Calculate in-house control limits for surrogates using the surrogate recoveries from a minimum of 30 samples, using the average %R plus or minus three standard deviations.

8.2.2.1.1 Use the following default control limits until in-house limits are calculated:

2-fluorophenol:	Detected–84%
phenol-d_6:	10–94%
nitrobenzene-d_5:	23–116%
2-fluorobiphenyl:	22–111%
2,4,6-tribromophenol:	Detected–91%
terphenyl-d_{14}:	18–137%.

8.2.2.2 If the surrogate recoveries do not meet the control limits, check instrument performance, correct any identified problem and reanalyze the sample extract.

8.2.2.2.1 If no instrument performance problem is identified, reextract and reanalyze the sample if there is sufficient sample volume and the reextraction can be performed within the preparation holding time.

8.2.2.2.2 If no instrument performance problem is identified and sample preparation holding time has expired or there is insufficient sample volume for re-extraction, qualify the data (see Table 5) and discuss in the report narrative.

8.2.2.2.3 See instructions in Step 8.3.1 and 8.3.2 for LB and LCS surrogate recovery evaluation.

8.3 Analytical Batch QC Requirements

Batch QC samples consist of a laboratory blank (LB), a laboratory control sample (LCS), a matrix spike (MS) and a matrix spike duplicate (MSD). These QC samples are prepared for analysis per CCP-TP-187.

8.3.1 Laboratory Blank (LB)

8.3.1.1 Analyze a minimum of one unique LB with every analytical batch.

8.3.1.2 Compare LB target analyte results to the acceptance criteria in Table 5.

8.3.1.3 If the LB results do not meet the acceptance criteria, take corrective action to locate and reduce the source of contamination and re-extract and reanalyze the samples associated with the contaminated laboratory blank, if the preparation holding time has not expired and there is sufficient sample volume for re-extraction.

8.3.1.4 If the LB results do not meet the acceptance criteria and samples cannot be re-extracted, qualify the associated sample data (see Table 5) and discuss in the report narrative.

8.3.1.5 Compare LB surrogate recoveries to the limits defined in Step 8.2.2.

8.3.1.5.1 If LB surrogate recoveries do not meet the acceptance criteria, reextract all associated samples if the re-extraction can be performed within preparation holding time and if there is sufficient sample volume.

8.3.1.5.2 If LB surrogate recoveries do not meet the acceptance criteria and reextraction of associated samples is not possible, qualify the associated sample data (see Table 5) and discuss in the report narrative.

8.3.2 Laboratory Control Sample

8.3.2.1 Analyze a minimum of one unique LCS with every analytical batch.

8.3.2.2 Compare the LCS %Rs to the acceptance criteria in Table 5.

8.3.2.3 If the %Rs do not meet the QC limits, check instrument performance, correct any identified problem and reanalyze the sample extract.

8.3.2.3.1 If no instrument performance problem is identified, re-extract and reanalyze the associated samples if there is sufficient sample volume and the re-extraction can be performed within the preparation holding time.

8.3.2.3.2 If no instrument performance problem is identified and associated sample preparation holding time has expired or there is insufficient sample volume for re-extraction, qualify associated sample data (see Table 5) and discuss in the report narrative.

8.3.2.4 Compare LCS surrogate recoveries to the limits defined in Step 8.2.2.

8.3.2.4.1 If LCS surrogate recoveries do not meet the acceptance criteria, re-extract all associated samples if the re-extraction can be performed within preparation holding time and if there is sufficient sample volume.

8.3.2.4.2 If LCS surrogate recoveries do not meet the acceptance criteria and re-extraction of associated samples is not possible, qualify the associated sample data (see Table 5) and discuss in the report narrative.

8.3.3 Matrix Spike/Matrix Spike Duplicate

8.3.3.1 Analyze a minimum of one MS and one MSD with every analytical batch.

8.3.3.2 Compare the %Rs and RPDs to the accuracy and precision acceptance limits listed in Table 5.

8.3.3.3 If the %R or RPD results do not meet the acceptance criteria, evaluate the data for potential laboratory performance issues or matrix-related problems. Compare MS/MSD recoveries with LCS %Rs and surrogate %Rs to aid this evaluation. Evaluate differences in target analyte or surrogate recoveries between the MS and MSD. If a sample preparation problem is indicated, re-extract all samples in the batch if there is sufficient sample volume and the preparation holding times have not expired. If no sample preparation problem is indicated, or if re-extraction is not possible, qualify the associated sample data (see Table 5) and discuss in the report narrative.

8.4 Method Performance Requirements

8.4.1 Minimum Detection Limits (MDLs)

8.4.1.1 Determine MDLs for all target analytes and surrogates on each analytical instrument used for this analysis.

8.4.1.2 Determine MDLs initially before sample analysis.

8.4.1.3 Redetermine MDLs semiannually.

8.4.1.4 Redetermine MDLs if significant changes are made to the method or instrumentation.

8.4.1.5 Use a minimum of seven replicate standards having concentrations approximately three to ten times the anticipated MDLs.

NOTE: *Preparation instructions for MDL standards are provided in CCP-TP-187.*

8.4.1.6 Calculate the MDLs using Equation 9.14.

8.4.1.7 Compare the MDLs to the program-required MDLs in Table 4. If calculated MDLs are greater than the program-required MDLs, repeat the MDL study until acceptable MDLs are achieved.

8.4.2 Method Precision and Accuracy (P&A) Demonstration

8.4.2.1 Demonstrate method P&A for all target analytes & surrogates.

8.4.2.2 Demonstrate method P&A initially before sample analysis.

8.4.2.3 Redemonstrate method P&A semiannually.

8.4.2.4 Redemonstrate method P&A if significant changes are made to the method or instrumentation.

8.4.2.5 Use a minimum of seven P&A standards for initial method P&A demonstration and a minimum of four P&A standards for continuing P&A demonstrations.

NOTE: *Preparation instructions for P&A standards are provided in CCP-TP-187.*

8.4.2.6 Calculate the mean %R of each analyte and the %RSD of the recoveries of each analyte.

8.4.2.7 Compare the mean %R and %RSD to the accuracy (%R) and precision (%RSD) criteria listed in Table 4.

8.4.2.8 If one or more of the analytes fail the P&A acceptance criteria, locate and correct the source of the problem, and repeat the demonstration until acceptable P&A is achieved.

9. CALCULATIONS

NOTE: *Some or all of the calculations in this section may be performed by instrument data systems or data reporting software.*

9.1 Relative Response Factor (RRF)

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

where

A_s	=	peak area of the analyte or surrogate
A_{is}	=	peak area of the nearest internal standard
C_s	=	concentration (or amount) of the analyte or surrogate
C_{is}	=	concentration (or amount) of the nearest internal standard.

9.2 Arithmetic Mean

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$

where

$\bar{x}$	=	arithmetic mean
x_i	=	i^{th} data point
n	=	total number of data points.

9.3 Standard Deviation (s)

$$s = \sqrt{\frac{\sum_{i=1}^{n}\left(x_i - \bar{x}\right)^2}{n-1}}$$

where

s	=	standard deviation
$\bar{x}$	=	arithmetic mean
x_i	=	i^{th} data point
n	=	total number of data points.

9.4 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{s}{\bar{x}} \times 100$$

where

s	=	standard deviation
$\bar{x}$	=	arithmetic mean.

9.5 Least Squares Linear Regression

$$y = mx + b$$

where

y	=	instrument response
m	=	slope of the line
x	=	concentration of the calibration standard
b	=	intercept.

Using internal standard quantitation, the linear regression equation is:

$$\frac{A_s C_{is}}{A_{is}} = mC_s + b \text{ or } C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - \mathrm{b}\right]}{\mathrm{m}}$$

where

A_s	=	area of the peak for the target analyte in the sample
A_{is}	=	area of the peak for the internal standard
C_s	=	concentration of the target analyte in the sample
C_{is}	=	concentration of the internal standard
m	=	slope of the line
b	=	intercept.

9.6 Correlation Coefficient (r)

$$r = \frac{\left[n\sum_{i=1}^{n} x_i y_i\right] - \sum_{i=1}^{n} x_i \sum_{i=1}^{n} y_i}{\sqrt{n\sum_{i=1}^{n} x_i^{\,2} - \left(\sum_{i=1}^{n} x_i\right)^2}\sqrt{n\sum_{i=1}^{n} y_i^{\,2} - \left(\sum_{i=1}^{n} y_i\right)^2}}$$

where

x	=	C_s (concentration of target analyte in standard)
y	=	instrument response.

9.7 Quadratic Regression

$$y = ax^2 + bx + c$$

where

y	=	instrument response
x	=	concentration of the calibration standard
a	=	regression factor relating instrument response and concentration
b	=	regression factor relating instrument response and concentration
c	=	intercept.

Using internal standard quantitation, the quadratic regression equation is:

$$\frac{A_s C_{is}}{A_{is}} = a(C_s)^2 + b(C_s) + c$$

where

A_s	=	area of the peak for the target analyte in the sample
A_{is}	=	area of the peak for the internal standard
C_s	=	concentration of the target analyte in the sample
C_{is}	=	concentration of the internal standard
a	=	regression factor relating instrument response and concentration
b	=	regression factor relating instrument response and concentration
c	=	intercept.

9.8 Coefficient of Determination (COD)

$$COD = \frac{\sum_{i=1}^{n}\left(y_{obs} - \bar{y}\right)^2 - \left(\frac{n-1}{n-p}\right)\sum_{i=1}^{n}\left(y_{obs} - Y_i\right)^2}{\sum_{i=1}^{n}\left(y_{obs} - \bar{y}\right)^2}$$

where

y_{obs} = observed area for each concentration from each initial calibration standard

$\bar{y}$ = mean observed response from the initial calibration

Y_I = calculated (or predicted) area at each concentration from the initial calibration

n = total number of calibration points

p = number of adjustable parameters in the polynomial equation (i.e., 2 for a second-order or quadratic regression).

9.9 Percent Difference (%D)

$$\%D = \frac{|I - C|}{I} \times 100$$

where

I = average RRF from the initial calibration or the known concentration of the CCAL standard (for modeled compounds)

C = RRF from the continuing calibration or the measured concentration of the CCAL standard (for modeled compounds).

9.10 Extract Concentration

9.10.1 Average RRF Quantitation

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

where

C_s = concentration of analyte in extract (µg/mL)

A_s = area of sample (EICP for target analytes, total ion area counts for TICs)

A_{is} = area of nearest internal standard (EICP for target analytes, total ion area counts for TICs)

C_{is} = concentration of internal standard in extract (µg/mL)

$\overline{RRF}$ = average relative response factor (assume $\overline{RRF}$ = 1 for TICs).

9.10.2 Linear Regression Quantitation

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - \mathrm{b}\right]}{\mathrm{m}}$$

where

C_s = concentration of the target analyte in the extract (µg/mL)

A_s = area of the peak for the target analyte in the sample

A_{is} = area of the peak for the internal standard

C_{is} = concentration of the internal standard in extract (µg/mL)

m = slope of the line

b = intercept.

9.10.3 Quadratic Regression Quantitation

$$\frac{A_s C_{is}}{A_{is}} = a(C_s)^2 + b(C_s) + c$$

where

As	=	area of the peak for the target analyte in the sample
Ais	=	area of the peak for the internal standard
Cs	=	concentration of the target analyte in the extract (µg/mL)
Cis	=	concentration of the internal standard in µg/mL
a	=	regression factors relating instrument response and concentration
b	=	regression factors relating instrument response and concentration
c	=	intercept.

9.11 Sample Concentration

$$Concentration\ (mg/kg) = \frac{(C_s)\ (V_t)\ (DF)}{(W_s)}$$

where

Cs	=	concentration of sample extract (µg/mL) (Equation 9.10)
Vt	=	volume of total extract (mL)
DF	=	dilution factor of extract
Ws	=	weight of sample aliquot (g).

9.12 Percent Recovery

9.12.1 LCSs, Surrogates, P&A Standards

$$\%R = \frac{C_m}{C_t} \times 100$$

where

C_m = measured (calculated) concentration

C_t = true (known) concentration.

9.12.2 MS and MSD

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

where

SSR = measured concentration of the spiked sample

SR = measured concentration of the unspiked sample

SA = concentration of the spike added.

9.13 Relative Percent Difference

$$RPD = \frac{|C_1 - C_2|}{0.5(C_1 + C_2)} \times 100$$

where

C_1 = original (first) measured sample result

C_2 = duplicate (second) measured sample result.

9.14 Minimum Detection Limit

$$MDL = t_{(n-1,\,1-\alpha=0.99)} \times s$$

where

s = standard deviation of the data set

$t_{(n-1,\,1-\alpha=0.99)}$ = students-t statistic for a 99% confidence level (one tailed) and standard deviation estimate with n-1 degrees of freedom (n is the number of measurements).

Values of *t* for a 99% confidence level are listed below.

n-1	*t*
6	3.143
7	2.998
8	2.896
9	2.821

9.15 Q-Test

The Q-test evaluates small data sets (n<10) for single outliers.

$$Q = \frac{X_2 - X_1}{X_n - X_1} \quad \textit{for the lowest value } (X_1)$$

$$Q = \frac{X_n - X_{n-1}}{X_n - X_1} \quad \textit{for the highest value } (X_n)$$

Reject the questionable value if the calculated Q is greater than the established rejection Q at the 90% confidence level listed below.

Number of values (n)	Rejection Quotient ($Q_{0.90}$)
3	0.94
4	0.76
5	0.64
6	0.56
7	0.51
8	0.47
9	0.44
10	0.41

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9.16 Grubbs Test

The Grubbs-Test evaluates any normally-distributed data set with more than three observations for single outliers.

$$T = \frac{\overline{X} - X_1}{s} \quad if\ X_1\ is\ suspect \text{ or } T = \frac{X_n - \overline{X}}{s} \quad if\ X_n\ is\ suspect$$

Where

$\overline{X}$ = average of the *n* results

s = standard deviation of the results.

Reject the suspect datum if the calculated T-value is greater than the established rejection criterion at α=0.10 (a 10% chance of falsely concluding that the datum is an outlier).

Number of Values (n)	Rejection Criteria
4	1.425
5	1.602
6	1.729
7	1.828
8	1.909
9	1.977
10	2.036

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10. RECORDS

Records generated during the performance of this procedure are maintained as QA records in accordance with CCP-QP-008, *CCP Records Management*. The records are the following:

QA/Nonpermanent

- Initial Calibration (ICAL) Files
- Method Detection Limit (MDL) Files
- Precision and Accuracy (P&A) Demonstration Files

The following records generated during the performance of this procedure will be compiled into the Supporting Data Package, in accordance with CCP-TP-188, *CCP Analytical Data Recording, Review and Reporting.*

QA/Nonpermanent

- Copy of applicable pages of Instrument Run Logs
- Instrument Printouts/Raw Data

11. REFERENCES

11.1 ACLP-0.21, "Work Control Procedures for Analysis of Nonroutine Samples"

11.2 ACLP-0.24, "Laboratory Spill Cleanup"

11.3 ACLP-0.40, "Analytical Laboratories Waste Management"

11.4 ACLP-9.01, "Equipment Maintenance for the Organic Laboratory"

11.5 CCP-QP-002, *CCP Training and Qualification Plan*

11.6 CCP-QP-005, *CCP TRU Nonconforming Item Reporting and Control*

11.7 CCP-QP-008, *CCP Records Management*

11.8 CCP-TP-187, *CCP Sample Preparation for Semivolatile Organic Compounds*

11.9 CCP-TP-188, *CCP Analytical Data Recording, Review and Reporting*

11.10 EPA SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," Third Edition, Method 8000B, "Determinative Chromatographic Separations," U.S. Environmental Protection Agency, 1986

11.11 EPA SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," Third Edition, Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry," U.S. Environmental Protection Agency, 1986

11.12 EPA SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," Third Edition, Method 8081A, "Organochlorine Pesticides by Gas Chromatography," U.S. Environmental Protection Agency, 1986

11.13 MCP-7, "Radiological Work Permit"

11.14 MCP-3635, "Chemical Hygiene Plan"

11.15 Hazardous Waste Facility Permit, Waste Isolation Pilot Plant EPA # NM4890139088 (WIPP WAP)

Appendix A

Tables

Table 1. GC/MS Recommended Operating Conditions.

GC PARAMETERS							
Oven Settings							
Start (°C)		End (°C)		Rate (°C/min)		Hold (min)	
40	[125]*	40	[125]	0.0		3.0	[2.0]
40	[125]	280	[280]	13	[15]	3.0	[2.0]
280		320		25		6.0	
*[DFTPP conditions]							

Injector Settings
Temp (°C)
250

Electronic Pressure Control Settings
To maintain constant flow of 1.5 mL/min.

Pulsed Splitless
Injection Pulse Pressure @ 50 psi until 1.5 min.

Injection Volume
0.5 µL

MS PARAMETERS

Solvent Delay	3.0 min
Acquisition Mode	Scan
Low Mass	35
High Mass	550
Scans/sec	2.86
Source Temp	230 °C
Quad Temp	150 °C

Table 2. Calibrated Analytes and Quantitation Ions

Analyte	CAS #	WAP Analyte*	Primary Ion	Secondary Ions
Acenaphthene (CCC)	83-32-9		154	153, 152
Acenaphthene-d_{10} (IS)	15067-26-2		164	162, 160
Benzo(a)pyrene (CCC)	50-32-8		252	253, 125
Bis(2-ethylhexyl)phthalate	117-81-7	AMWTP ARP	149	167, 279
4-Chloro-3-methylphenol (CCC)	59-50-7		107	144, 142
Chrysene-d_{12} (IS)	1719-03-5		240	120, 236
1,2-Dichlorobenzene (ortho-Dichlorobenzene)	95-50-1	X	146	148, 111
1,4-Dichlorobenzene (CCC)	106-46-7	X	146	148, 111
1,4-Dichlorobenzene-d_4 (IS)	3855-82-1		152	150, 115
2,4-Dichlorophenol (CCC)	120-83-2		162	164, 98
2,4-Dinitrophenol (SPCC)	51-28-5	X	184	63, 154
2,4-Dinitrotoluene	121-14-2	X	165	63, 89
Di-n-octyl phthalate (CCC)	117-84-0		149	167, 43
Fluoranthene (CCC)	206-44-0	AMWTP	202	101, 203
2-Fluorobiphenyl (surr)	321-60-8		172	171
2-Fluorophenol (surr)	367-12-4		112	64
Hexachlorobenzene	118-74-1	X	284	142, 249
Hexachlorobutadiene (CCC)	87-68-3	ARP	225	223, 227
Hexachlorocyclopentadiene (SPCC)	77-47-4		237	235, 272
Hexachloroethane	67-72-1	X	117	201, 199
2-Methylphenol (o-cresol)	95-48-7	X	107	108, 77, 79, 90
3-Methylphenol (m-cresol)	108-39-4	X	107	108, 77, 79, 90
4-Methylphenol (p-cresol)	106-44-5	X	107	108, 77, 79, 90
Naphthalene-d_8 (IS)	1146-65-2		136	68
Nitrobenzene	98-95-3	X	77	123, 65
Nitrobenzene-d_5 (surr)	4165-60-0		82	128, 54

Analyte	CAS #	WAP Analyte*	Primary Ion	Secondary Ions
2-Nitrophenol (CCC)	88-75-5		139	109, 65
4-Nitrophenol (SPCC)	100-02-7		139	109, 65
N-nitrosodiphenylamine (CCC)	86-30-6		169	168, 167
N-Nitrosodi-n-propylamine (SPCC)	621-64-7		70	42, 101, 130
Pentachlorophenol (CCC)	87-86-5	X	266	264, 268
Perylene-d_{12} (IS)	1520-96-3		264	260, 265
Phenanthrene-d_{10} (IS)	1517-22-2		188	94, 80
Phenol (CCC)	108-95-2	AMWTP	94	65, 66
Phenol-d_6 (surr)	13127-88-3		99	42, 71
Terphenyl-d_{14} (surr)	1718-51-0		244	122, 212
2,4,6-Tribromophenol (surr)	118-79-6		330	332, 141
2,4,6-Trichlorophenol (CCC)	88-06-2		196	198, 200

*. In the WAP Analyte column, “X” indicates a required target analyte for all samples. “AMWTP” indicates a site-specific target analyte for the Advanced Mixed Waste Treatment Project. “ARP” indicates a site-specific target analyte for the Accelerated Retrieval Project.

Table 3. DFTPP Tuning Criteria.

Mass	Ion Abundance Criteria
51	30–60% of mass 198
68	<2% of mass 69
69	Mass 69 relative abundance
70	<2% of mass 69
127	40–60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5–9% of mass 198
275	10–30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17–23% of mass 442

Table 4. Data Quality Assurance Objectives (QAOs).

Compound	Precision[a] (%RSD or RPD)	Accuracy[a] (%R)	MDL (mg/kg)	PRQL (mg/kg)
o-Cresol	≤50	25–115	5	40
m & p – Cresol	≤50	25–115	5	40
1,4-Dichlorobenzene	≤86	20–124	5	40
ortho-Dichlorobenzene	≤64	32–129	5	40
2,4-Dinitrophenol	≤119	D–172[b]	5	40
2,4-Dinitrotoluene	≤46	39–139	0.3	2.6
bis(2-Ethylhexyl)phthalate[c]	≤100	8–158	5	40
Fluoranthene[c]	≤80	26–137	5	40
Hexachlorobenzene	≤319	D–152[b]	0.3	2.6
Hexachlorobutadiene[c]	≤64	24-116	1.2	10
Hexachloroethane	≤44	40–113	5	40
Nitrobenzene	≤72	35–180	5	40
Pentachlorophenol	≤128	14–176	5	40
Phenol[c]	≤55	5–112	5	40

a. Criteria apply to PRQL concentrations. Applies to laboratory control samples and laboratory matrix spikes. If a solid laboratory control sample material which has established statistical control limits is used, then the established control limits for that material should be used for accuracy requirements.
b. Detected; result must be greater than zero.
c. Site-specific target analyte; quantitative analysis may not be required for samples from all TRU Waste Generator/Storage Sites or waste streams.

Table 5. Summary of Quality Control Samples and Frequencies.

QC Sample	Minimum Frequencies	Acceptance Criteria	Corrective Action
Method Performance Samples	Seven (7) samples initially and four (4) semiannually	Meet Table 4 QAOs	Repeat until acceptable
DFTPP Tune	Every 12 hours	Meet Table 3criteria	Repeat until acceptable
Initial Calibration	5 point minimum Initially and as needed	CCC %RSDs ≤30 SPCC RRFs ≥0.050 RRFs for non-SPCC ≥0.01 Average RRF used if %RSD ≤15 Linear or quadratic regression used if %RSD >15; r or COD ≥0.990	Repeat until acceptable
Continuing Calibration	Every 12 hours	CCC %Ds ≤20 SPCC RRFs ≥0.050 RRFs for non- SPCC ≥0.01 Internal Standard RTs ±30 seconds from last daily calibration RTs Internal Standard areas 50 – 200% of last daily calibration areas	Repeat until acceptable
Laboratory Blanks	One (1) per analytical batch, or one per extraction group, whichever is more frequent	≤3× Program Required MDLs in Table 4	Reanalyze and/or re-extract, if possible. Flag sample data with BZ qualifiers; ensure an NCR* is initiated if a noncompliant blank is associated with reported data.
Matrix Spikes	One (1) per analytical batch	Meet Table 4 accuracy QAO	See Section 8.3.3.3 Flag sample data with Z qualifier; ensure an NCR* is initiated if the failure was not due to sample matrix.
Matrix Spike Duplicates	One (1) per analytical batch	Meet Table 4 precision and accuracy QAOs.	See Section 8.3.3.3 Flag sample data with Z qualifier. Ensure an NCR* is initiated if the failure was not due to sample matrix.

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QC Sample	Minimum Frequencies	Acceptance Criteria	Corrective Action
Laboratory Control Samples	One (1) per analytical batch	Meet Table 4 accuracy QAOs	Reanalyze and/or re-extract, if possible Ensure an NCR* is initiated if a noncompliant LCS is associated with reported data, and flag data with Z qualifier.
Surrogate Compounds	Each analytical sample	Average %R from minimum of 30 samples of similar matrix ±3 standard deviations	Reanalyze and/or re-extract, if possible Flag surrogate data with Z qualifier; ensure an NCR* is initiated if the failure was not due to sample matrix.

NCRs are initiated in accordance with CCP-QP-005, *CCP TRU Nonconforming Item Reporting and Control